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May 24, 2017

Delivered via FedEx Overnight Delivery

Ms. Bobbi Coleman
South Carolina Department of Health and Environmental Control
Assessment Section, UST Management Division
Bureau of Land and Waste Management
2600 Bull Street
Columbia, SC 29201



Subject: Response to Comments in SCDHEC Letter titled "Quality Assurance Project Plan Revision Request," dated April 26, 2017
Plantation Pipe Line Company
Lewis Drive Remediation Site
Belton, South Carolina
Site ID #18693, "Kinder Morgan Belton Pipeline Release"

Dear Ms. Coleman,

On behalf of Plantation Pipe Line Company (Plantation), CH2M HILL Engineers, Inc. (CH2M) has prepared this response to comments received from the South Carolina Department of Health and Environmental Control (SCDHEC) in their letter date-stamped April 26, 2017, requesting that an addendum to the *Quality Assurance Project Plan, Revision 2 (QAPP)* submitted on March 1, 2017 (CH2M, 2016), be provided within 30 days.

Each of the comments provided in the SCDHEC correspondence are listed below, followed by Plantation's response to the comment.

Comment 1: Section A3. All field personnel should be added to the Distribution and Project Organization List.

Response: Section A3 has been revised to include field personnel.

Comment 2: Section A3. A statement that documents that everyone on the Distribution and Project Organization List has received the most recent version of the Site Specific Quality Assurance Project Plan for the Plantation Pipe Line Lewis Drive Release and the UST Management Division Quality Assurance Program Plan (QAPP), as it is referenced in the Site Specific Quality Assurance Project Plan for the Plantation Pipe line Lewis Drive Release.

Response: Section A3 has been revised to include the requested statement.

Comment 3: Section A4. More details need to be provided regarding the surrounding properties, impact to health and the environment, receptors, areas affected, and the environmental media affected (e.g., soil, surface water, and ground water).

Response: Section A4 has been revised as requested.

Comment 4: Section A6. A Topographic Map should be provided as Figure 1.

Response: A topographic map is provided on Figures 2 and 3

Comment 5: Section A6. For the subsections entitled "Monitoring Well Installation and Development" through "Well Development" detailed Standard Operating Procedures (SOPs) will need to be included as appendices.

Response: SOPs have been included as an attachment and are referenced in Section A6.

Comment 6: Section A6. In accordance with Section B.32 of South Carolina Well Regulations and Standards (R.61-71) an open hole in a bedrock aquifer is designated as a Type I Well. The remainder of the wells, as described, would fall under the definition of a Type III Well.

Response: Section A6 well type text has been revised.

Comment 7: Section A8. This section should state that personnel will receive the most current version of the QAPP Contractor Addendum and UST QAPP, since this document is referenced in the Site Specific Quality Assurance Project Plan for the Plantation Pipe Line Lewis Drive Release.

Response: Section A8 text has been revised to include the UST QAPP.

Comment 8: Section B1. Table 3A end dates for "Surface Water Sampling and Analysis" and "Groundwater Sampling and Analysis" should be changed to "Upon approval by DHEC or Project Completion".

Response: Section B1 text has been revised as requested.

Comment 9: Section B2. All sampling methods must be addressed in SOPs in appendices.

Response: Sampling methods are addressed in the SOPs that are included as Appendix A.

Comment 10: Section B4. A section included in Table 5A needs to be provided includes the equipment needed and references the attached SOP for each method.

Response: Table 5A has been revised as requested.

Comment 11: Section B4. Water quality parameters, to include turbidity, dissolved oxygen, specific conductance, temperature, and pH will need to be included.

Response: Section B4 text has been revised to include the water quality parameters.

Comment 12: Sections B6 and B7. Tables 6A and 7A should list the make and model of all pumps and meters that will be used for this project.

Response: Tables 6A and 7A have been revised to include the requested information.

Comment 13: Sections B6 and B7. This section should include specifically how the laboratory prevents unauthorized changes to laboratory notebooks & computer records.

Response: These matters are addressed in Section B10 Data Management. ECS Laboratories are certified by the State of South Carolina as an environmental laboratory. Such certification requires that the laboratory meet certain criteria regarding acceptable procedures, methodology, techniques, facilities, quality control, records keeping, and equipment, in accordance with South Carolina Regulation 61-81

Comment 14: Additional Items. SOPs must be provided for each task as appendices for all types of work performed at the site. This should cover, but not be limited to, the following (to include accompanying diagrams if applicable):

- *Groundwater purging and sampling procedures using low flow methods*
- *Groundwater sampling procedures using HydraSleeves*
- *Collection of surface water samples*
- *Installation of permanent groundwater monitoring well via each potential method*
- *Installation of permanent bedrock monitoring wells*
- *Well development procedures*
- *Well gauging for groundwater elevation*
- *Well gauging for free product*
- *Stabilization procedures when collecting groundwater samples*
- *Calibration of instruments procedures*
- *Free product recovery procedures*

Response: QAPP has been revised to include Appendix A. Appendix A includes the requested SOPs.

Comment 15: Additional Items. A list of all existing wells, sumps, trench points, and surface water sampling locations will also need to be included in the revision.

Response: A list of the requested features has been included in the revised QAPP.

If you have any further questions or concerns, please call me at (919) 760-1777, Mr. Scott Powell/CH2M at (678) 530-4457, or Mr. Jerry Aycock/Plantation at (770) 751-4165.

Regards,
CH2M HILL Engineers, Inc.



William M. Waldron, P.E.
Senior Project Manager

Attachments:

Attachment 1 – Quality Assurance Project Plan (Revision 3)

c: Jerry Aycock, Plantation (Digital, Jerry_Aycock@kindermorgan.com)
Mary Clair Lyons, Esq., Plantation (Digital, Mary_Lyons@kindermorgan.com)
Richard Morton, Esq., Womble Carlyle Sandridge & Rice, PLLC (Digital, rmorton@wcsr.com)
File

References

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

South Carolina Department of Health and Environmental Control (SCDHEC). 2017. "Quality Assurance Project Plan Revision Request." Letter to Plantation Pipeline Company. April 26.

South Carolina Regulation 61-81. State Environmental Certification Program.
<http://www.scdhec.gov/environment/EnvironmentalLabCertification/LawsRegulations/>

Attachment 1
QAPP Rev. 3

Quality Assurance Project Plan (Revision 3)

Section A: Project Management

A1 Title and Approval Page

Quality Assurance Project Plan
Addendum to the SCDHEC UST Programmatic QAPP
For
Plantation Pipe Line Company/Site ID No. 18693

Lewis Drive, Belton, Anderson County, South Carolina

Prepared by: CH2M HILL Engineers, Inc. (CH2M)

Date: February 9, 2015
Revised: May 24, 2017
CH2M HILL Engineers, Inc. (CH2M)

Approvals


Bobbi Coleman
SCDHEC Project Manager

Signature Date _____

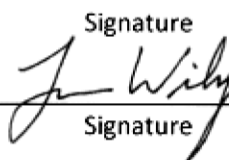
Jonathan Grimes
Contractor QA Manager


Signature Date 5/24/2017

William Waldron
Contractor Project Manager


Signature Date 5/24/2017

Tom Wiley
Plan Preparer


Signature Date 5/24/2017

Other signatures may be required and should be added as directed by SCDHEC UST Management Division.

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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 |
|-----------------|----------------------------------|--------------------------|---|------------------|--|
| Bobbi Coleman | SCDHEC Technical Project Manager | | SCDHEC, UST Management Division, 2600 Bull St., Columbia, SC, 29201 | 803-898-0673 | colemabi@shec.sc.gov |
| Thomas Kessler | Senior Technical Consultant | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4197 | Thomas.Kessler@CH2M.com |
| William Waldron | Contractor Project/Site Manager | | CH2M 3120 Highwoods Blvd Suite 214 Raleigh, NC 27604 | 919-760-1777 | wwaldron@ch2m.com |
| Tom Wiley | Site Assessment Manager | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4388 | twiley@ch2m.com |
| Jonathan Grimes | Lead Hydrologist | PG/ 2235/6/30/19 | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4146 | jgrimes@ch2m.com |
| Gerald Couch | Contractor Field Team Leader | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-488-8837 | Gerald.Couch@ch2m.com |
| Chris McCord | Laboratory Manager | | ESC Lab Sciences 12065 Lebanon Rd Mt. Juliet, TN | 704-614-2660 | bkroll@esclabsciences.com |
| Bill Barnes | Drilling Manager | Driller/ 562-A/ 6/30/17 | AE Drilling 2 United Way Greenville, SC 29607 | 864-288-1986 | mjohnson@aedrilling.com |
| James Pearsall | Surveyor | Surveyor/ 27458/ 6/30/18 | Taylor Wiseman & Taylor 700 Forest Point Circle Suite 166 Charlotte, NC 28273 | 704-527-2535 | pearsall@taylorwiseman.com |
| Justine McCann | Geologist | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4347 | Justine.McCann@ch2m.com |

| Name | Title/Role from UST Master QAPP | License/Number/Exp. date | Organization/Address | Telephone Number | Email Address |
|----------------|---------------------------------|--------------------------|--|------------------|--|
| Melissa Warren | Biologist | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4316 | Melissa.Warren@ch2m.com |
| Ryan Brown | Engineer | | CH2M Embassy Row 400 6600 Peachtree Dunwoody Road NE, Suite 600 Atlanta, GA 30328 | 678-530-4055 | Ryan.Brown2@CH2M.com |
| Paula Kramer | Engineer | | CH2M 3120 Highwoods Blvd Suite 214 Raleigh, NC 27604 | 919-760-1754 | Paula.Kramer@ch2m.com |
| Micheal Tekle | Geologist | | CH2M Cullman Building 14120 Ballantyne Corporate Place Suite 200 Charlotte, NC | 704-544-4028 | Micheal.Tekle@ch2m.com |
| Matthew Sumner | Geologist | | CH2M Cullman Building 14120 Ballantyne Corporate Place Suite 200 Charlotte, NC | 517-515-9358 | Matt.Sumner@ch2m.com |

It is understood that certification records must be produced if requested by SCDHEC. 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 Plantation Pipe Line Lewis Drive Release and the UST Management Division Quality Assurance Program Plan (QAPP).

A4 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.

- Plantation Pipe Line Company (Plantation) 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 the 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, Plantation determined the release to be gasoline with a minor amount of diesel, Plantation and its contractors repaired the pipeline, installed product recovery sumps, product recovery wells, temporary wells, and product interceptor trenches upgradient of Brown's Creek (Figure 1). Between December 2014 and February 2017, Plantation and its contractors have installed 60 monitoring wells, 36 piezometers, and a sparging remediation system consisting of 45 vertical wells and 3 horizontal wells. Figure 1 also illustrates the extent of product as of May 2016.
- Impacts to groundwater have been contained, with the exception of a small area where impacted groundwater flows to Brown's 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 can be developed. The lateral extent of

impacts to groundwater extend in three directions from the release point: to the north approximately 900 feet into an adjacent hay field; to the northeast approximately 1,000 feet to Brown's 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.

- The results of a local well survey performed in December 2014 indicate no public or private water wells 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.
- Minor impacts to surface water in Brown's Creek have occurred in the area where impacted groundwater discharges to Brown's 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-12. 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 SCDHEC UST Management Division.

A5 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 product body area, 2) the establishment of additional surface water sampling stations to monitor surface water quality in Cupboard Creek and Browns Creek and wetland area that borders the southern edge of the site; 3) the installation of up to 16 vertical bedrock sparging wells within the shallow bedrock layer to allow the injected air to be distributed via the same fracture network that transmitted impacts to groundwater in this zone.
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.

A6 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.

- Figure 2 shows the locations of existing and proposed monitoring wells and sparging wells. Figure 3 shows the locations of surface water sampling points.

Monitoring Well Installation and Development

Regolith Monitoring Well Construction

The regolith monitoring wells will be constructed as Type III 2-inch diameter monitoring wells, and will be constructed in accordance with SCDHEC Well Standards R.61-71 and 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.

Hand Installation of Regolith Monitoring Well

At one location near Brown's Creek, it was determined that installing a well was not feasible using a mechanical drill rig, due to steep slopes and ditches and unstable ground surface (super saturated soils). Therefore the well (MW-34) will be installed using a hand auger due to site access issues.

The borehole will be advanced to a target depth of approximately 5 feet using a hand auger to create a nominal 4-inch diameter borehole. During borehole advancement, soil samples will be field screened for VOCs using a photoionization detector and characterized for lithology using the soil cuttings collected from the auger bucket.

The well will be constructed using 2.5-feet of schedule 40 PVC 2-inch prepacked internal diameter (ID) by 2.8-inch OD 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. Additional sand pack shall be placed in the annular space between the borehole and prepacked well screen and shall be brought to a height 0.5-foot above the top of the well screen. A bentonite seal with a minimum thickness of 12-inches shall be placed above the sand pack and shall be hydrated. The seal shall be allowed to hydrate for a minimum of 1-hour before placing grout above the seal. A grout seal of at least 1-foot length, containing Portland cement mixed with 3 to 5 percent bentonite shall be placed above the grout seal and shall be brought to within 1-foot of ground surface.

The aboveground completion will be constructed above grade using a 6-inch diameter, approximately 3-feet high, locking anodized aluminum protective well casing set in a cylindrical concrete pad. The concrete pad will extend 1 ft above and 1 ft below the ground surface to ensure a better surface seal and protect the well from flooding. A weep hole will be drilled in the protective casing.

Bedrock Monitoring Wells

The five bedrock wells will be constructed as Type I wells (open hole in bedrock aquifer). The wells will be constructed in accordance with SCDHEC 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 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.

A7 Certification

The following laboratory will be used for this project:

Commercial Lab(s)

Full Name of the Laboratory: ESC Lab Sciences

Name of Lab Director: Eric Johnson

SCDHEC Certification Number: 84004002

Please note: SCDHEC may require that the contractor submit some or all of the Laboratory's SOPs as part of this QAPP.

A8 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 2A*Record Identification, Storage, and Disposal*

| Record | Produced By | Hardcopy/ Electronic | Storage Location For how long? | Archival |
|-------------------|--------------------|--|---|--|
| Monitoring Report | CH2M | Hardcopy and electronic copies to be provided to SCDHEC | Five years from date of report | Electronic copy is stored on CH2M and PLANTATION network |

Section B Measurement/Data Acquisition

B1 Sampling Process/Experimental Design

Table 3A
Sampling Activities

| Task | Start Date | End Date | Comments |
|--|-------------------|---|-----------------|
| QAPP revision preparation and submittal | May 8, 2017 | May 26, 2017 | |
| QAPP approval | | June 9, 2017 | |
| Monitoring well and bedrock sparging well installation and development | July 10, 2017 | July 21, 2017 | |
| Surface water sampling and analysis | June 26, 2017 | Upon approval by DHEC or project completion | |
| Groundwater Sampling and analysis | June 27, 2017 | Upon approval by DHEC or project completion | |
| Surveying | July 20, 2017 | July 21, 2017 | |

Note: This schedule assumes regulatory approval by June 9, 2017

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:

| Matrix | Number of Samples (per event) |
|-----------------------------------|-------------------------------|
| Groundwater from monitoring wells | 26 |
| From surface water | 16 |
| Duplicate samples | 3 |
| Field blanks | 5 |
| Trip blanks | 6 |
| Total number of samples | 56 |

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 SCDHEC UST Division Programmatic QAPP (May 2015).
- 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 as close to the top of the water column as possible. 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.
- 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 homogenized or split 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.

If decontamination procedures differ from Appendix H, please provide details.

- Decontamination procedures will be performed in accordance with the procedures described in Appendix H.

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

- CH2M 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 4A
Field Corrective Action

| Failure | Response | Documentation | Individual Responsible |
|--|---|----------------------------|------------------------|
| 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 other 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 chain of custody 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 5A
Analytical SOPs and Referenced Methods

| Parameter | Method Referenced | Comments/SOP |
|--|-------------------|--|
| Soils | | |
| BTEX, naphthalene | EPA Method 8260B | Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016* |
| Groundwater | | |
| BTEX Naphthalene MTBE 1,2-DCA | EPA Method 8260B | Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016* |
| Surface Water | | |
| BTEX, naphthalene | EPA Method 8260B | Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016* |
| Water Quality Parameters | | |
| 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 6A
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 7A
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 SCDHEC 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, i.e.,
3. Provide its relevance to the project.
4. Indicate the justification criteria for use of these data sources and/or models.

Table 8A
Non-Direct Measurements

| Data Source | Used for | Relevance | Justification for use in this project | Comments |
|---------------------------|--|--|---|-----------------|
| 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 SCDHEC 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 | |

5. Identify key resources/support facilities needed.
 - Not applicable.

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 Chain-of-Custody (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 CH2M's 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 chain of custody, 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 CH2M's networks that are backed up each day on to an off-site 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 samples 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 CH2M 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 "My ESC" web link. After two years, the reports will be archived electronically on-site or off-site 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 CH2M server with the same unique number as the hard copy.
 - CH2M 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 5-years.

Section C Assessment and Oversight

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 CH2M employees working on this project will verify that the samples are collected and handled properly. Additionally, all CH2M employees working on this project have the authority to stop work, report the problem and effect a correction that is agreed upon by the CH2M PM.

2. The SCDHEC 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 SCDHEC Office of Environmental Laboratory Certification. The laboratory is accredited by the SCDHEC 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 SCDHEC UST Programmatic QAPP (UST Master QAPP).

Section D Data Validation and Usability

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



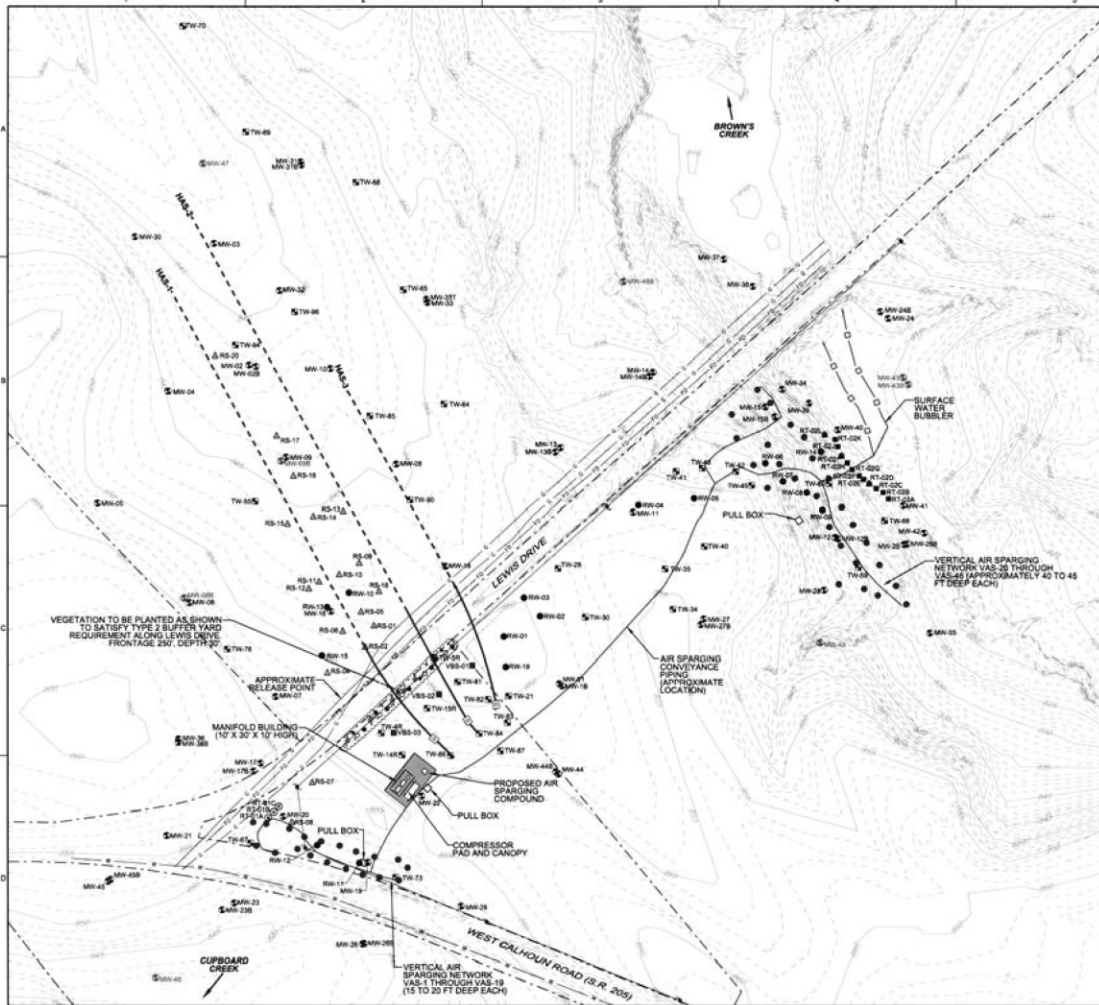
LEGEND

- ★ Release Point
- Monitoring Well
- Bedrock Monitoring Well
- Proposed Location of Monitoring Well MW-34
- △ Recovery Sump
- ▲ Abandoned Recovery Sump
- Piezometer ("R" indicates Replacement)
- Abandoned Temporary Piezometer
- Recovery Well (4" diameter)
- Surface Water Sampling Location
- Septic Tank
- Recovery Trench Point
- Recovery Trench
- Surface Water Flow Direction
- Utility (approximate)
- Soft Boom
- Access Route
- ~ Extent of Product based on 5/06/2016 and 5/10/2016 data
- ~ National Hydrography Dataset Stream
- ▨ Delineated Wetland
- ▨ Beaver Dam
- ▭ Detail Area
- 0.04 Product Thickness in feet as of 5/06/2016 and 5/10/2016
- NP No Product detected

Source Data:
 *ESRI World Imagery Layer, 2015
 *United States Geological Survey (USGS) National Hydrography Dataset (NHD)

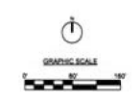
0 175 350
 Scale in Feet

Figure 1. Product Thickness Map, May 2016
 Quality Assurance Project Plan
 Lewis Drive Remediation Site, Belton, South Carolina
 Site ID #18693 "Kinder Morgan Belton Pipeline Release"
ch2m



- ### LEGEND
- ⊕ MONITORING WELL
 - ⊕ MONITORING WELL PROPOSED
 - ⊕ PIEZOMETER
 - △ RECOVERY SLUMP
 - RECOVERY WELL
 - RECOVERY TRENCH POINT
 - HORIZONTAL BIOSPARING ENTRY POINT
 - VERTICAL SAPROLITE BIOSPARING WELL
 - VERTICAL BEDROCK BIOSPARING WELL
 - PULL BOX
 - CANOPY
 - UNDERSTORY
 - EVERGREEN
 - SHRUB
 - SURFACE WATER BUBBLER
 - HORIZONTAL SPARGE WELL
 - - - HORIZONTAL SPARGE WELL (SCREENED)
 - AIR SPARGING CONVEYANCE PIPING
 - PROPOSED FENCE
 - POWER LINE
 - - - EXISTING FENCE
 - PIPE LINE
 - FIBER OPTIC LINE
 - - - PROPERTY BOUNDARY
 - WATER LINE

NOTES
 1. BASEMAP SURVEY PERFORMED BY TAYLOR, WISEMAN & TAYLOR, FEBRUARY 4, 2016.



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PLANTATION PIPELINE COMPANY
 LEWIS DRIVE REMEDIATION
 QUALITY ASSURANCE PROJECT PLAN
 SITES 1 & 2 (MHW-100)
 KENDLER MORGAN BELT TYPICAL RELEASE

ch2m.
 Figure 2 Sparging Layout
 Quality Assurance Project Plan
 Lewis Drive Remediation, Belton, South Carolina
 Site # 16002 "Pulp Mill Sludge Spillage Recovery"

DATE: MARCH 2017
 PROJ: 856401
 CHW:
 SHEET

(PW: CH2MHILL.DEN01 | 856401 | LEWIS DRIVE (BOD) FILENAME: Figure 2_856401_Rw2.dgn PLOT DATE: 20170923 PLOT TIME: 9:33:11 AM



LEGEND

- ★ Release Point
- Surface Water Sampling Location
- Fish Pond Surface Water Sampling Location
- Utility (approximate)
- *** Inspection Route for Sheen or Distressed Vegetation
- Flow Direction of Creek
- ~ Topographic Contour (5-foot Interval)
- ~ National Hydrography Dataset Stream
- Delineated Welland
- ⊗ Beaver Dam



Base Map Source:
 *Environmental Systems Research Institute (ESRI) ArcMap
 World Imagery, 2015
 *United States Geological Survey (USGS) National
 Hydrography Dataset (NHD)

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

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)



PROJECT NUMBER

WELL NUMBER

SHEET 1 OF 1

WELL COMPLETION DIAGRAM

PROJECT :

LOCATION :

DRILLING CONTRACTOR :

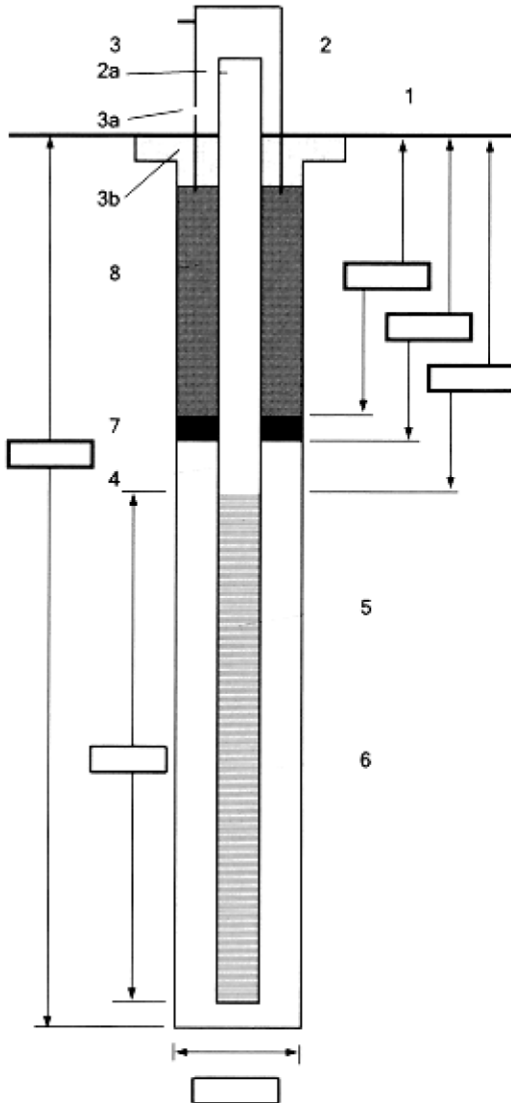
DRILLING METHOD AND EQUIPMENT USED :

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- 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 (MWDDoubleDiag.xls)



PROJECT NUMBER

WELL NUMBER

SHEET 1 OF 1

WELL COMPLETION DIAGRAM

PROJECT :

LOCATION :

DRILLING CONTRACTOR :

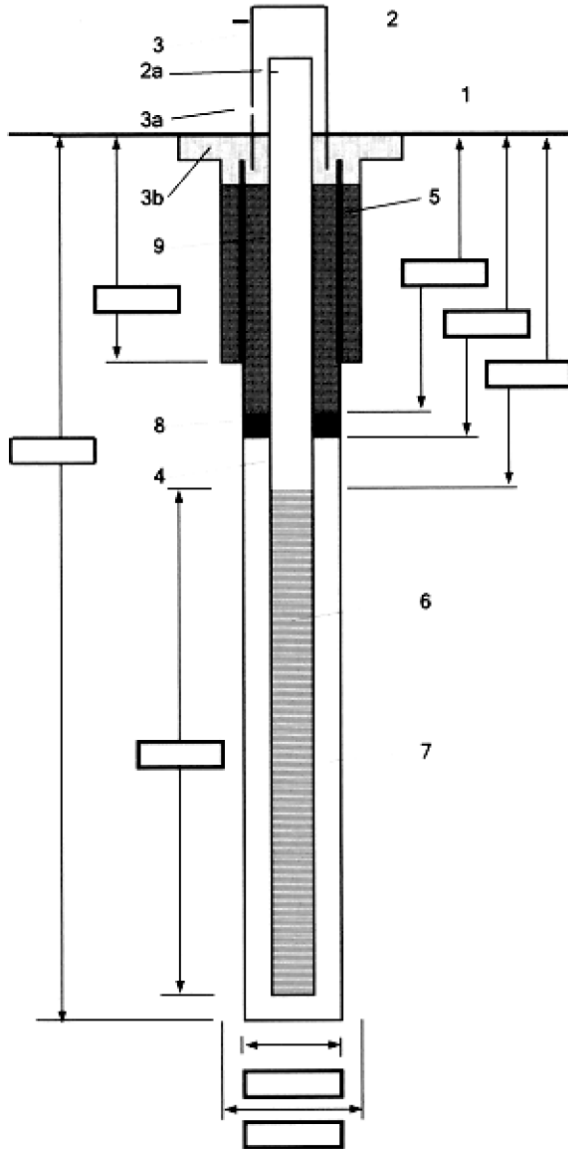
DRILLING METHOD AND EQUIPMENT USED

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 (MWDDoubleDiag.xls)



PROJECT NUMBER

WELL NUMBER

SHEET 1 OF 1

WELL COMPLETION DIAGRAM

PROJECT :

LOCATION :

DRILLING CONTRACTOR :

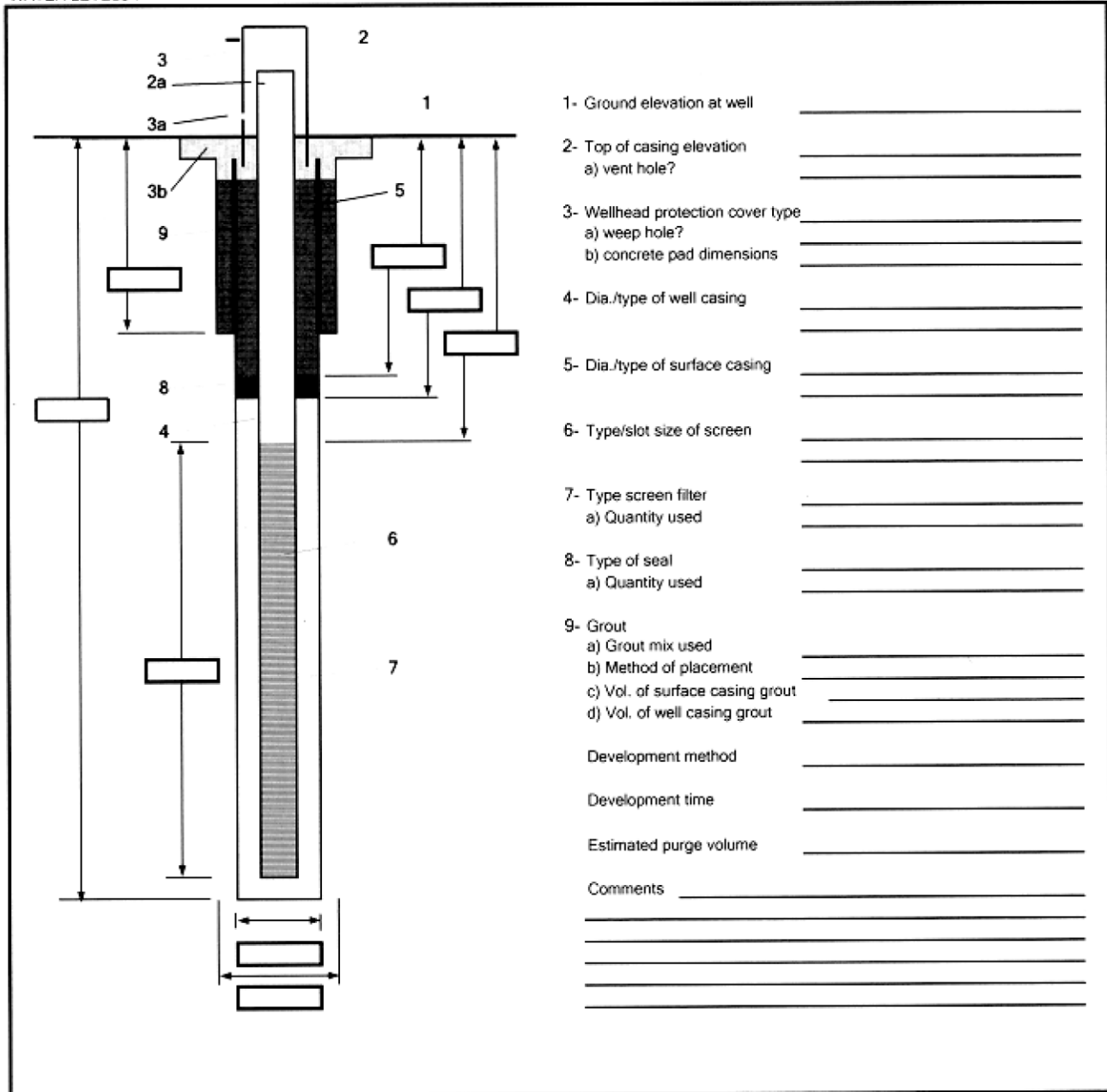
DRILLING METHOD AND EQUIPMENT USED

WATER LEVELS :

START :

END :

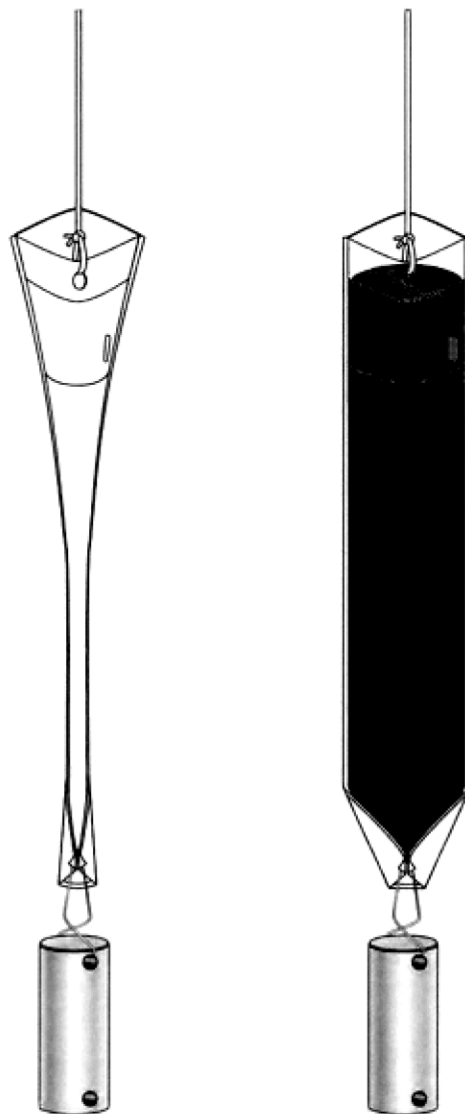
LOGGER :



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.

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Introduction

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

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.

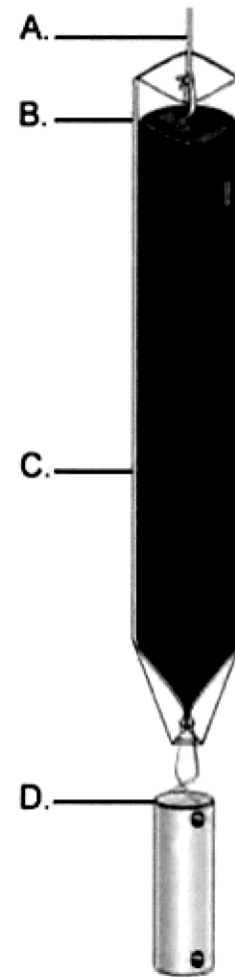


Figure 1. HydraSleeve components.

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

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 ½ feet) to 81" (6 ¾ 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.

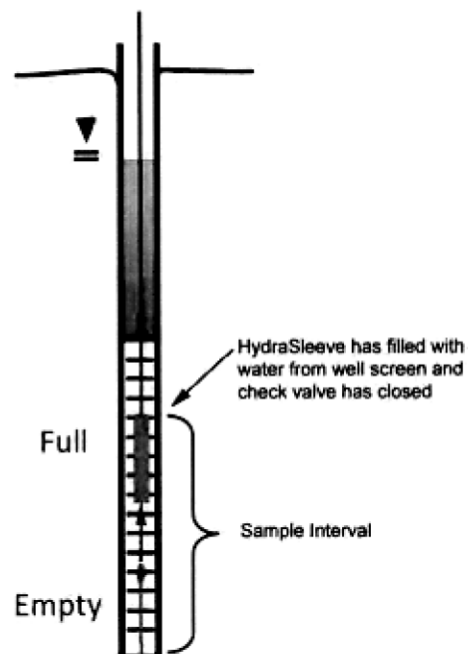


Figure 2. Correct placement of HydraSleeve.

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.

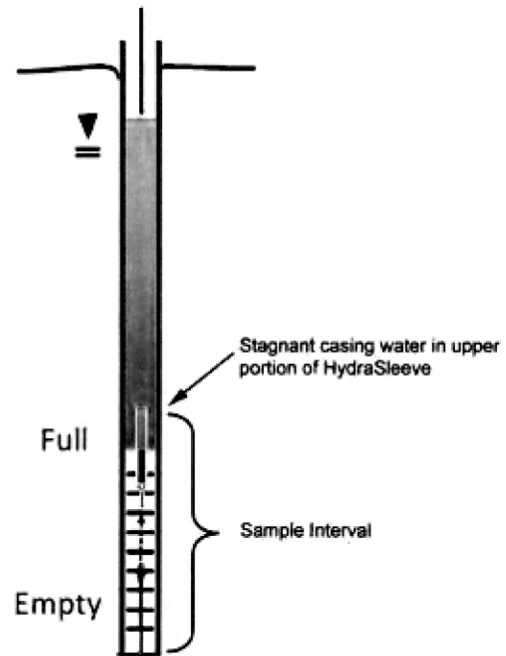


Figure 3. Incorrect placement of HydraSleeve.

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

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

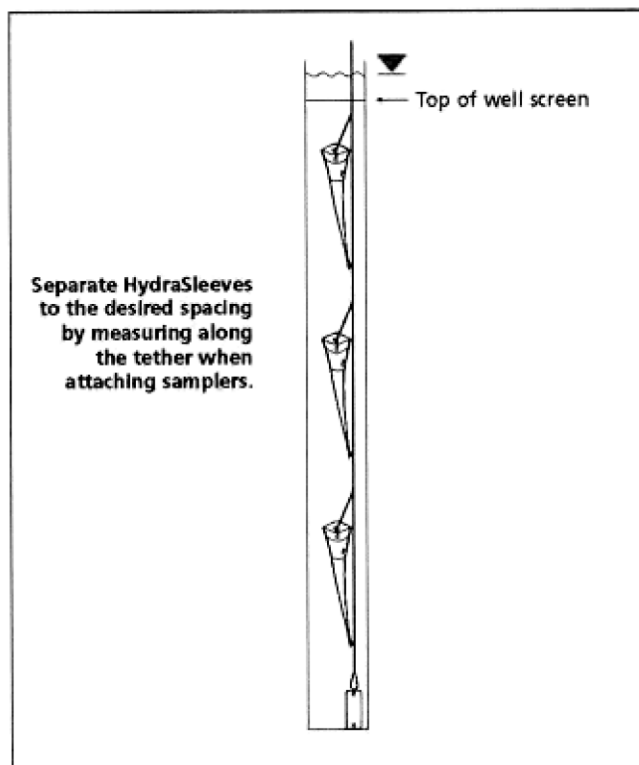


Figure 4. Multiple HydraSleeve deployment.

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).

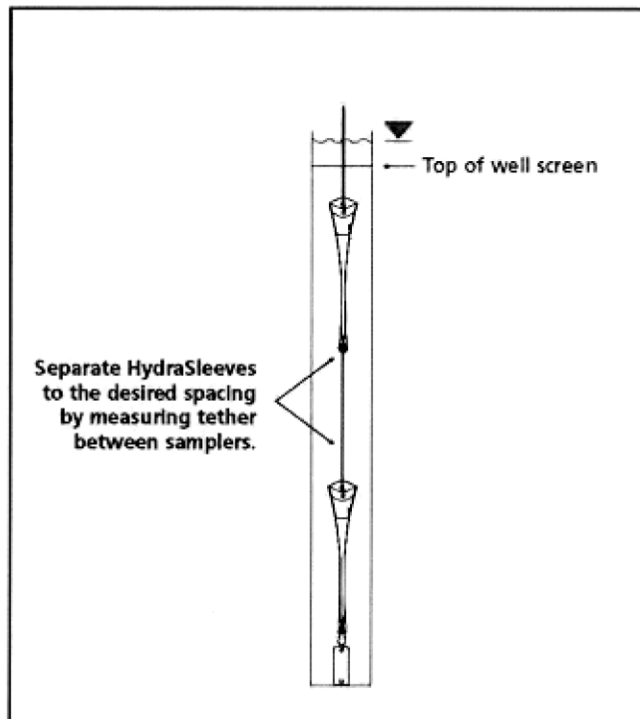


Figure 5. Alternative method for deploying multiple HydraSleeves.

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

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

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 rinsate 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.

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.



SOP Minor Revision Summary

| SOP: | | | | | |
|------------|---|-------------|------------|--------------|-----|
| Author - | M. Jacobs | Number - | 330363 | Department - | VOC |
| Title - | VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B) | | | | |
| Revision - | R23 | Rev. Date - | 10/18/2016 | | |

This Standard Operating Procedure has been amended to include changes required during normal business operations. These changes as defined by SOP 010103 (Document Control and Distribution) are routine modifications that will be incorporated into the SOP upon the next scheduled review.

| Rev. | Date | Section | Brief Description |
|------|----------|---------|---|
| a | 11/4/16 | 10.9 | Revised State Note regarding South Carolina LCS/LCSD recovery limit values. |
| | | 10.10 | Added State Note regarding South Carolina MS/MSD recovery limit values. |
| b | 12/28/16 | 8.3.3 | Add Methyl cyclohexane to the poor performer list. |
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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Reviewed by: Heidi Ferrell, Steve Miller

Department Manager

QA Department

This document bears a watermark in the lower right hand corner of each page. This watermark is an insignia of the document's approval. The signed original is on file in the ESC Quality Assurance Department.

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize SOP #330363OH.

STATE NOTE: For samples analyzed in conjunction with the NY ELAP or WA DOE, utilize the criteria for EPA Method 8260C.

- 1.1 This standard operating procedure is used to determine volatile organic compounds in a variety of matrices. This SOP is designed for EPA methods 8260B, 624, 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 Attachment III, which contains a list of the typical primary and secondary ions used in determining these compounds.
- 1.2 Reporting Limits (RLs) are listed in the Attachment II. Compounds routinely analyzed by this method and their typical reporting limits are included in the following table (subject to change).
- 1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ESC SOP #030206. Updated MDL records are filed and stored in a central location within the department.
 - 1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ESC SOP #030206, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*. Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.
 - 1.3.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.3 and 10.15).

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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

2.0 METHOD SUMMARY AND DEFINITIONS

- 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 (SOP #330752), 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 to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.
- 2.2 For other samples, Method 5035 (SOP #330751), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.
- 2.3 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.
- 2.4 See the current Quality Assurance Manual for definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

- 3.1 The toxicity or carcinogenicity of each reagent 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. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.
- 3.2 Glycol ethers are suspected carcinogens. All solvent handling should be performed in a hood while using proper protective equipment to minimize exposure to liquid and vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

- 4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

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- 4.2 Volatile analysis for water and sodium bisulfate 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 7 days. It is also an ESC requirement that water samples with 2-chloroethylvinyl 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 hrs from the time of collection, added to preservative or otherwise frozen at $\leq -7^{\circ}\text{C}$. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (SOP #330751).

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 VOC's require 7-day Holding Time.

- 4.3 Aqueous samples must be collected in at least duplicate in 40mL vials with 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ per liter if residual chlorine is present. Sample kits can be configured to request additional vials per client request. The pH must be adjusted to <2 with HCl. Soil samples must 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_4 to a $\text{pH}<2$ and one 5g sample preserved in the field with methanol (for high level analysis) 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. All samples and extracts must be shipped and stored at $<6^{\circ}\text{C}$.

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 $\text{pH}>12$.

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 9.9 for the calculation.

5.0 INTERFERENCES

- 5.1 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.

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- 5.2 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.
- 5.3 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 must be analyzed to check for cross contamination.
- 5.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.
- 5.3.2 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.
- 5.3.3 In extreme situations, the whole purge and trap device may require dismantling and cleaning.
- 5.3.4 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 with an automated headspace technique by SW-846 Method 3820, Hexadecane Extraction and Screening of Purgeable Organics), or screening the sample using an HNU or equivalent portable PID.
- 5.4 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 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.
- 5.5 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 blank is prepared from organic-free water and is placed in the cooler for a period of two weeks. Every two weeks, it is analyzed to verify that no contamination of client samples has taken place due to contamination in the storage unit. Analysis of a storage blank

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must be performed for the volatiles laboratory walk-in cooler as well as the Drinking Water Refrigerator

- 5.6 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.
- 5.7 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.
- 5.8 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.
- 5.9 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP #030701, *Glassware Cleaning*.

6.0 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.

- 6.1 Instrumentation: All instrumentation meets or exceeds EPA method requirements. There are a total of 30 instruments; GCs include models 5890, 7890 or equivalent and MSs include 5973, 5975, 5977 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
- 6.2 Glassware must be scrupulously cleaned. All glassware must be cleaned per EPA protocol, as stated in SOP #030701, *Glassware Cleaning*. Rinsing with methanol and laboratory reagent water cleans the volumetric flasks and graduated cylinders. The volumetric flasks are dried in a low temperature oven at less than 120°C and never cleaned with a brush or strong alkali solution.
- 6.3 The carrier gas used for volatiles analysis is Helium-5.0 grade.
- 6.4 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.

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6.5 Glass Sample (VOA) and Standard Vials:

- 6.5.1 40mL VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.
- 6.5.2 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards)

6.6 Miscellaneous:

- 6.6.1 Stainless Steel Spatula or wooden tongue depressor.
- 6.6.2 Disposable aluminum drying dishes - VWR #25433-008, or equivalent
- 6.6.3 Teflon™-coated stir bars, 8mm x 16mm
- 6.6.4 Glass beads –VWR #EM1.04015.0500, or equivalent.

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

6.8 Analytical Balance, capable of weighing to 0.01g, or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

7.2 Laboratory water created by reverse osmosis/DI filtration evaluated to .055uS/cm to ensure purity. Laboratory water is used in all blanks to assure that it contains less than the MDL 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 MDL all samples associated with this blank must be flagged (see Section 11.4).

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.

7.3 Methanol, CH₃OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

7.4 Sodium Bisulfate, Na₂S₂O₃ - QEC Level 3 Certified, or equivalent

7.5 STOCK SOLUTIONS – Calibration and LCS

- Stock calibration solutions must be purchased as certified solutions.
- Certificates must be kept on file.

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- All Stock standards must be stored below -10° C
- 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.
- 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.

7.5.1 CALIBRATION - Primary source standards is used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the initial calibration verification (ICV), and continuing calibration verification (CCV) standard. The ICV and CCV are analyzed to verify the initial calibration and are prepared using the primary source standards used to produce the calibration curve. See Section 8.2.6 through 8.2.8 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 date of the stock standards from which they are prepared. Once diluted, the standard must be replaced weekly.

ICV Mix

The ICV solution is prepared in methanol in a 50mL volumetric flask by adding:

| Manufacturer | Product | Cat. # | Amount added (mL) | Final Conc. (ppm) |
|--------------|---------------------------|-----------|-------------------|-------------------|
| Restek | 8260 Custom Mix 1 | 565050 | 1 | 50 |
| NSI | 8260 Custom Mix 2 | Q-4147 | 1 | 250 |
| Absolute | n-Hexane & Heptane | 92484 | 2.5 | 50 |
| Ultra | 1+2 Methyl-naphthalenes | CUS-11449 | 2.5 | 50 |
| Restek | Acrolein | 30645 | 1 | 250 |
| SPEX | AZ fuel additive 4 comps. | VO-ESCTN9 | 2 | 50 |
| Ultra | 8260 Gases Mix | CUS-13691 | 25 | 50 |

7.5.1.1 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 -10° C.

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GRO ICV Mix

| Manufacturer | Product | Cat. # | Conc. (ppm) |
|--------------|-------------------------|-----------|-------------|
| Restek | TX TPH matrix spike mix | Cat 31484 | 10,000 |

AP9/Oxygenates ICV 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 | 5 |
| Absolute | Isopropanol | 70941 | 0.625 | 25 |

7.5.2 LABORATORY CONTROL STANDARDS - The standard list of target LCS compounds are those compounds listed in Attachment VIII. Secondary source standards must be used to prepare the laboratory control standard (LCS) and the matrix spike and their duplicates. 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 -10°C and have a six month holding time once opened, except the ICV gases which have 1 week holding time. Once diluted, the standard must be replaced weekly.

LCS & Matrix Spikes

Prepare the LCS/LCSD in methanol in a 50mL 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 Attachment VIII.

| Manufacturer | Product | Cat. # | Amount added (mL) | Final Conc. (ppm) |
|---------------|------------------------------|-----------|-------------------|-------------------|
| NSI | 8260 Custom Mix 1 | Q-4146 | 5 | 50 |
| NSI | 8260 Custom Mix 2 | Q-6354-O | 1 | 250 |
| Absolute | n-Hexane & Heptane | 92484 | 2.5 | 50 |
| Ultra | 1+2 Methyl-naphthalenes | CUS-11449 | 2.5 | 50 |
| Restek | Acrolein | 30645 | 1 | 250 |
| Ultra | 8260 Gases Mix | CUS-5661 | 25 | 50 |
| SpexCertiPrep | AZ Fuel Additive 4 Compounds | VO-ESCTN9 | 2 | 50 |

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GRO LCS

| Manufacturer | Product | Cat. # | Conc. (ppm) |
|--------------|------------|--------|-------------|
| Ultra | GRO-MO Mix | Q-4208 | 5000 |

7.5.2.1 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.

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

AP9/Oxygenates LCS

The AP9/Oxygenates LCS is prepared in methanol in a 25mL volumetric flask by adding:

| Manufacturer | Product | Cat. # | Amount added (mL) | Final Conc. (ppm) |
|--------------|---------------------|----------|-------------------|-------------------|
| NSI | Custom AP9 Standard | Q-6603-O | 3.125 | 5 |
| NSI | Ethyl Acetate | 681 | .125 | 50 |
| Absolute | Isopropanol | 70941 | .625 | 25 |

The LCS/LCSD 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.

7.6 Surrogate standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened. Surrogate spiking solutions are purchased from Ultra as custom standard CUS-10259, 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.

7.6.1 The following are ESC designated volatiles' analysis surrogates:

- Toluene-d8
- 4-Bromofluorobenzene
- Dibromofluoromethane
- $\alpha\alpha$ -Trifluorotoluene

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7.7 Internal standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below -10° C and have a six month holding time, once opened. [Internal standard and surrogate standard – Ultra custom MS/IS/SS mix CUS-10259, or equivalent]

7.7.1 The following are ESC designated volatiles' analysis internal standards:

- 1,4-Difluorobenzene
- 2-Bromo-1chloropropane
- 1,4-Dichlorobenzene-d4
- Pentafluorobenzene

7.8 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 -10° C and have a six month holding time, once opened.

7.9 Matrix spike (MS) standard - Stock standards must be stored at or below -10° C and have a six month holding time, once opened. Once diluted, the standard must be replaced weekly.

7.9.1 The matrix spike standard is prepared from the stock standard in Section 7.5.

7.9.2 The spike should be at a mid-level of the calibration range. Some contracts may require a site-specific concentration.

7.9.3 Standard spiking practice requires the use of ALL TARGET ANALYTES as specified in Attachment VIII and must be evaluated against the current control limits presented in the LIMS.

Project Specific Requirements (Non-South Carolina Projects): Individual projects may specify required spike compounds. In addition to any project specific requirements, the following table lists the minimum required compounds that must be included in the spike solution.

| Minimum Spiking Compounds for Project Specific Requirements |
|---|
| 1,1-Dichloroethene |
| Trichloroethene |
| Chlorobenzene |
| Toluene |
| Benzene |
| n-Hexane (when requested as a target analyte) |

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%.

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8.0 PROCEDURE

Analysis Summary: Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler, as described on Section 2. If soil samples are high in contamination, a methanolic extraction, SOP #330760, may be necessary prior to purge and trap analysis. Soils require method 5035 for sample preparation, See SOP #330751.

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

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

| | |
|-------------------|---------------------------------|
| Inlet | off |
| Detector | 200°C |
| Oven Equip. 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 |

8.1.2 Typical conditions for each autosampler are listed below:

| | |
|----------------|--------------------|
| Heating sample | 1 minute at 40°C |
| Purge | 11 minutes at 40°C |
| Desorb | 1 minutes at 250°C |
| Bake | 2 minutes at 260°C |

8.1.3 Typical condition 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 – 3×10^{-6} torr

8.2 Initial calibration

8.2.1 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 8260B, 8260C, and 6200B. BFB tuning for method 624 is every 24 hours.

| BFB Key Ions and Ion Abundance Criteria | |
|---|------------------------------------|
| Mass | Ion Abundance Criteria |
| 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 |

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| BFB Key Ions and Ion Abundance Criteria | |
|---|---|
| 176 | > 95.0%, but less than 101% of mass 174 |
| 177 | 5.0-9.0% of mass 176 |

STATE NOTE: BFB tuning must be performed every 12 hours when analyzing samples from South Carolina by Method 624.

8.2.2 Calibration Curve – General Criteria

- A minimum of 5-point calibration is performed using the primary standards listed in Section 7.5.1. 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: A reporting level standard must be run 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.

- 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

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analyte to meet the calibration requirements outlined in Sections 8.2.6 through 8.2.8.

- When using Method 5035, SOP #330751, 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.
- The method reference spectra must be updated from the mid-point of each calibration.

8.2.3 Calibration Levels for single analytes

8.2.3.1 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 ICV Mix (See Section 7.5.1) into 5mL of reagent water..

| Calibration Curve - GC/MS Soil (into 5mL water) | |
|---|---------------------------------|
| Intermediate solution volume (μ L) | Concentration of standard (ppb) |
| 0.05 | 0.5 |
| 0.1 | 1 |
| 0.2 | 2 |
| 0.5 | 5 |
| 1 | 10 |
| 2.5 | 25 |
| 5.0 | 50 |
| 10. | 100 |
| 20. | 200 |

| Calibration Curve - GC/MS Soil (into 50mL water) | |
|--|---------------------------------|
| Intermediate solution volume (μ L) | Concentration of standard (ppb) |
| 0.5 | 0.5 |
| 1.0 | 1 |

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| Calibration Curve - GC/MS Soil (into 50mL water) | |
|--|-----|
| 2.0 | 2 |
| 5.0 | 5 |
| 10 | 10 |
| 25 | 25 |
| 50 | 50 |
| 100 | 100 |
| 200 | 200 |

| AP9/Oxygenates Calibration Curve - GC/MS Soil (into 5mL of water) | |
|--|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppb) |
| 1 | 1 |
| 2.5 | 2.5 |
| 5 | 5 |
| 7.5 | 7.5 |
| 10 | 10 |
| 12.5 | 12.5 |
| 15 | 15 |
| 17.5 | 17.5 |
| 20. | 20 |

| AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water) | |
|---|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppb) |
| 10 | 1.0 |
| 25 | 2.5 |
| 50 | 5 |
| 75 | 7.5 |
| 10 | 10 |
| 125 | 12.5 |
| 150 | 15 |
| 175 | 17.5 |
| 200. | 20 |

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Note 1: When analyzing soil samples by the low-concentration method (Section 8.7.1), the calibration standards must be heated to $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$ 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.

8.2.3.2 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 ICV Mix (See Section 7.5.1.) into 5mL of water.

| Calibration Curve - GC/MS Water (into 50mL water) | |
|---|---------------------------------|
| Intermediate solution volume (μL) | Concentration of standard (ppb) |
| 0.5 | 0.5 |
| 1 | 1 |
| 2 | 2 |
| 5 | 5 |
| 10 | 10 |
| 25 | 25 |
| 40 | 40 |
| 75 | 75 |
| 100 | 100 |
| 200 | 200 |

| AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water) | |
|--|---------------------------------|
| Intermediate solution volume (μL) | Concentration of standard (ppb) |
| 10 | 1.0 |
| 25 | 2.5 |
| 50 | 5 |
| 75 | 7.5 |
| 10 | 10 |
| 125 | 12.5 |
| 150 | 15 |
| 175 | 17.5 |

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| AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water) | |
|---|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppb) |
| 200. | 20 |

| Calibration Curve - GC/MS Soil (into 50mL water) | |
|---|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppm) |
| .2 | 0.4 |
| 5 | 1 |
| 10 | 2 |
| 20 | 4 |
| 25 | 5 |
| 35 | 7 |
| 50 | 10 |
| 100 | 20 |

8.2.4 Calibration Levels for GRO by MS

8.2.4.1 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 ICV Mix (See Section 7.5.1.) into 5mL of reagent.

| Calibration Curve - GC/MS Soil (into 5mL water) | |
|--|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppm) |
| .02 | 0.4 |
| .5 | 1 |
| 1 | 2 |
| 2 | 4 |
| 2.5 | 5 |
| 3.5 | 7 |
| 5 | 10 |
| 10 | 20 |

| Calibration Curve - GC/MS Soil (into 50mL water) | |
|---|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppm) |
| .2 | 0.4 |
| 5 | 1 |
| 10 | 2 |

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| Calibration Curve - GC/MS Soil (into 50mL water) | |
|--|---------------------------------|
| Intermediate solution volume (µL) | Concentration of standard (ppm) |
| 20 | 4 |
| 25 | 5 |
| 35 | 7 |
| 50 | 10 |
| 100 | 20 |

8.2.5 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 to 5mL of sample is equivalent of 40µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix. Internal Standards are listed Section 7.7.1 and Surrogates are listed in Section 7.6.1.

Tabulation of the Internal Standards

Tabulate the area response of the characteristic ions (see Attachment III) 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.

8.2.6 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) 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.

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

Using the RF's 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 9.2. Linearity can be assumed if the RSD criteria is met, thus allowing quantitation calculations to be performed using RF.

8.2.7.1 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 |

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

| |
|--|
| <15% RSD for all 8260B Target Analytes |
| <35% RSD for all 624 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 |

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

8.2.7.3 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 criteria of 0.99 or better. SM 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 – $1/x^2$ Weighting
- Quadratic

8.2.7.4 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:

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- 8.2.7.4.1 Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.
- 8.2.7.4.2 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.

8.2.8 EPA 8260C: Response Factors (RF's)

- 8.2.8.1 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 Attachment VII, 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.
- 8.2.8.2 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%.

8.3 Calibration Verification

8.3.1 Calibration Verification for EPA Methods 8260B, 624 and SM 6200B:

8.3.1.1 SSCV's

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After a successful calibration, a Second Source Calibration Verification (SSCV) 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 |

8.3.1.2 CCC's

The curve must be verified initially by a calibration standard known as the Initial Calibration Verification standard (ICV). In addition, the same standard is analyzed every 12 hours for continuing calibration verification (CCV). 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,

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

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 $\leq 20\%$.

8.3.1.3 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.

8.3.1.4 All Target Analytes and Non-CCC's

When analyzing 8260B and 624 concurrently, calibration verifications are evaluated using both the 8260B criteria (sections 8.3.1.2 through 8.3.1.3 and the 624 criteria (Attachment VI). For analytes not on the 624 list, all target analytes (except for the poor performers (8.3.3) 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.

Poor performers are allowed a maximum of 50% drift from the calibration curve. See section 8.3.3 for a listing of poor performing analytes.

STATE NOTE: For South Carolina 624 samples, the target analytes reported must agree with the compound list found in the EPA 624 published method.

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8.3.2 Calibration Verification for EPA Method 8260C:

8.3.2.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) 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 (8.3.3) | in-house LCS limits |

8.3.2.2 Target Analytes

The curve must be verified initially by a calibration standard known as the Initial Calibration Verification standard (ICV). In addition, the same standard is analyzed every 12 hours for continuing calibration verification (CCV). A closing standard must also be analyzed at the end of the sequence for SM 6200. 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 or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large 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, the concentrations must be reported as estimated values.

8.3.3 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 |
| Chloroethane | Alcohols (Ethanol, TBA, TAA, ETBA, Butanol) |
| 1,3-Butadiene | Iodomethane. |
| 2,2-Dibromo-3-chloropropane | Naphthalene |
| 1- Methylnaphthalene | 2-Butanone |

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| | |
|-----------------------|----------------------|
| 2- Methyl-naphthalene | 2-Hexanone |
| Acetone | 4-Methyl-2-pentanone |
| Pentachloroethane | Cyclohexanone |
| Tert-butyl Formate | |

8.3.4 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 11.6.1, 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 Attachment VI for Method 624. No failures are acceptable for 8260 or 624, qualifiers cannot be used. Failures require a batch re-analysis. See Section 10 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, 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%.

8.3.5 Internal Standard Evaluation

8.3.5.1 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. 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.

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8.3.5.2 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 ICV. 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.

8.4 Gas chromatographic analysis:

8.4.1 Typical sequence order for loading the autosampler with calibration:

| Sample/QC Type | Use |
|---|---|
| Cleanup Blank | Verify system is contamination free |
| BFB Tune | Tuning criteria |
| Calibration standard(s) | Initial volatiles calibration and 7-point for GRO (if analyzed) |
| Second Source Calibration Verification (SSCV) | Verify initial calibration with second source. |
| Laboratory Control Sample/ Laboratory Control Sample Duplicate | 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 |

8.4.2 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 Control Sample Duplicate | 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 |

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| Sample/QC Type | Use |
|---|---|
| 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 |

8.5 GC/MS Analysis -- Water Samples

- 8.5.1 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. Three screening techniques that can be used are the headspace sampler, using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector (ELCD) (SW-846 Method 3810), extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (SW-846 Method 3820), and screening of 5mL of sample using an HNU or equivalent portable PID. See ESC SOP #330365, *Volatile Organic Compounds Screening using the RAE Systems Photoionization Gas Detector Model MiniRAE 3000*.
- 8.5.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.
- 8.5.3 Set up the GC/MS system as outlined in Section 8.1.
- 8.5.4 BFB tuning criteria and GC/MS calibration criteria must be met (Section 8.2.1) before analyzing samples.
- 8.5.5 Load the unopened VOA vial onto the autosampler for analysis.

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".

STATE NOTE: For West Virginia compliance samples, check for residual chlorine after sample analysis. Samples containing residual chlorine must be flagged as such. 8.5.6 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.

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8.5.7 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.

8.5.7.1 All vials for the sample are combined in an appropriate 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.

8.5.7.2 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.

8.5.7.3 Invert the volumetric flask 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.

8.5.8 Surrogate/Internal Standards – The autosampler adds 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution to 5mL of sample is equivalent of 40µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

8.5.9 Refer to SOP #330752, *Purge and Trap for Aqueous Samples* and SOP #330751, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples* for additional information on sample introduction.

8.5.10 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.

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8.6 GC/MS Analysis -- Water-miscible liquids

- 8.6.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with laboratory water.
- 8.6.2 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.
- 8.6.3 Alternatively, prepare dilutions directly in a clean 40mL vial filled with organic-free reagent water by adding at least 0.5 μ L, but not more than 25mLs of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 8.5.8.

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

- 8.7.1 Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1 mg/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 clean sand or clean soil.

STATE NOTE: This option cannot be used for South Carolina samples. Please refer to SOP #330751 that addresses Method 5035 for sample preparation.

- 8.7.1.1 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.
- 8.7.1.2 The GC/MS system must be set up as in Sections 8.1 and 8.2. This must be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. Both the initial and daily calibration standards (Sections 8.2 and 8.3) must be heated to 40°C purge temperature. Refer to Method 5035 (SOP #330751) for additional instructions for 8260B soil analysis.
- 8.7.1.3 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 in Section

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8.7.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

8.7.1.4 Add distilled 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 8.7.1.4 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.

8.7.2 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 is purged at ambient temperature. All samples with an expected concentration of >1.0 mg/Kg must be analyzed by this method.

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 8.7.2.1 or 8.7.2.2. 5035 must be used for all high-level soil samples, see SOP #330751.

8.7.2.1 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 5g (wet weight) of sample into a tared 40-mL 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 5g (wet weight) into a 40mL vial.

8.7.2.2 Add 5mL Methanol. Shake well for 2 minutes. See SOP #330751.

NOTE: Sections 8.7.2.1 and 8.7.2.2 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.

8.7.2.3 The GC/MS system must be set up as in Sections 8.1.

8.7.2.4 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 200µL.

8.7.2.5 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 1µL of the IS/surrogate mix to all of the samples.

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8.7.2.6 Proceed with the analysis as outlined in Sections 8.5.9-8.5.10. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks must also contain 200 μ L of the dilution solvent to simulate the sample conditions.

8.7.2.7 For a matrix spike in the high-concentration of sediment/soil samples, Add a 200 μ L aliquot of this extract to 5mL of organic-free reagent water for purging (as per Section 8.7.2.6) in a clean/baked 40mL VOA vial and add 20 μ L spiking solution, 1 μ L internal and surrogate standard solution (IS/Surr solution added by autosampler).

8.8 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.

- 8.8.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- 8.8.2 The RRT of the sample component is within + 0.06 RRT units of the RRT of the standard component.
- 8.8.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum the corresponding abundance in a sample spectrum can range between 20% and 80%.)
- 8.8.4 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.
- 8.8.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current

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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.

8.8.6 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 ESC.

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative 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 30% 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.

8.9 Quantitative Analysis

- 8.9.1 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

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described in Section 9. The internal standard used must be the one nearest the retention time of that of a given analyte.

- 8.9.2 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 ESC, 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.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 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.

9.2 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

9.2.1 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 (μL) (use 10,000 μL or a factor of this when dilutions are made).
 V_i = Volume of extract added (μL) 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.

9.2.2 Low-concentration procedure

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

where:

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

V_t = Volume of total extract (μL) (use 10,000μL or a factor of this when dilutions are made).

V_i = Volume of extract added (μL) 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.

9.4.3 Soil Weight determination with Methanol (samples received with MeOH).

$$\text{SoilWeight} = \text{VialTotalWeight(vial, soil, MeOH)} - \text{TareVialWeight} - \text{MeOHWeight.}$$

9.3 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_t. This total solvent/water volume is calculated as follows:

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

9.4 Percent Error (%Error)

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

where:

x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units

9.5 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in SOP #030205, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

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STATE NOTE: IDOCs for South Carolina DHEC compliance need to meet 70-130% for all compounds except the poor purgers. Poor purgers need to recover at 60-140%. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

METHOD NOTE: IDOCs for SM 6200B should have an average recovery of 80-120% for each target analyte and the RSD of the replicates should be <20%.

10.2 Batches:

10.2.1 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/Laboratory Control Sample Duplicate pair (LCS/LCSD), 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, an LCS/LCSD pair.

Additional instructions on Batch QC including required frequency and corrective actions can be found in Section 11 while acceptance criteria are found in the LIMS.

10.2.2 Analytical Batches (sequences):

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.

- 10.3 Perform BFB tune every 12 hours for 8260B, 8260C, and 6200B. BFB tuning for method 624 is every 24 hours. Tuning acceptance criteria are presented in Section 8.2.1. The computer software automatically evaluates the tune information. The analyst must be aware of the process used. 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. Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The scans must be averaged and background corrected. Average scans 0.1 minute before to 0.1 minute after the target peak including 2 scans and the peak apex.

STATE NOTE: BFB tuning must be performed every 12 hours when analyzing samples from South Carolina by Method 624.

- 10.4 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 8.2. Calibration is

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verified by analyzing Second Source Calibration Verification (SSCV) standard; acceptance criteria for the SSCV are presented in Section 8.3.1.

- 10.5 Run a mid-point Initial Calibration Verification (ICV) using the primary source standards every 12 hours before sample analysis. Also run a CCV every 12 hours during an analytical sequence for 8260B, 8260C, 624 and 6200B. For 6200B, a closing standard must also be analyzed at the end of the sequence. See sections 8.3.2 – 8.3.6 for acceptance criteria.
- 10.6 Retention Time Evaluation
- 10.6.1 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.
- 10.6.2 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.
- 10.7 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.
- 10.8 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.
- 10.9 LABORATORY CONTROL SAMPLES - Assess that LCSs were prepared at the required frequency of 1 per batch of 20. Routine LCS Control limits are presented in the LIMS.
- STATE NOTE:** SOUTH CAROLINA DHEC requires that ALL LCS compounds analyzed for compliance purposes meet the 70 – 130% acceptance criteria for all Method 8260 target analytes and the limits given in Attachment VI for Method 624 target analytes. All MS/MSD compounds must also meet the established criteria for all target analytes. No qualifiers are accepted. Samples analyzed in South Carolina for non-compliance testing utilize in house established acceptance limits to demonstrate control.
- 10.10 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.

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- 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.

- 10.11 **SURROGATE EVALUATION:** Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS, MSD, and MD. Acceptance criteria can be found in the LIMS: The surrogate recoveries for all QC samples must be within established control limits.
- 10.12 **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 ICV. 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 8.3.6
- 10.13 **SECOND SOURCE:** The second source calibration verification standard must be analyzed following each new initial calibration to verify the validity of the calibration standards. The recovery of the analytes in the SSCV 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 8.3.4 must recover within in-house calculated LCS recovery acceptance limits.

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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.

10.14 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*.

10.15 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) 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.

10.15.1 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).

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

10.15.3 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 3 years.

10.15.4 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.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 SITE-SPECIFIC requirements and STATE SPECIFIC criteria must be reviewed and used, if known, for data review.

11.2 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.

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- 11.3 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 8.0. 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 9.5). 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.

11.4 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.
- 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.

- 11.4.1 **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

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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.

- 11.5 LABORATORY CONTROL SAMPLES - If the recovery does not meet criteria, see section 11.6 for marginal failures. If it is still out of control limits, then all field and QC samples in the batch must be re-analyzed.

REQUIRED RE-ANALYSIS - None of the following compounds can recover beyond established criteria: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. If any of these compounds fail, the LCS/LCSD and all affected batch samples must be re-analyzed.

STATE NOTE: SOUTH CAROLINA DHEC Compliance LCS: responses must be within 70 – 130% for Method 8260 and within the limits given in Attachment VI for Method 624. No failures are acceptable; Qualifiers cannot be used. Failures require a batch re-analysis. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

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.

- 11.6 LCS/LCSD & MS/MSD CRITERIA

- 11.6.1 Quality control criteria must be checked for the LCS and LCSD.

LCS or LCSD 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 contains typical 90 analytes; therefore only five analyte can be considered as marginal exceedances. If the failure persists, re-prepare and re-analyze the entire sample batch.

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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.

11.6.2 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 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. 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

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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.

- 11.7 **MATRIX SPIKE ASSESSMENT:** If acceptance criteria are not met, perform the following corrective actions as appropriate.
- If both LCS/LCSD 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. Re-analyze to verify the matrix effect. If a matrix effect is still suspected, then the project manager must be contacted to discuss further alternatives and the potential impact on the project. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.

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.
- If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project.

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 and confirmed.

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

- 11.8 **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 surrogates 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 data with J1 (outside upper limit) or J2 (outside lower limit). When one or two surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).

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- 11.9 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 8.3.6, 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 criteria 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 criteria 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).
- 11.10 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.
- 11.11 SECOND SOURCE: 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.
- 11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 11.12.1 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.
- 11.12.1.1 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.
- 11.12.2 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.
- 11.12.3 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".

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- 11.13 Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in SOP #030215, *Manual Integration Procedure*. "Before" and "After" quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.
- 11.14 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.
- 11.14.1 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.
- 11.14.2 All tentatively identified compounds (TICs) are reported with a "J" qualifier for estimated value and an "N" for presumptive evidence of material.
- 11.15 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.
- 12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT
- 12.1 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 *ESC Waste Management Plan*.
- 12.2 See SOP #030302, *Environmental Sustainability & Pollution Prevention*.
- 13.0 METHOD MODIFICATIONS/CLARIFICATIONS
- 13.1 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.
- 13.2 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.
- 13.3 Heated purge may be used for all samples regardless of matrix or method.
- 14.0 REFERENCES
- 14.1 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, December 1996.
- 14.2 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003.
- 14.3 *Determinative Chromatographic Separations*, SW846 Method 8000D, Revision 4, July 2014.

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- 14.4 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260B, Revision 2, December 1996.*
- 14.5 *Purgeables, 40 CFR 136, EPA Method 624*
- 14.6 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method, SM 6200B, 20th edition.*
- 14.7 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference
- 14.8 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260C, Revision 3, August 2006.*
- 14.9 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method, SM 6200B-2011.*

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Attachment I: Revision History

Current Version:

| Version | Date | Description of Revisions |
|---------|------------|--|
| 23 | 10/19/2016 | Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.0, 2.3, 2.4, 4.2, 4.3, 5.3.4, 5.5, 6.1, 6.6.4, 6.8, 7.2, 7.5.1.1, 7.5.2.1, 8.2.3.1, 8.2.4.1, 8.2.7.3, 8.2.8.2, 8.5.5, 8.5.6, 8.5.7.1, 8.5.7.3, 8.6.3, 8.7.2.4, 8.7.2.6, 8.7.2.7, 9.1, 9.2, 9.3, 9.5, 10.2.2, 10.9, 10.10, 11.3, 11.13, 14.1, 14.4, 14.5, 14.6, 14.8, 14.9, Attachment V, Attachment VIII, and Attachment IX Table 2. Added Sections 1.3.2, 9.4, 10.15, 10.15.1, 10.15.2, 10.15.3, 10.15.4, 11.4.1, 11.6.2, 11.15, 13.3, 14.2, and 14.3. Deleted Sections 2.4 through 2.17, 8.5.7.4, 8.5.7.5, 9.1, 9.2, 9.5, 9.6, 9.7, and 9.8. |

Superseded Versions:

This document supersedes the following:

| Version | Date | Description of Revisions |
|---------|----------|--|
| 0 | 8/23/94 | Origination |
| 1 | 7/25/95 | |
| 2 | 9/12/97 | |
| 3 | 8/4/98 | |
| 4 | 2/11/00 | |
| 5 | 8/21/00 | |
| 6 | 4/1/01 | |
| 7 | 10/16/01 | |
| 8 | 8/19/02 | |
| 9 | 7/23/03 | |
| 10 | 10/30/03 | |
| 11 | 1/26/04 | |
| 12 | 6/28/04 | |
| 13 | 12/11/04 | |
| 14 | 3/23/05 | |
| 15 | 12/19/07 | |
| 16 | 1/30/09 | Technical and Quality Review and update. Updated Table 1; Clarified holding times; Updated Note in Section 7; Updated section 7.4.1 & 7.4.2; Updated section 7.6, 7.8.4, 8.1.2, 8.2.3, 8.2.3.2, 8.2.4.1, 8.2.6.2 & 12.0; Added state note (MN) section 8.2.2, added final bullet item, added state note (OH); Added section 8.2.6.4; section 9.11 (state note), section 11.1 (state note), section 11.7.1 (state note), section 11.13, & 13.1; Ohio VAP approved 1/30/09 |

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| Version | Date | Description of Revisions |
|---------|------------|---|
| 17 | 3/12/12 | Technical and Quality Review and update. Added Attachment VIII and sections 2.15 through 2.17, 8.2.7, 8.3.2, 9.10, 10.8 through 10.13, 13.2, 14.5, 14.6, state note in section 1.0, and client note in section 11.11; Revised Attachments II through IV, VII and sections 1.3, 3.1, 4.1, 4.2, 4.3, 7.1, 7.5.1, 7.5.2, 7.9.3, 8.2.2, 8.2.3, 8.2.6, 8.3.1, 8.3.3, 8.3.4, 8.7.2, 9.1, 9.2, 9.6 through 9.8, 10.2, 10.3, 10.5, and 12.1; Removed section 9.3. |
| 18 | 12/11/13 | Technical and Quality Review and update. Added state notes in sections 1.0, 10.1, 10.3 and sections 10.14 and 14.7; Revised state notes in sections 1.0, 4.2 and sections 5.5, 6.1, 6.4, 7.1, 7.2, 7.5.1, 7.5.2, 7.6, 7.7, 8.2.4, 8.2.7.3, 8.3.1.4, 8.3.2.2, 8.5.1 and 10.5; Revised Attachments II, IV, VIII; Removed state notes pertaining to Ohio VAP throughout and sections 8.2.3.3 and 11.14.1. |
| 19 | 2/13/15 | Technical and Quality Review and update. Revised all state notes regarding South Carolina sample analyses; Revised sections 7.5.1, 7.5.2 and 8.3.3; Revised Attachments VIII. |
| 20 | 9/2/2015 | Header and signature block re-formatting. Technical and Quality Review and update. Added Section 9.11 and Attachment IX. Revised Sections 10.2.1, 10.6, 10.6.1, 10.6.2, 10.9, 10.10, 10.11, and 11.7. Deleted Sections 10.6.3 and 10.6.4. Revised footnote in Attachment II. Replaced Attachment VIII. |
| 21 | 10/29/2015 | Technical and quality review and update. Revised Sections 8.3.4, 10.8, and 11.4. Added State Note in Sections 8.2.1 and 10.3. |
| 22 | 11/17/2015 | Technical and quality review and update. Revised Sections 1.3.1, 7.1, 8.3.4, 8.5.9, 10.9, 11.5, and 12.2. |

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Attachment II: 8260/624/6200B Reporting Limits and Common Analyte List

| Compound | Water | | Low Soil | | High Soil | |
|-----------------------------|--------|-------|----------|-------|-----------|-------|
| | RL | Units | RL* | Units | RL | Units |
| Acetone | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Acrolein | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Acrylonitrile | 0.010 | mg/L | 0.010 | 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 |
| 1,3-Butadiene | 0.0025 | mg/L | 0.0025 | mg/Kg | 0.125 | mg/Kg |
| n-Butylbenzene | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| sec-Butylbenzene | 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 |
| 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 |
| 2-Chloroethyl vinyl ether | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Chloroform | 0.002 | mg/L | 0.002 | mg/Kg | 0.10 | mg/Kg |
| Chloromethane | 0.0025 | mg/L | 0.001 | mg/Kg | 0.05 | 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 |
| 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 |
| Dibromomethane | 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 |
| 1,3-Dichlorobenzene | 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 |
| Dichlorodifluoromethane | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| 1,1-Dichloroethane | 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 |
| 1,1-Dichloroethene | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| cis-1,2-Dichloroethene | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| trans-1,2-Dichloroethene | 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 |
| 1,1-Dichloropropene | 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 |

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| Compound | Water | | Low Soil | | High Soil | |
|-----------------------------|--------|-------|----------|-------|-----------|-------|
| | RL | Units | RL* | Units | RL | Units |
| cis-1,3-Dichloropropene | 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 |
| 2,2-Dichloropropane | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | 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 |
| 4-Ethyltoluene | 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 | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| Isopropylbenzene | 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 |
| 2,2,4-Trimethyl Pentane | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| 2-Butanone (MEK) | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| Methylene Chloride | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| 4-Methyl-2-pentanone (MIBK) | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| Methyl tert-butyl ether | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| Naphthalene | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| n-Propylbenzene | 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 |
| 1,1,1,2-Tetrachloroethane | 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 |
| Tetrachloroethene | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | mg/Kg |
| Toluene | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| 1,2,3-Trichlorobenzene | 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 |
| 1,1,1-Trichloroethane | 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 |
| 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 |
| 1,2,3-Trichloropropane | 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 |
| 1,3,5-Trimethylbenzene | 0.001 | mg/L | 0.001 | mg/Kg | 0.05 | 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 |
| Additional Compounds | | | | | | |
| Acetonitrile | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Allyl Chloride | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Chloroprene | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Compound | Water | | Low Soil | | High Soil | |
|-----------------------------|--------|-------|----------|-------|-----------|-------|
| | RL | Units | RL* | Units | RL | Units |
| Trans-1,4-Dichloro-2-butene | 0.0025 | mg/L | 0.0025 | mg/Kg | 0.125 | mg/Kg |
| Isobutanol | 0.100 | mg/L | 0.100 | mg/Kg | 5.0 | mg/Kg |
| 1,4-Dioxane+ | 0.100 | mg/L | 0.100 | mg/Kg | 5.0 | mg/Kg |
| Methacrylonitrile | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Methyl Methacrylate | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Ethyl methacrylate | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Propionitrile | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Pentachloroethane | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Carbon Disulfide | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Cyclohexanone | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| 2-Hexanone | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| Iodomethane | 0.010 | mg/L | 0.010 | mg/Kg | 0.50 | mg/Kg |
| Isobutanol | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Propionitrile | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Vinyl Acetate | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Tetrahydrofuran | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Bromoethane | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| 2-Butanol | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Ethanol | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Tert-Butyl Alcohol | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Di-isopropyl ether | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Ethyl tert-butyl ether | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Methyl-tert-butyl ether | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Tert-Butyl alcohol | 0.050 | mg/L | 0.050 | mg/Kg | 2.5 | mg/Kg |
| Tert-Amyl Methyl Ether | 0.001 | mg/L | 0.001 | mg/Kg | 0.050 | mg/Kg |
| Tert-Butyl Formate | 0.020 | mg/L | 0.020 | mg/Kg | 1.0 | mg/Kg |
| Tert Butyl Ethyl Alcohol | 0.100 | mg/L | 0.100 | mg/Kg | 5.0 | mg/Kg |
| Tert Amyl Alcohol | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| Dichlorofluoromethane | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |
| 2-Propanol | 0.005 | mg/L | 0.005 | mg/Kg | 0.25 | mg/Kg |

RLs are based on a 5mL purge volume

Low Soil - Using a 5g soil sample to 5mL water – See Method 5035 (SOP #330751) Section 8.2.4.1

High Soil – Using 200uL extract from 5g. soil sample to 5mL methanol; see Method 5035 (SOP #330751) Sect. 8.3.1.2

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

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

**Attachment III: 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 | 75 | 155, 157 |
| Dibromochloromethane | 129 | 127 |
| 1,2-Dibromoethane | 107 | 109, 188 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Compound | Primary Characteristic | Secondary Characteristic |
|-----------------------------|------------------------|--------------------------|
| | Ion | Ion(s) |
| Dibromomethane | 93 | 95, 174 |
| 1,2-Dichlorobenzene | 146 | 111, 148 |
| 1,2-Dichlorobenzene-d4 | 152 | 115, 150 |
| 1,3-Dichlorobenzene | 146 | 111, 148 |
| 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 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Compound | Primary Characteristic | Secondary Characteristic |
|---------------------------------------|------------------------|--------------------------|
| | Ion | Ion(s) |
| 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 |
| 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-Trichlorobenzene | 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 |
| Internal Standards/Surrogates: | | |
| 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 | |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Compound | Primary Characteristic Ion | Secondary Characteristic Ion(s) |
|--------------------|----------------------------|---------------------------------|
| 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).

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment IV: 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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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment V: 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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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment VI: EPA 624 CCC Criteria:

TABLE 5—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624^a

| Parameter | Range for Q ($\mu\text{g/L}$) | Limit for s ($\mu\text{g/L}$) | Range for X ($\mu\text{g/L}$) | Range for P, P _r (%) |
|---------------------------|------------------------------------|---------------------------------------|------------------------------------|------------------------------------|
| Benzene | 12.8–27.2 | 6.9 | 15.2–26.0 | 37–151 |
| Bromodichloromethane | 13.1–26.9 | 6.4 | 10.1–28.0 | 35–155 |
| Bromoform | 14.2–25.8 | 5.4 | 11.4–31.1 | 45–169 |
| Bromomethane | 2.8–37.2 | 17.9 | D–41.2 | D–242 |
| Carbon tetrachloride | 14.6–25.4 | 5.2 | 17.2–23.5 | 70–140 |
| Chlorobenzene | 13.2–26.8 | 6.3 | 16.4–27.4 | 37–160 |
| Chloroethane | 7.6–32.4 | 11.4 | 8.4–40.4 | 14–230 |
| 2-Chloroethylvinyl ether | D–44.8 | 25.9 | D–50.4 | D–305 |
| Chloroform | 13.5–26.5 | 6.1 | 13.7–24.2 | 51–138 |
| Chloromethane | D–40.8 | 19.8 | D–45.9 | D–273 |
| Dibromochloromethane | 13.5–26.5 | 6.1 | 13.8–26.6 | 53–149 |
| 1,2-Dichlorobenzene | 12.6–27.4 | 7.1 | 11.8–34.7 | 18–190 |
| 1,3-Dichlorobenzene | 14.6–25.4 | 5.5 | 17.0–28.8 | 59–156 |
| 1,4-Dichlorobenzene | 12.6–27.4 | 7.1 | 11.8–34.7 | 18–190 |
| 1,1-Dichloroethane | 14.5–25.5 | 5.1 | 14.2–28.5 | 59–155 |
| 1,2-Dichloroethane | 13.6–26.4 | 6.0 | 14.3–27.4 | 49–155 |
| 1,1-Dichloroethene | 10.1–29.9 | 9.1 | 3.7–42.3 | D–234 |
| trans-1,2-Dichloroethene | 13.9–26.1 | 5.7 | 13.6–28.5 | 54–156 |
| 1,2-Dichloropropane | 6.8–33.2 | 13.8 | 3.8–36.2 | D–210 |
| cis-1,3-Dichloropropene | 4.8–35.2 | 15.8 | 1.0–39.0 | D–227 |
| trans-1,3-Dichloropropene | 10.0–30.0 | 10.4 | 7.6–32.4 | 17–183 |
| Ethyl benzene | 11.8–28.2 | 7.5 | 17.4–26.7 | 37–162 |
| Methylene chloride | 12.1–27.9 | 7.4 | D–41.0 | D–221 |
| 1,1,2,2-Tetrachloroethane | 12.1–27.9 | 7.4 | 13.5–27.2 | 46–157 |
| Tetrachloroethene | 14.7–25.3 | 5.0 | 17.0–26.6 | 64–148 |
| Toluene | 14.9–25.1 | 4.8 | 16.6–26.7 | 47–150 |
| 1,1,1-Trichloroethane | 15.0–25.0 | 4.6 | 13.7–30.1 | 52–162 |
| 1,1,2-Trichloroethane | 14.2–25.8 | 5.5 | 14.3–27.1 | 52–150 |
| Trichloroethene | 13.3–26.7 | 6.6 | 18.6–27.6 | 71–157 |
| Trichlorofluoromethane | 9.6–30.4 | 10.0 | 8.9–31.5 | 17–181 |
| Vinyl chloride | 0.8–39.2 | 20.0 | D–43.5 | D–251 |

Q= Concentration measured in QC check sample, in $\mu\text{g/L}$ (Section 7.5.3).

s= Standard deviation of four recovery measurements, in $\mu\text{g/L}$ (Section 8.2.4).

X= Average recovery of four recovery measurements, in $\mu\text{g/L}$ (Section 8.2.4).

P, P_r= Percent recovery measured, (Section 8.3.2, Section 8.4.2).

D= Detected; result must be greater than zero.

^aCriteria were calculated assuming a QC check sample concentration of 20 $\mu\text{g/L}$.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment VII: 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-Triichlorobenzene | 0.200 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment VIII: 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-TRIMETHYLBENZENE | METHYLENE CHLORIDE |
| 1,3-BUTADIENE | NAPHTHALENE |
| 1,3-DICHLOROBENZENE | N-BUTYLBENZENE |
| 1,3-DICHLOROPROPANE | N-HEXANE |
| 1,4-DICHLOROBENZENE | N-PROPYLBENZENE |
| 1-METHYLNAPHTHALENE | O-XYLENE |
| 2,2,4-TRIMETHYLPENTANE | P-ISOPROPYLTOLUENE |
| 2,2-DICHLOROPROPANE | PROPENE |
| 2-BUTANONE (MEK) | SEC-BUTYLBENZENE |
| 2-CHLOROETHYL VINYL ETHER | STYRENE |
| 2-CHLOROTOLUENE | TERT-BUTYLBENZENE |
| 2-HEXANONE | TETRACHLOROETHENE |
| 2-METHYLNAPHTHALENE | TETRAHYDROFURAN |
| 4-CHLOROTOLUENE | TOLUENE |
| 4-ETHYLTOLUENE | TPH (GC/MS) LOW FRACTION |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

STANDARD ANALYTE LIST

4-METHYL-2-PENTANONE (MIBK)

ACETONE

ACROLEIN*

ACRYLONITRILE*

BENZENE

BROMOBENZENE

BROMOCHLOROMETHANE

BROMODICHLOROMETHANE

BROMOFORM

BROMOMETHANE

CARBON DISULFIDE

CARBON TETRACHLORIDE

CHLOROBENZENE

TRANS-1,2-DICHLOROETHENE

TRANS-1,3-DICHLOROPROPENE

TRANS-1,4-DICHLORO-2-BUTENE

TRICHLOROETHENE

TRICHLOROFLUOROMETHANE

VINYL ACETATE

VINYL CHLORIDE

XYLENES, TOTAL

SURROGATE LIMITS

4-BROMOFLUOROBENZENE

A,A,A-TRIFLUOROTOLUENE

DIBROMOFLUOROMETHANE

TOLUENE-D8

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Attachment IX: 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

| Table 1. GCMS Troubleshooting Guide | | |
|--|--|---|
| Problem | Cause | Treatment |
| 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 |

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| Table 1. GCMS Troubleshooting Guide | | |
|--|--|---|
| Problem | Cause | Treatment |
| Poor chromatographic resolution | Column temperature or program not optimized | Modify method by changing temperature ramp segment slopes |
| | 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.

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- 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.
- 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:

| Table 2. Support Equipment Checks | | |
|---|--|---|
| Performance Check | Frequency | Acceptance Criteria |
| 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°C to 6°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) |

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Table 2. Support Equipment Checks

| Performance Check | Frequency | Acceptance Criteria |
|--|---|--|
| 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] |
| 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.

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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 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 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.

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- 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 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 Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum 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 Addendum 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:

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- 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 1/0th 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.

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)
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 |

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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 |
|------------|------------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 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 |
| 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 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 3. LCS Control Limits – Method 8260 Solid Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|-------------|------------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 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 |
| 75-43-4 | 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 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 3. LCS Control Limits – Method 8260 Solid Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|------------|-----------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 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 |
| 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 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 3. LCS Control Limits – Method 8260 Solid Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|------------|-----------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 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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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|------------|---|-----------|-------|--------------------|---------------------|---------------------|
| 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 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|----------|------------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 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 |
| 460-00-4 | 4-Bromofluorobenzene | 9971 | 99.7 | 4.9 | 85 | 114 |
| 106-43-4 | 4-Chlorotoluene | 23616 | 99.9 | 7.4 | 78 | 122 |
| 108-10-1 | 4-Methyl-2-pentanone [MIBK] | 25796 | 98.5 | 10.6 | 67 | 130 |
| 67-64-1 | Acetone | 25006 | 99.5 | 20.1 | 39 | 160 |
| 75-05-8 | Acetonitrile | 13308 | 95.8 | 15.2 | 50 | 142 |
| 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 |
| 460-00-4 | 4-Bromofluorobenzene | 9971 | 99.7 | 4.9 | 85 | 114 |
| 106-43-4 | 4-Chlorotoluene | 23616 | 99.9 | 7.4 | 78 | 122 |
| 108-10-1 | 4-Methyl-2-pentanone [MIBK] | 25796 | 98.5 | 10.6 | 67 | 130 |
| 67-64-1 | Acetone | 25006 | 99.5 | 20.1 | 39 | 160 |
| 75-05-8 | Acetonitrile | 13308 | 95.8 | 15.2 | 50 | 142 |
| 107-02-8 | Acrolein [Propenal] | 16380 | 96.8 | 19.3 | 39 | 155 |
| 107-13-1 | Acrylonitrile | 20173 | 99 | 11.9 | 63 | 135 |
| 107-05-1 | Allyl chloride | 15758 | 99 | 10.4 | 68 | 130 |

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|------------|------------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 71-43-2 | Benzene | 34376 | 99.4 | 6.9 | 79 | 120 |
| 100-44-7 | Benzyl chloride | 10675 | 90.1 | 15.9 | 42 | 138 |
| 108-86-1 | Bromobenzene | 23762 | 99.7 | 6.7 | 80 | 120 |
| 74-97-5 | Bromochloromethane | 24356 | 100.8 | 7.5 | 78 | 123 |
| 75-27-4 | Bromodichloromethane | 26888 | 101.8 | 7.8 | 79 | 125 |
| 75-25-2 | Bromoform | 27675 | 97.8 | 10.8 | 66 | 130 |
| 74-83-9 | Bromomethane | 26717 | 97 | 14.7 | 53 | 141 |
| 75-15-0 | Carbon disulfide | 25719 | 98.8 | 11.5 | 64 | 133 |
| 56-23-5 | Carbon tetrachloride | 28870 | 103.8 | 10.7 | 72 | 136 |
| 108-90-7 | Chlorobenzene | 29802 | 100 | 6.1 | 82 | 118 |
| 124-48-1 | Chlorodibromomethane | 27424 | 100 | 8.5 | 74 | 126 |
| 75-45-6 | Chlorodifluoromethane | 7197 | 84.4 | 14.9 | 40 | 129 |
| 75-00-3 | Chloroethane | 27069 | 99 | 13 | 60 | 138 |
| 67-66-3 | Chloroform | 29373 | 101.1 | 7.5 | 79 | 124 |
| 74-87-3 | Chloromethane | 27697 | 94.5 | 15 | 50 | 139 |
| 156-59-2 | cis-1,2-Dichloroethene | 27935 | 100.1 | 7.5 | 78 | 123 |
| 10061-01-5 | cis-1,3-Dichloropropene | 27197 | 99.5 | 8 | 75 | 124 |
| 1476-11-5 | cis-1,4-Dichloro-2-butene | 1524 | 101.5 | 14.9 | 57 | 146 |
| 110-82-7 | Cyclohexane | 20438 | 100.4 | 10 | 71 | 130 |
| 1868-53-7 | Dibromofluoromethane | 5702 | 99.1 | 6.5 | 80 | 119 |
| 74-95-3 | Dibromomethane | 24473 | 101.1 | 7.3 | 79 | 123 |
| 75-71-8 | Dichlorodifluoromethane [Freon-12] | 25410 | 92 | 20.1 | 32 | 152 |
| 107-02-8 | Acrolein [Propenal] | 16380 | 96.8 | 19.3 | 39 | 155 |
| 107-13-1 | Acrylonitrile | 20173 | 99 | 11.9 | 63 | 135 |
| 107-05-1 | Allyl chloride | 15758 | 99 | 10.4 | 68 | 130 |
| 71-43-2 | Benzene | 34376 | 99.4 | 6.9 | 79 | 120 |
| 100-44-7 | Benzyl chloride | 10675 | 90.1 | 15.9 | 42 | 138 |
| 108-86-1 | Bromobenzene | 23762 | 99.7 | 6.7 | 80 | 120 |
| 74-97-5 | Bromochloromethane | 24356 | 100.8 | 7.5 | 78 | 123 |
| 75-27-4 | Bromodichloromethane | 26888 | 101.8 | 7.8 | 79 | 125 |
| 75-25-2 | Bromoform | 27675 | 97.8 | 10.8 | 66 | 130 |
| 74-83-9 | Bromomethane | 26717 | 97 | 14.7 | 53 | 141 |
| 75-15-0 | Carbon disulfide | 25719 | 98.8 | 11.5 | 64 | 133 |
| 56-23-5 | Carbon tetrachloride | 28870 | 103.8 | 10.7 | 72 | 136 |
| 108-90-7 | Chlorobenzene | 29802 | 100 | 6.1 | 82 | 118 |
| 124-48-1 | Chlorodibromomethane | 27424 | 100 | 8.5 | 74 | 126 |
| 75-45-6 | Chlorodifluoromethane | 7197 | 84.4 | 14.9 | 40 | 129 |
| 75-00-3 | Chloroethane | 27069 | 99 | 13 | 60 | 138 |
| 67-66-3 | Chloroform | 29373 | 101.1 | 7.5 | 79 | 124 |
| 74-87-3 | Chloromethane | 27697 | 94.5 | 15 | 50 | 139 |

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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|-------------|------------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 156-59-2 | cis-1,2-Dichloroethene | 27935 | 100.1 | 7.5 | 78 | 123 |
| 10061-01-5 | cis-1,3-Dichloropropene | 27197 | 99.5 | 8 | 75 | 124 |
| 1476-11-5 | cis-1,4-Dichloro-2-butene | 1524 | 101.5 | 14.9 | 57 | 146 |
| 110-82-7 | Cyclohexane | 20438 | 100.4 | 10 | 71 | 130 |
| 1868-53-7 | Dibromofluoromethane | 5702 | 99.1 | 6.5 | 80 | 119 |
| 74-95-3 | Dibromomethane | 24473 | 101.1 | 7.3 | 79 | 123 |
| 75-71-8 | Dichlorodifluoromethane [Freon-12] | 25410 | 92 | 20.1 | 32 | 152 |
| 75-43-4 | Dichlorofluoromethane | 1504 | 101.5 | 9.8 | 72 | 131 |
| 60-29-7 | Diethyl ether | 17189 | 98.6 | 10.2 | 68 | 129 |
| 108-20-3 | Diisopropyl ether | 22989 | 97.5 | 10.3 | 67 | 128 |
| 64-17-5 | Ethanol | 9543 | 99.2 | 17.1 | 48 | 151 |
| 141-78-6 | Ethyl acetate | 9208 | 96.8 | 13.9 | 55 | 138 |
| 97-63-2 | Ethyl methacrylate | 16674 | 98.7 | 9 | 72 | 126 |
| 637-92-3 | Ethyl tert-butyl ether | 19841 | 98.3 | 9.4 | 70 | 127 |
| 100-41-4 | Ethylbenzene | 33325 | 99.8 | 7 | 79 | 121 |
| 462-06-6 | Fluorobenzene | 1373 | 97.9 | 6.1 | 80 | 116 |
| 142-82-5 | Heptane | 11878 | 94.4 | 15 | 49 | 140 |
| 87-68-3 | Hexachlorobutadiene | 23535 | 100.1 | 11.3 | 66 | 134 |
| 67-72-1 | Hexachloroethane | 8718 | 102.9 | 10.3 | 72 | 134 |
| 110-54-3 | Hexane | 15545 | 95.5 | 15.9 | 48 | 143 |
| 74-88-4 | Iodomethane | 20229 | 100 | 10.4 | 69 | 131 |
| 78-83-1 | Isobutyl alcohol | 14123 | 97.7 | 11.7 | 63 | 133 |
| 108-21-4 | Isopropyl acetate [Acetic acid] | 7216 | 97.8 | 11.6 | 63 | 133 |
| 98-82-8 | Isopropylbenzene | 28636 | 101.5 | 9.9 | 72 | 131 |
| 179601-23-1 | m/p-Xylene [3/4-Xylene] | 28168 | 100.5 | 6.9 | 80 | 121 |
| 126-98-7 | Methacrylonitrile | 15982 | 97.9 | 11.6 | 63 | 133 |
| 79-20-9 | Methyl acetate | 19698 | 96 | 13.2 | 56 | 136 |
| 80-62-6 | Methyl methacrylate | 16524 | 97.7 | 10.2 | 67 | 128 |
| 1634-04-4 | Methyl tert-butyl ether [MTBE] | 29660 | 97.3 | 8.8 | 71 | 124 |
| 108-87-2 | Methylcyclohexane | 20025 | 101.8 | 10.1 | 72 | 132 |
| 75-09-2 | Methylene chloride | 27659 | 99.4 | 8.3 | 74 | 124 |
| 123-86-4 | n-Butyl acetate | 7247 | 96.8 | 9.4 | 69 | 125 |
| 75-43-4 | Dichlorofluoromethane | 1504 | 101.5 | 9.8 | 72 | 131 |
| 60-29-7 | Diethyl ether | 17189 | 98.6 | 10.2 | 68 | 129 |
| 108-20-3 | Diisopropyl ether | 22989 | 97.5 | 10.3 | 67 | 128 |
| 64-17-5 | Ethanol | 9543 | 99.2 | 17.1 | 48 | 151 |
| 141-78-6 | Ethyl acetate | 9208 | 96.8 | 13.9 | 55 | 138 |
| 97-63-2 | Ethyl methacrylate | 16674 | 98.7 | 9 | 72 | 126 |
| 637-92-3 | Ethyl tert-butyl ether | 19841 | 98.3 | 9.4 | 70 | 127 |
| 100-41-4 | Ethylbenzene | 33325 | 99.8 | 7 | 79 | 121 |

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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|-------------|---------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 462-06-6 | Fluorobenzene | 1373 | 97.9 | 6.1 | 80 | 116 |
| 142-82-5 | Heptane | 11878 | 94.4 | 15 | 49 | 140 |
| 87-68-3 | Hexachlorobutadiene | 23535 | 100.1 | 11.3 | 66 | 134 |
| 67-72-1 | Hexachloroethane | 8718 | 102.9 | 10.3 | 72 | 134 |
| 110-54-3 | Hexane | 15545 | 95.5 | 15.9 | 48 | 143 |
| 74-88-4 | Iodomethane | 20229 | 100 | 10.4 | 69 | 131 |
| 78-83-1 | Isobutyl alcohol | 14123 | 97.7 | 11.7 | 63 | 133 |
| 108-21-4 | Isopropyl acetate [Acetic acid] | 7216 | 97.8 | 11.6 | 63 | 133 |
| 98-82-8 | Isopropylbenzene | 28636 | 101.5 | 9.9 | 72 | 131 |
| 179601-23-1 | m/p-Xylene [3/4-Xylene] | 28168 | 100.5 | 6.9 | 80 | 121 |
| 126-98-7 | Methacrylonitrile | 15982 | 97.9 | 11.6 | 63 | 133 |
| 79-20-9 | Methyl acetate | 19698 | 96 | 13.2 | 56 | 136 |
| 80-62-6 | Methyl methacrylate | 16524 | 97.7 | 10.2 | 67 | 128 |
| 1634-04-4 | Methyl tert-butyl ether [MTBE] | 29660 | 97.3 | 8.8 | 71 | 124 |
| 108-87-2 | Methylcyclohexane | 20025 | 101.8 | 10.1 | 72 | 132 |
| 75-09-2 | Methylene chloride | 27659 | 99.4 | 8.3 | 74 | 124 |
| 123-86-4 | n-Butyl acetate | 7247 | 96.8 | 9.4 | 69 | 125 |
| 71-36-3 | n-Butyl alcohol | 10122 | 95.1 | 12 | 59 | 131 |
| 104-51-8 | n-Butylbenzene | 24088 | 101.1 | 8.8 | 75 | 128 |
| 109-60-4 | n-Propyl acetate | 602 | 100.8 | 8.3 | 76 | 126 |
| 103-65-1 | n-Propylbenzene | 24419 | 101 | 8.5 | 76 | 126 |
| 91-20-3 | Naphthalene | 27847 | 94.6 | 11.3 | 61 | 128 |
| 95-47-6 | o-Xylene | 31776 | 100 | 7.2 | 78 | 122 |
| 99-87-6 | p-Isopropyltoluene [p-Cymene] | 24335 | 102 | 8.5 | 77 | 127 |
| 76-01-7 | Pentachloroethane | 11688 | 101.1 | 10.7 | 69 | 133 |
| 109-66-0 | Pentane | 3915 | 74.8 | 19.7 | 16 | 134 |
| 107-12-0 | Propionitrile [Ethyl cyanide] | 15701 | 99.9 | 12 | 64 | 136 |
| 135-98-8 | sec-Butylbenzene | 24191 | 101.1 | 8.1 | 77 | 126 |
| 100-42-5 | Styrene | 26985 | 100.5 | 7.6 | 78 | 123 |
| 994-05-8 | tert-Amyl methyl ether [TAME] | 19726 | 98.1 | 10.1 | 68 | 128 |
| 75-65-0 | tert-Butyl alcohol | 21112 | 98.6 | 10.1 | 68 | 129 |
| 762-75-4 | tert-Butyl formate | 6651 | 98.1 | 11.1 | 65 | 132 |
| 98-06-6 | tert-Butylbenzene | 23919 | 101 | 7.7 | 78 | 124 |
| 127-18-4 | Tetrachloroethene | 29017 | 101.3 | 9.3 | 74 | 129 |
| 109-99-9 | Tetrahydrofuran | 18021 | 95 | 12.8 | 57 | 133 |
| 108-88-3 | Toluene | 33510 | 100.1 | 6.8 | 80 | 121 |
| 2037-26-5 | Toluene-d8 | 9809 | 100.4 | 3.8 | 89 | 112 |
| 156-60-5 | trans-1,2-Dichloroethene | 27663 | 99.5 | 8.2 | 75 | 124 |
| 10061-02-6 | trans-1,3-Dichloropropene | 27134 | 100 | 8.9 | 73 | 127 |

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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 4. LCS Control Limits – Method 8260 Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|------------|-----------------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 110-57-6 | trans-1,4-Dichloro-2-butene | 19320 | 91.5 | 16.1 | 43 | 140 |
| 79-01-6 | Trichloroethene | 30150 | 101.1 | 7.3 | 79 | 123 |
| 75-69-4 | Trichlorofluoromethane | 26108 | 103 | 12.8 | 65 | 141 |
| 71-36-3 | n-Butyl alcohol | 10122 | 95.1 | 12 | 59 | 131 |
| 104-51-8 | n-Butylbenzene | 24088 | 101.1 | 8.8 | 75 | 128 |
| 109-60-4 | n-Propyl acetate | 602 | 100.8 | 8.3 | 76 | 126 |
| 103-65-1 | n-Propylbenzene | 24419 | 101 | 8.5 | 76 | 126 |
| 91-20-3 | Naphthalene | 27847 | 94.6 | 11.3 | 61 | 128 |
| 95-47-6 | o-Xylene | 31776 | 100 | 7.2 | 78 | 122 |
| 99-87-6 | p-Isopropyltoluene [p-Cymene] | 24335 | 102 | 8.5 | 77 | 127 |
| 76-01-7 | Pentachloroethane | 11688 | 101.1 | 10.7 | 69 | 133 |
| 109-66-0 | Pentane | 3915 | 74.8 | 19.7 | 16 | 134 |
| 107-12-0 | Propionitrile [Ethyl cyanide] | 15701 | 99.9 | 12 | 64 | 136 |
| 135-98-8 | sec-Butylbenzene | 24191 | 101.1 | 8.1 | 77 | 126 |
| 100-42-5 | Styrene | 26985 | 100.5 | 7.6 | 78 | 123 |
| 994-05-8 | tert-Amyl methyl ether [TAME] | 19726 | 98.1 | 10.1 | 68 | 128 |
| 75-65-0 | tert-Butyl alcohol | 21112 | 98.6 | 10.1 | 68 | 129 |
| 762-75-4 | tert-Butyl formate | 6651 | 98.1 | 11.1 | 65 | 132 |
| 98-06-6 | tert-Butylbenzene | 23919 | 101 | 7.7 | 78 | 124 |
| 127-18-4 | Tetrachloroethene | 29017 | 101.3 | 9.3 | 74 | 129 |
| 109-99-9 | Tetrahydrofuran | 18021 | 95 | 12.8 | 57 | 133 |
| 108-88-3 | Toluene | 33510 | 100.1 | 6.8 | 80 | 121 |
| 2037-26-5 | Toluene-d8 | 9809 | 100.4 | 3.8 | 89 | 112 |
| 156-60-5 | trans-1,2-Dichloroethene | 27663 | 99.5 | 8.2 | 75 | 124 |
| 10061-02-6 | trans-1,3-Dichloropropene | 27134 | 100 | 8.9 | 73 | 127 |
| 110-57-6 | trans-1,4-Dichloro-2-butene | 19320 | 91.5 | 16.1 | 43 | 140 |
| 79-01-6 | Trichloroethene | 30150 | 101.1 | 7.3 | 79 | 123 |
| 75-69-4 | Trichlorofluoromethane [Freon-11] | 26108 | 103 | 12.8 | 65 | 141 |
| 108-05-4 | Vinyl acetate | 18941 | 100.2 | 15.3 | 54 | 146 |
| 75-01-4 | Vinyl chloride | 29472 | 97.4 | 13.2 | 58 | 137 |
| 1330-20-7 | Xylenes [total] | 23426 | 100.1 | 7 | 79 | 121 |

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 Analysis: 624/8260/6200
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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|--|---|-----------------------------------|------------------------------|---|
| Tune Check | 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. | No samples shall be analyzed without a valid tune. |
| Initial calibration (ICAL) for all analytes (including surrogates) | At instrument set-up, prior to sample analysis | Each analyte must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$. | Correct problem then repeat ICAL. | Flagging is not appropriate. | Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met. |
| Retention Time window position establishment | Once per ICAL and at the beginning of the analytical sequence. | Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used. | NA. | NA. | Required for each analyte and surrogate. |
| Evaluation of Relative Retention Times(RRT) | With each sample. | RRT of each reported analyte within ± 0.06 RRT units. | Correct problem, then rerun ICAL. | NA | RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs. |

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 Analysis: 624/8260/6200
 Date/rev: 10/18/16 R23
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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|--|---|---|---|---|
| Initial Calibration Verification (ICV) | Once after each ICAL, analysis of a second source standard prior to sample analysis | All reported analytes within $\pm 20\%$ of true value. | Correct problem. Rerun ICV. If that fails, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source. |
| Continuing Calibration Verification (CCV) | Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run. | All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the | If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. | Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met. |

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012 013 014 015 016 017 018 019 020 021 022 023 024 025 026 027 028 029 030 031 032 033 034 035 036 037 038 039 040 041 042 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 059 060 061 062 063 064 065 066 067 068 069 070 071 072 073 074 075 076 077 078 079 080 081 082 083 084 085 086 087 088 089 090 091 092 093 094 095 096 097 098 099 100



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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|-------------------------|---|--|---|---|---|
| Internal standards (IS) | Every field sample, standard and QC sample. | Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard. | Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory. | If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards. | |
| Method Blank (MB) | One per preparatory batch. | No analytes detected $> \frac{1}{2}$ LOQ or $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit, whichever is greater. Common contaminants must not be detected $> \text{LOQ}$. | 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. | Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

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 Analysis: 624/8260/6200
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Standard Operating Procedure

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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---------------------------------|----------------------------|---|--|--|---|
| Laboratory Control Sample (LCS) | One per preparatory batch. | A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. | Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available | If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| Matrix Spike (MS) | One per preparatory batch. | A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. | Examine the project specific requirements. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative. | Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error. |

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0123 4567 8910 1112 1314 1516 1718 1920 2122 2324 2526 2728 2930 3132 3334 3536 3738 3940 4142 4344 4546 4748 4950 5152 5354 5556 5758 5960 6162 6364 6566 6768 6970 7172 7374 7576 7778 7980 8182 8384 8586 8788 8990 9192 9394 9596 9798 9900



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TITLE: VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|----------------------------|---|---|--|--|
| Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD) | One per preparatory batch. | A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD). | Examine the project specific requirements. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative. | MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. |
| Surrogate Spike | All field and QC samples. | QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 and 4 or in-house LCS limits (see the LIMS) if analyte(s) are not listed. | Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. | Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative. | Alternative surrogates are recommended when there is obvious chromatographic interference. |

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**Environmental Science Corporation
SOP MINOR REVISION FORM**

SOP/DOC# 330363 Current revision date & number: R23 10/18/2016
 Procedure/Method : VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Date | Requested By | Section | Revision | Reason* | Approvals | |
|---------|--------------|---------|---|-------------------------------|---------------|----------------|
| | | | | | Supervisor | QA |
| 11/4/16 | SCM | 10.9 | Revise the State Note to read: 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% | SC DHEC 10/19/16 Letter | HF 11/4/16 | SCM 11/4/16 |

**Environmental Science Corporation
SOP MINOR REVISION FORM**

| Date | Requested By | Section | Revision | Reason* | Approvals | |
|---------|--------------|------------------|---|-------------------------------|---------------|----------------|
| 11/4/16 | SCM | 10.9 (cont'd) | 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% 1,1,2-Trichloroethane 70 – 130% Trichloroethene 71 – 130% Trichlorofluoromethane 70 – 130% Vinyl chloride 70 – 130% Xylenes, total 70 – 130% | SC DHEC 10/19/16 Letter | HF 11/4/16 | SCM 11/4/16 |
| 11/4/16 | SCM | 10.10 | Add the following State Note: "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 and 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% 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% | SC DHEC 10/19/16 Letter | HF 11/4/16 | SCM 11/4/16 |

**Environmental Science Corporation
SOP MINOR REVISION FORM**

SOP/DOC# 330363 Current revision date & number: 10/18/16 R23a
 Procedure/Method : VOLATILE ORGANIC COMPOUNDS BY GC/MS (EPA 8260B, 8260C, 624 AND SM6200B)

| Date | Requested By | Section | Revision | Reason* | Approvals | |
|----------|--------------|---------|---|---------|-------------|--------------|
| | | | | | Supervisor | QA |
| 12/28/16 | H. Ferrell | 8.3.3 | Add Methyl Cyclohexane to the poor performer list | LOD/LOQ | HF 12/28/16 | Shu 12/29/16 |
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*Comments:

Groundwater Sampling

A8.1 Purpose and Applicability

This procedure conforms to the EPA Quality Assurance Requirements and describes methods for purging and sampling a groundwater monitoring well to ensure that the sample collected is representative of the formation groundwater. The procedure follows EPA guidance detailed in *Region 4 Science and Ecosystem Support Division (SESD) Operating Procedure (OP) for Groundwater Sampling, SESDPROC-301-R3*. (EPA, 2013).

A8.2 Definitions

Bailer: A hollow tube constructed of stainless steel or Teflon® that is used to collect groundwater samples. A dedicated bailer remains in the well casing.

A8.3 Procedures

A8.3.1 Purging

The following equipment is required for well purging:

- Bailer or pump. The device used depends upon aquifer properties, individual well construction, well yield, and data quality objectives (DQOs).
- Water level measuring device.
- Tape measuring device.
- pH, specific conductance, turbidity, and temperature measuring device.

Well purging is performed as follows:

- For the well to be purged/sampled, the following information is obtained and recorded in the groundwater purging/sampling data sheet or the field log book: date, field conditions, well location, well ID, well diameter, groundwater elevation, total well depth, screened interval, water quality field measurements (pH, specific conductance, turbidity, and temperature), and the method for disposal of purged water.
- Field instruments are calibrated prior to use and according to manufacturers' instructions.
- Prior to opening the well, plastic sheeting is placed on the ground surrounding the well head to prevent contamination by sample spillage;
- The well is unlocked and opened and an FID/PID reading is immediately taken.
- The water level and the total depth of the well are measured.
- The volume in gallons of water in the well casing or sections of telescoping well casing is calculated as follows:

$$(\pi r^2 h) 7.48 = \text{gallons}$$

$$\text{where: } \pi = 3.142$$

$$r = \text{Radius of the well pipe in feet}$$

h = Linear feet of water in well

7.48 = Gallons per cubic foot of water

The volume of water in typical well casings may be calculated as follows:

gallons/feet x ___ (linear feet of water) = total gallons

where:

2-inch well = 0.163 gallons/foot

3-inch well = 0.367 gallons/foot

4-inch well = 0.653 gallons/foot

5-inch well = 1.02 gallons/foot

6-inch well = 1.469 gallons/foot

7-inch well = 1.999 gallons/foot

8-inch well = 2.611 gallons/foot

10-inch well = 4.28 gallons/foot

12-inch well = 5.87 gallons/foot

- Purging the well will begin by lowering the decontaminated purging apparatus (pump or bailer) to the standing water column so that the water will be pulled through the casing and the entire static volume will be removed. A bailer is used when the well does not yield sufficient water for pumping; otherwise, a pump is preferred. For low-flow sampling techniques, see section A8.3.4.
- The initial pH, specific conductance, turbidity, and temperature of water are measured and recorded in the field logbook along with the odor, color, clarity, silt concentrations and general water condition. During purging, field parameters are measured at least once during each well volume (more often is preferable). Record changes in the physical condition of the monitoring wells that could affect the well integrity.
- For purging to be complete, a total of at least 3-5 volumes of groundwater should be removed from the well, and the field parameters must stabilize. The amount of purged fluid will be measured by filling a graduated bucket or by using a stopwatch and noting the flow rate of the pump versus elapsed time. Field parameter stabilization is as follows: pH measurements ± 0.1 units, temperature measurements $\pm 1^\circ\text{C}$, specific conductance measurements $\pm 10\%$, and $\pm 10\%$ for turbidity).
- Wells with little or no recharge will be purged to near dryness, and the well is allowed to recover before sampling.
- When using a pump, prior to the completion of purging activities, the pump will be brought to the water surface to ensure complete removal of stagnant water.
- Purge water will be placed in a storage tank and disposed of as IDW

Wells will be sampled immediately after purging, if possible, but generally no later than 6 hours after purging. Wells that recharge slowly will be purged dry and allowed to recharge before sampling. If excessive time (greater than 10 hours) is required for the slow recharging wells to recharge, it will be documented in the field log.

A8.3.2 Sample Collection

Following are the general procedures for groundwater sampling along with methods for utilizing specific sampling devices and techniques.

A8.3.2.1 General

- With the exception of low-flow sampling (Section A8.3.4) and open borehole sampling, before samples are taken, the well is purged as described in Section A8.3.1.

- Sampling equipment will be cleaned and decontaminated prior to the commencement of sampling activities. A new pair of disposable gloves will be worn at each location by sampling personnel.
- Pre-labeled, pre-cleaned, sample bottles with preservative added, are used to contain the groundwater samples. VOA samples will be collected first followed by other organic analyses. Inorganic analyses are collected last except in the case where the influences of turbidity on metals concentrations is a concern. In this case metals samples will be collected immediately following the volatile organics.
- As the sample is taken, the sample container is tilted slightly allowing the water to run down the inside of the sample bottle with a minimum of splashing
- Adequate space is left in the bottle to allow for expansion, except for volatile organic analysis (VOA) vials, which are filled to overflowing and capped. VOAs vials are checked for air bubbles and if detected, more sample is carefully added to the vial (care must be taken to minimize the loss of preservative).
- Samples are placed in appropriate containers, and packed with ice in coolers immediately after the sample is collected.

Measure pH, conductivity, temperature, and turbidity after sample bottles have been filled and record the measurements in logbook.

A8.3.2.2 Bailer

A decontaminated Teflon® bailer can be used to remove groundwater samples from a well as follows:

- A decontaminated and properly secured, bailer is lowered to the sampling interval from which the sample is to be collected.
- The bailer is allowed to fill with a minimum of surface disturbance to prevent sample water aeration. When the bailer is raised, the bailer cord must not be allowed to touch the ground.
- The sample is slowly poured from the bailer and the bottle is tilted slightly allowing the water to run down the inside of the sample bottle with a minimum of splashing
- If the bailer is dedicated, it is returned to the well and the well is capped and locked. Non-dedicated samplers are cleaned and decontaminated after use.

A8.3.3 Purging/Sampling Using a Small Diameter, Electric Submersible Pump

Small Diameter Electric Submersible Pumps includes a range of small diameter, variable speed pumps capable of pumping rates ranging from 0.5 ml./min. to in excess of 9 gallons per minute. The power source for these pumps can be provided directly from an automobile battery or from a generator. While small diameter pumps are generally light-weight and easily handled by one person when lowering into a well, two people are generally needed when removing the pump, one to pull and another to reel the hose and power lead. Groundwater monitoring wells can be purged utilizing a decontaminated pump and clean flexible tubing as follows:

- Slowly lower the pump to the middle of the screened interval. This minimizes excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize re-suspension of solids, which will have collected at the bottom of the well (EPA, 1996).
- Follow the manufacturer's procedures, and begin pump-purging the monitoring well.
- If the recovery rate of the well is faster than the pump rate, the pump may be left hanging at the initial level. If the pump rate exceeds the recovery rate, the pump must be lowered to accommodate the drawdown, or the pump rate can be decreased.

- Once 3-5 well volumes have been removed from the well, and the field parameters have stabilized, remove the pump from the well, and sample utilizing a Teflon® bailer.

A8.3.4 Purging/Sampling Using Modified Low-Flow Techniques

Low-flow techniques are utilized to obtain a more representative sample from the aquifer formation. CH2M HILL is currently using this procedure for its quarterly and semiannual groundwater sample collection at Sites B-10 and B-90. In general, the advantages of low-flow purging include (EPA, 1996):

- Samples which are representative of the mobile load of contaminants present (dissolved and colloid-associated);
- Minimal disturbance of the sampling point thereby minimizing sampling artifacts (i.e. less turbidity);
- Less operator variability, greater operator control;
- Reduced stress on the formation (minimal drawdown);
- Less mixing of stagnant casing water with formation water;
- Reduce the need for filtration and, therefore, less time required for sampling;
- Smaller purging volume which decreases IDW disposal costs;
- Better sample consistency; reduced artificial sample variability.

The pumps selected for perform low-flow sampling, should be capable of producing purge rates sufficient to allow for the modified low-flow sampling technique. Pumps, which meet these requirements include but are not limited to, bladder-type pumps (provided that reagent grade nitrogen is used for bladder inflation) and the Grundfos Redi-Flow2 pump.

Following are the procedures for modified low-flow groundwater sampling. These procedures include adaptations from EPA's paper entitled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (EPA, 1996):

- Slowly lower the decontaminated pump to the middle of the screened interval. This is to minimize excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize re-suspension of solids, which will have collected at the bottom of the well.
- Once the pump is positioned in the well, an airtight flow-through cell (equipped with a YSI or Horiba-type water quality meter) is plumbed to the water discharge line.
- Lower a decontaminated water level gauge into the well to monitor the water table.
- Once purging is initiated, water level measurements should be continuously monitored, and pumping rates adjusted as necessary (e.g., 0.1 - 0.3 L/min) to maintain minimal drawdown. Modified low-flow techniques should cause less than three feet of drawdown during purging.
- While purging, the groundwater field parameters (including water level) should be continuously monitored every 3-5 minutes until all parameters have stabilized for 3 consecutive readings.
- Stabilization for each parameter is defined as follows: ± 0.1 for pH, $\pm 3\%$ for conductivity, ± 10 mv for redox potential, $\pm 10\%$ for turbidity, $\pm 10\%$ for dissolved oxygen (DO), and ± 3 ft. for drawdown.

Once field parameters have stabilized for 3 consecutive readings, samples may be taken. The same device used for purging should be used for sampling (remove flow-through cell).

Water-Level Measurements

A9.1 Purpose and Scope

The purpose of this procedure is to provide a guideline for the measurement of the depth to groundwater in piezometers and monitoring wells, even where a second phase of floating liquid (e.g., gasoline) is encountered, and on staff gages in surface-water bodies. This SOP includes guidelines for discrete measurements of static water levels and does not cover the use of continuously recording loggers (see SOP Use of Data Loggers and Pressure Transducers).

A9.2 Equipment and Materials

- Electronic water-level meter (Solinst® or equivalent) with a minimum 100-foot tape; the tape should have graduations in increments of 0.01 feet or less
- Interface probe (Solinst® Model 122 Interface Meter or equivalent)

A9.3 Procedures and Guidelines

Verify that the unit is turned on and functioning properly. Slowly lower the probe on its cable into the piezometer or well until the probe just contacts the water surface; the unit will respond with a tone or light signal. Note the depth from a reference point indicated on the piezometer or well riser. Typically this is the top of the PVC casing. If no reference is clearly visible, measure the depth to water from the northern edge of the PVC casing. If access to the top of the PVC casing is difficult, sight across the top of the locking casing adjacent to the measuring point, recording the position of the cable when the probe is at the water surface.

Measure the distance from this point to the closest interval marker on the tape, and record the water level reading in the logbook. Water levels will be measured to the nearest 0.01-foot. Also when specified in the project plans, measure and record the depth of the piezometer or well. The depth of the piezometer or well may be measured using the water-level probe with the instrument turned off.

Free product light or dense nonaqueous phase liquid may be present in the piezometer or well. If the presence of free product is suspected, the thickness of the product should be determined using appropriate equipment (e.g., Solinst® Model 122 Interface Meter). The depth to water also is determined with this equipment and the water-level meter should not be used in the piezometer or well as long as product is present. Typically, a constant sound is emitted from the device when free product is encountered and an alternating on/off beep sound is emitted when water is encountered.

The apparent elevation of the water level in the well or piezometer is determined by measuring both the apparent depth to water and the thickness of free product. The corrected water-level elevation is calculated by the following equation:

$$WL_c = WL_a + (\text{Free-product thickness} \times 0.80)$$

Where WL_c = Corrected water-level elevation

WL_a = Apparent water-level elevation

0.80 = Typical value for the density of petroleum hydrocarbon products.

If free product is detected on the surface of the water in the piezometer or well, the value of sampling should be reconsidered because of the potential for contaminating the sampling equipment.

Staff gages may be installed in some surface-water bodies. These facilities typically are constructed by attaching a calibrated, marked staff gage to a wood or metal post, driving the post into the bottom of the surface-water body, and surveying the elevation of the top of the post to a resolution of 0.01-foot. The elevation of the water in the surface-water body then can be determined by reading off the distance the water level is from the top of the post. A shield or other protection may be needed to calm the fluctuations in water level if the gage is installed at a location exposed to wind or wave.

A9.4 Attachments

None.

A9.5 Key Checks

- Before each use, verify that the battery is charged by pressing the test button on the water-level meter.
- Verify that the unit is operating correctly by testing the probe in distilled or de-ionized water. Leave the unit turned off when not in use.

Use of Data Loggers and Pressure Transducers

A10.1 Purpose and Scope

The purpose of this procedure is to provide a guideline for the use of continuously recording data loggers and pressure transducers to monitor changes in water levels and barometric pressure. Procedures for using a particular type of data logger are not included and should be obtained from the equipment manuals. The SOP *Water-Level Measurements* provides guidance for collecting discrete measurements of water levels.

A10.2 Equipment and Materials

- Continuously recording data logger (e.g., Campbell® or In Situ® Hermit or In Situ® Trolls)
- Pressure transducers (e.g., Druck® or In Situ® Trolls) of adequate pressure ranges and cable lengths
- Electronic water-level meter (e.g., Solinst®) with a minimum 100-foot tape; the tape should have graduations in increments of 0.01 feet or less
- Duct tape

A10.3 Procedures and Guidelines

The procedures for using the particular type of data logger should be obtained from the operating manuals for the equipment. In general, all data loggers have a means of specifying such data as the pressure range of the transducer, the type of monitoring to be performed (e.g., slug test), the frequency of measurements, the length of the test, and the number of transducers to be monitored.

Transducers should be selected with the appropriate pressure range for the expected range of water-level or barometric changes. A change of 1 pound per square inch (psi) of pressure is equivalent to a change of 2.3 feet of water level. Therefore, if the change in water level is expected to be small (i.e., less than 10 feet), a transducer with a range from 0 to 5 psi would be best.

For downhole applications, measure the initial water level with the water-level meter. Lower the transducer into the piezometer or well until it is comfortably below the surface. Tape the transducer cable to the casing or affix the cable holder to the locking cap or stickup with zip-ties so that the transducer does not move in the piezometer or well. Attach the transducer cable to a rugged reader or a port on the back of a laptop computer. Connect to the transducer and confirm that the transducer is at the proper depth in the water by reading the data logger. Program the data logger for the appropriate frequency of data collection.

Transducers for measuring barometric pressure are attached to a secure fixture (e.g., casing or light post) and are programmed with the rugged reader or laptop. Transducers for measuring the changes in water level in surface-water bodies are attached to a weight and lowered to the bottom. The depth of water above the transducer can be read from the data logger. Care should be taken to prevent the cable from interfering with boat traffic.

A10.4 Attachments

None.

A10.5 Key Checks and Preventive Maintenance

- Prior to each use, verify that the batteries are charged and that there is sufficient space in the data-logger memory for the data to be collected.
- Also prior to each use, the date and time should be synchronized between the data logger and the rugged reader or laptop.
- Ensure that all settings are appropriate for the transducer pressure range and the type of test.
- Protect the logger from inclement weather and vandalism.

Use and Calibration of Field Instruments

A11.1 Purpose and Applicability

This procedure conforms to the applicable EPA QA requirements and the QAPP, and it establishes standard methodologies for the use, calibration, and maintenance of field instruments. The procedure applies to all field investigations.

A11.2 Definitions

None.

A11.3 Procedure

Field instruments will be used for the collection of field data and measurement of various conditions observed during the site investigations. In general, take the following steps when using field instruments:

- Before field use, remove the instrument from its container and assemble and clean it according to the manufacturer's instructions.
- Before commencement of field activities each day, calibrate the instrument according to the manufacturer's instructions. Record the instrument's ID and serial number, along with the calibration process/results, in the field logbook and/or calibration logbook.
- Use the instrument to make the appropriate physical/chemical measurements and clean/decontaminate the instrument, if necessary, after each measurement.
- If erroneous measurements are observed or if changes in environmental conditions warrant recalibration, recalibrate the instrument as specified by the manufacturer. Record the recalibration information in the field logbook and/or the calibration logbook.
- At the end of each day, clean and decontaminate the instrument then return it to the storage location. Recharge instrument as necessary.
- Perform factory maintenance and calibration at the intervals specified by the manufacturer. Have repairs, maintenance, and calibration performed by trained individuals according to the manufacturer's requirements. Record repairs, maintenance, and calibration in the field logbook or instrument calibration/maintenance logbook.

Vacuum Extraction of Product

A12.1 Purpose and Scope

The purpose of this procedure is to provide a guideline for the recovery of petroleum light non-aqueous phase liquid (LNAPL) and petroleum-impacted groundwater from recovery features (i.e., recovery wells, recovery sumps, and recovery trenches) via vacuum extraction.

A12.2 Equipment and Materials

- Vacuum truck and ancillary petroleum resistant vacuum hoses, sight glass, and fittings.
- Oil-water interface probe.

A12.3 Procedures and Guidelines

- Mobilize vacuum truck to central location where multiple recovery features can be accessed without constantly repositioning the vacuum truck. Set parking brake and deploy wheel chocks.
- Utilizing grounding cable mounted on vacuum truck, ground vacuum truck by attaching grounding cable to the steel outer casing of a nearby monitoring well or recovery feature.
- Connect petroleum-resistant vacuum hoses to influent connection on vacuum truck and ensure camlock fittings on vacuum hoses are properly secured.
- Lower vacuum hose into recovery feature just above the LNAPL.
- Transfer power to vacuum truck blower and open vacuum valve on vacuum truck
- Lower vacuum hose within recover feature to begin extracting LNAPL. Minimize recovery of groundwater by watching the sight glass and adjusting the elevation of the hose inlet to only vacuum LNAPL. LNAPL can be observed in the sight glass as a colored fluid. When the sight glass runs clear, groundwater is being extracted, in which case the hose inlet should be adjusted to confirm that no more LNAPL is available to be extracted.
- When all available LNAPL has been removed from the recovery feature, remove vacuum hose from the recovery feature, secure the wellhead with an approved lock, and proceed to the next recovery feature and repeat until all recovery features in the vicinity of the vacuum truck have been extracted from.
- When all recovery features in the vicinity of the vacuum truck have been extracted from, clear vacuum hoses, close vacuum valve, and remove power from the vacuum truck blower. Disconnect hoses and stow hoses on vacuum truck. Remove grounding cable from grounding point and stow on vacuum truck. Remove wheel chocks and stow on vacuum truck.
- Proceed to next centralized location and repeat the aforementioned procedures until all required recovery features are extracted.
- Once the extraction event has been completed, transfer recovered LNAPL and groundwater to storage tank. Park vacuum truck in close proximity to the storage tank and set parking brake. Deploy wheel chocks and grounding cable. Connect hoses to discharge port of vacuum truck and to storage

tank influent fitting. Switch blower direction, transfer power to the blower, and open the discharge valve. Monitor fluids in storage tank to ensure the tank is not overfilled.

- Once discharge has been completed, remove power from blower and disconnect hoses and stow on vacuum truck. Disconnect grounding cable and stow on vacuum truck. Remove wheel chocks and stow on truck.

A12.4 Attachments

None.

A12.5 Key Checks

- Ensure all recovery features are locked once extraction is complete.
- Ensure vacuum truck hydraulic fluid levels are at manufacture's recommended levels.
- Using oil-water interface probe, check LNAPL and water levels in vacuum truck after extraction event is complete.



APR 26 2017

JERRY AYCOCK
PLANTATION PIPE LINE
1000 WINDWARD CONCOURSE
SUITE 450
ALPHARETTA GA 30005

Re: Quality Assurance Project Plan Revision Request
Plantation Pipe Line Lewis Drive Release, 112 Lewis Dr., Belton SC
Site #18693
Petroleum Pipeline Release Reported December 8, 2014
Quality Assurance Project Plan: Revision 2 (3/1/17), received March 2, 2017
Anderson County

Dear Mr. Aycock,

The Underground Storage Tank (UST) Management Division of the South Carolina Department of Health and Environmental Control (DHEC) has reviewed the referenced document. **Based upon this review, an approvable revision is requested within 30 days of the date stamped on this correspondence that addresses the following:**

Section A3

- 1) All field personnel should be added to the Distribution and Project Organization List.
- 2) A statement that documents that everyone on the Distribution and Project Organization List has received the most recent version of the Site Specific Quality Assurance Project Plan for the Plantation Pipe Line Lewis Drive Release and the UST Management Division Quality Assurance Program Plan (QAPP), as it is referenced in the Site Specific Quality Assurance Project Plan for the Plantation Pipe Line Lewis Drive Release.

Section A4

- 3) More details need to be provided regarding the surrounding properties, impact to health and the environment, receptors, areas affected, and the environmental media affected (e.g., soil, surface water, and ground water).

Section A6

- 4) A Topographic Map should be provided as Figure 1.
- 5) For the subsections entitled "Monitoring Well Installation and Development" through "Well Development" detailed Standard Operating Procedures (SOPs) will need to be included as appendices.
- 6) In accordance with Section B.32 of South Carolina Well Regulations and Standards (R.61-71) an open hole in a bedrock aquifer is designated as a Type I Well. The remainder of the wells, as described, would fall under the definition of a Type III Well.

Section A8

- 7) This section should state that personnel will receive the most current version of the QAPP Contractor Addendum and UST QAPP, since this document is referenced in the Site Specific Quality Assurance Project Plan for the Plantation Pipe Line Lewis Drive Release.

Section B1

- 8) Table 3A end dates for "Surface Water Sampling and Analysis" and "Groundwater Sampling and Analysis" should be changed to "Upon approval by DHEC or Project Completion"

Section B2

- 9) All sampling methods must be addressed in SOPs in appendices.

Section B4

- 10) A section included in Table 5A needs to be provided includes the equipment needed and references the attached SOP for each method
- 11) Water quality parameters, to include turbidity, dissolved oxygen, specific conductance, temperature, and pH will need to be included.

Sections B6 and B7

- 12) Tables 6A and 7A should list the make and model of all pumps and meters that will be used for this project.
- 13) This section should include specifically how the laboratory prevents unauthorized changes to laboratory notebooks & computer records.

Additional Items

- 14) SOPs must be provided for each task as appendices for all types of work performed at the site. This should cover, but not be limited to, the following (to include accompanying diagrams if applicable):
 - Groundwater purging and sampling procedures using low flow methods
 - Groundwater sampling procedures using HydraSleeves
 - Collection of surface water samples
 - Installation of permanent groundwater monitoring well via each potential method
 - Installation of permanent bedrock monitoring wells
 - Well development procedures
 - Well gauging for groundwater elevation
 - Well gauging for free product
 - Stabilization procedures when collecting groundwater samples
 - Calibration of instruments procedures
 - Free product recovery procedures
- 15) A list of all existing wells, sumps, trench points, and surface water sampling locations will also need to be included in the revision.

Documents should continue to be provided in paper format and pdf via a disk with Site ID # 18693 and Plantation Pipeline Lewis Drive Release noted in a prominent location. Should you have any questions, I can be reached at (803) 898-0628 or colemabj@dhec.sc.gov. Faxes can be sent to (803) 898-0673.

Sincerely,



Bobbi Coleman, Hydrogeologist
Assessment Section
Underground Storage Tank Management Division
Bureau of Land and Waste Management

CC: Chris McCluskey, Upstate Region EQC (Anderson Office)
William Waldron, CH2M Hill, 3120 Highwoods Blvd., Suite 214, Raleigh NC, 27604
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Technical File