

GUIDELINES FOR SAMPLING

Underground Storage Tank Section

Division of Waste Management

North Carolina Department of Environmental Quality

DRAFT January 12, 2022 Version

Change 4, Effective xxxxxxxxxxxx, 2021

Table of Contents

Definitions	v
Acronyms	ix
1.0 Purpose and Application of the Guidelines	1
2.0 General Sampling Procedures	2
2.1 Planning	2
2.1.1 Selecting a Laboratory	2
2.1.2 Equipment and Sampling Containers.....	7
2.1.3 Decontamination of Field Equipment.....	8
2.1.4 Calibration and Maintenance of Field Equipment	12
2.1.5. Preparation of Methanol Preserved Vials	14
2.2 Health and Safety	15
2.3 Preservation, Holding Times and Container Types.....	16
2.4 Sample Collection	17
2.4.1. Contamination Prevention	17
2.4.2 Sample Labels.....	18
2.4.3 Sample Collection Order	18
2.4.4 Collecting Soil Samples.....	18
2.4.5 Surface Water Samples.....	36
2.4.6 Groundwater Samples.....	39
2.4.7 Equipment Decontamination and Quality Control Samples	58
2.4.8 Product (Tank) Sampling and Drum Sampling (See Waste Sampling)	58
2.4.9 Collecting Air Samples from vapor extraction units.....	58
2.4.10 Vapor Intrusion (VI) Sampling	59
2.5 Sample Storage and Transport	60
2.5.1 Shipping Methanol Preserved Samples.....	61
2.6 Laboratory Reports.....	62
2.6.1 Required Report Elements.....	63
2.6.2 Required Document Retention Criteria	64
2.6.3 Required Blank Evaluation Criteria.....	64
2.6.4 Required Target Analytes for Approved Methods.....	65
2.6.5 Required Reporting Limits	65
3.0 References	66
4.0 Figures.....	68
Figure 1 - Regional Office Locations and Map	69
Figure 2 - Volumes of Stockpiles	70
Figure 3 - Soil Stockpile Sampling Map (Example).....	71
Figure 4 - EPA Method 18 Integrated Bag Sampling Train	72
Figure 5 - EPA Method 15 Summa Canister	73

COMPREHENSIVE TABLES FOR CORRECTIVE ACTION GUIDELINES

Table 1	Maximum Soil Contaminant Concentration Levels (MSCCs)
Table 2	Gross Contamination Levels for Groundwater
Table 3	Approved Soil Analyses Methods for Petroleum UST Closures, Over-Excavation and Site Checks
Table 4	Approved Soil Analyses Methods for Advanced Phases of Petroleum UST and Non-UST Release Investigations
Table 5	Approved Groundwater Analyses Methods for Petroleum UST and Non-UST Release Investigations (All Phases)
Table 6	Approved Soil Analyses Methods for Non-Petroleum UST Closures and Release Investigations
Table 7	Approved Groundwater Analysis Methods for Non-Petroleum UST Closures and Release Investigations
Table 8	Soil Analyses Sample Containers and Preservatives
Table 9	Groundwater Analyses Sample Containers and Preservatives
Table 10	Worksheet for Calculating MADEP Soil Sample Results
Table 11	Worksheet for Calculating MADEP Groundwater Sample Results
Table 12	Approved Methods for Soil Analyses for a Permit
Table 13	Equipment Construction Materials
Table 14	Construction Material Selection for Sample Collection Equipment
Table 15	Equipment for Collecting Groundwater Samples
Table 16	Water Sampling Equipment Use and Construction
Table 17	Soil Sampling Equipment Use and Construction

COMPREHENSIVE GUIDELINES APPENDICES FOR STIRA,
ASSESSMENT GUIDELINES AND CORRECTIVE ACTION
GUIDELINES

Available at <https://deq.nc.gov/about/divisions/waste-management/ust/guidance-documents>

Appendix A – Report Formats

1. Site Check Report
2. UST-3 - Notice of Intent: UST Permanent Closure or Change-in-Service
3. 24-Hour Release and UST Leak Reporting Form (UST 61 Form)
4. 24-Hour Notification of Discharge Form (UST 62 Form)
5. 20-Day Report
6. UST Closure Report (following UST-12 format) and Site Investigation Report for Permanent Closure or Change-in-Service of UST (UST-2 Form)
7. Site Investigation Report for Permanent Closure or Change-in-Service of UST
8. Initial Abatement Action (Site Check, UST Closure with UST-2 Form, Excavation, Post-Excavation Soil Contamination Assessment) Report
9. 45-Day Report (For non-petroleum UST releases only)
10. Free Product Recovery Report
11. Free Product Recovery System Specification Report
12. Initial Site Assessment Report
13. Limited Site Assessment Report
14. Request for Water Supply Well Information
15. Comprehensive Site Assessment Report
16. Soil Assessment Report
17. Corrective Action Plan
18. Soil Cleanup Plan
19. Monitoring Reports
20. System Enhancement Recommendation Report
21. New Technology Cleanup Plan
22. Soil Cleanup Report with Site Closure Request
23. Site Closure Report
24. Format of Individual Public Notice
25. Format of Public Notice by Posting (15A NCAC 2L .0409(a))
26. Format of Individual Public Notice for Non-Petroleum UST Releases
27. VPH (Aliphatics/Aromatics) Laboratory Reporting Form
28. EPH (Aliphatics/Aromatics) Laboratory Reporting Form

Appendix B – Reporting Tables

Table B-1: Site History – UST/AST System and Other Release Information

Table B-2: Site History - UST/AST Owner/Operator and Other Responsible Party Information

Table B-3: Summary of Soil Sampling Results

Table B-4: Summary of Groundwater and Surface Water Sampling Results

Table B-5: Public and Private Water Supply Well and Other Receptor Information

Table B-6: Contiguous Property Owners/ Occupants

Table B-7: Monitoring and Remediation Well Construction Information

Table B-8A: NAPL Recovery Information

Table B-8B: Cumulative Volume of NAPL Recovered from Site

Table B-9: Current and Historical Groundwater Elevations and NAPL Thickness

Table B-11: Summary of Air/Vapor Sampling Results

Table B-10: Land Use

Table B-12: Evaluation of Change in Contaminant Mass

Appendix C – UST Systems: Regulated or Not Regulated under 15A NCAC 02N

Appendix D - Collecting Soil Samples

Appendix E - Disposal of Contaminated Soil and Groundwater

Appendix F - Guidance Pertaining to Releases from Contaminant Sources Other than USTs

Petroleum ASTs and Petroleum Surface spills

Contamination Related to Naturally Occurring Conditions, Permitted Facilities, and Agricultural Activities

Hazardous Waste

Superfund

Pesticide Contamination

Dry Cleaning Facilities

Non-petroleum Releases not described by Above Items

Appendix G - Required Permits

Appendix H - Aquifer Testing

Appendix I - Notices of Residual Petroleum

Appendix J. Development of Maximum Soil Contaminant Concentrations

Definitions

Action Level: the concentration of a contaminant that if exceeded may require further regulatory action such as cleanup or monitoring.

Aquifer: a permeable body of rock or sediment that stores and transmits groundwater in sufficient quantity to supply wells or springs.

Bedrock: any consolidated rock which is encountered in the place in which it was formed or deposited and which cannot be readily excavated without the use of explosives or heavy rock cutting equipment. (15A NCAC 02L .0102) Bedrock generally underlies soil or other unconsolidated, superficial material.

Below Ground Surface (bgs): depth beneath the surface of the earth

Cleanup Level: the concentration of a contaminant at which *no* further cleanup actions are required based on the risk of harm posed by the contaminant

Closure: activities conducted during the permanent removal (or abandonment) of underground storage tank systems and not inclusive of abatement or corrective actions, or remediation.

Confining Layer: a layer having very low hydraulic conductivity, in relationship to adjacent stratigraphic units, that restricts the movement of water into and out of an aquifer (e.g., dense, unfractured clay).

Confirmed Release: a release for which an analytical result for sampled media shows any contaminant level above the Method Detection Limit.

Contaminant: means any substance occurring in groundwater in concentrations which exceed the groundwater quality standards specified in 15A NCAC 02L .0202.

De Minimis Concentration: amount of a regulated substance which does not exceed one percent (1%) of the capacity of the tank, excluding piping and vent lines (15A NCAC 02N.0203).

Department: the North Carolina Department of Environmental Quality.

Discharge: a release (See also Release).

Division: the Division of Waste Management.

Ex Situ Soil: soil that has been excavated.

Free Product: any accumulation of a substance of greater than or equal to 1/8 inch (0.010417 foot) in contact with groundwater or perched on the water table, with a density less than or greater than water, and existing as a non-aqueous phase liquid (i.e., not dissolved in water).

Gross Contamination Levels (GCLs): levels of groundwater contamination for any contaminant (except ethylene dibromide, benzene and the aliphatic and aromatic carbon fraction classes) that exceed 50 percent of the solubility of the contaminant at 25 degrees Celsius or 1,000 times the groundwater quality standard or interim groundwater quality standard established in 15A NCAC 02L .0202, whichever is lower: and levels of groundwater contamination for ethylene dibromide and benzene that exceed 1,000 times the federal drinking water standard set out in 40 CFR 141.

Groundwater: those waters occurring in the subsurface under saturated conditions.

Hazardous Substance: a hazardous substance defined in Section 101 (14) of the Comprehensive Environmental Response Compensation and Liability (CERCLA) Act of 1980 (but not including any substances regulated as a hazardous waste under Subtitle C or any mixture of such substances and petroleum).

Hazardous Waste: discarded material which, due to its quantity, concentration, or physical or chemical characteristics, may cause or significantly contribute to an increase in mortality, irreversible or incapacitating reversible illness, or pose a substantial threat or potential hazard to human health or the environment when improperly treated, stored, transported, disposed or otherwise managed (Federal regulations define a waste as a hazardous waste if it exhibits a characteristic of a hazardous waste (40 CFR 261.20 through 261.24); has been listed as hazardous (40 CFR 261.31 through 261.33); or is a mixture containing a listed hazardous waste and a non-hazardous solid waste (unless the mixture is specifically excluded or no longer exhibits any of the characteristics of a hazardous waste).

In Situ Soil: soil or fill material that is in the ground and has not been disturbed.

Investigation-Derived Waste (IDW): the water, soil and cuttings generated during drilling and sampling activities for the purpose of investigating an actual or potentially contaminated site

Land Application: the process of remediating contaminated soil by spreading soil over land. Land application may include remediating soil by natural biological methods, enhanced biological methods, or volatilization.

Maximum Soil Contaminant Concentration: the concentration of a soil contaminant at which no further cleanup actions are required based upon the risk of harm posed by the contaminant.

Method Detection Limit: the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR 136 Appendix B).

Minimum Reporting Limit: the minimum reporting limit that must be achieved by laboratories for target analyte results submitted to the UST Section; it is a reporting limit established by the UST Section for the target analytes required for each approved analytical method as an alternative to the detection limit indicated in the method description and is listed for each analyte in the *Guidelines for Sampling*.

Petroleum or Petroleum Product: crude oil or any fraction thereof which is liquid at standard conditions of temperature (60 degrees Fahrenheit) and pressure (14.7 pounds per square inch absolute), but excluding substances defined as a hazardous substance in Section 101 (14) of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) of 1980.

Petroleum Contaminated Soil or Soil Containing Petroleum Products: any soil that has been exposed to petroleum products because of any emission, spillage, leakage, pumping, pouring, emptying, or dumping of petroleum products onto or beneath the land surface and that exhibits characteristics or concentrations of typical petroleum product constituents in quantities that exceed the soil-to-groundwater MSCC or the residential MSCC, whichever is lower, as established by 15A NCAC 02L .0411.

Receptor: any human, plant or animal, structure or surface water body that is or has the potential to be adversely effected by the release or migration of contaminants.

Release: any spilling, leaking, emitting, discharging, escaping, leaching or disposing into groundwater, surface water or subsurface soils. (Refer to statutes and regulations relevant to UST releases or to AST and surface releases.)

Responsible Party: a UST owner, UST operator, and/or landowner seeking reimbursement from the State Trust Fund, or any person who is responsible for a discharge or release of petroleum or a hazardous substance. (Refer to statutes and regulations relevant to UST releases or to AST releases and spills.)

Soil or Regolith: a general term for the fragmental and unconsolidated geological material of highly varied character that nearly everywhere forms the surface of the land and overlies or covers bedrock. It includes rock debris of all kinds, volcanic ash, glacial till, alluvium, loess and eolian deposits, and vegetal accumulations.

Soil Scientist: an individual who is a Certified Professional in Soils through the NCRCPS (N.C. Registry of Certified Professionals in Soils) or a Certified Professional Soil Scientist or Soil Specialist by ARCPACS (American Registry of Certified Professionals in Agronomy, Crops and Soils) or a Registered Professional Soil Scientist by NSCSS (the National Society of Consulting Soil Scientist) or can provide documentation that he/she meets the minimum education and experience requirements for certification or registration by one or more of the organizations named in this Subparagraph or upon approval by the Director, an individual with a demonstrated knowledge of soil science. (15A NCAC 02T .0103(38)).

Source Area: point of release or discharge. The term ‘secondary source area’ refers to any zone of NAPL-impacted soil that continues to release contaminants in the subsurface.

Surface Water: all waters of the state as defined in North Carolina General Statute (NCGS) 143-215.77 Article 21A, except for underground waters, such that "waters" shall mean any stream, river, creek, brook, run, canal, swamp, lake, sound, tidal estuary, bay, reservoir, waterway, wetlands or any other body or accumulation of water, surface or underground, public or private, natural or artificial, which is contained within, flows through, or borders upon this State, or any portion thereof, including those portions of the Atlantic Ocean over which this State has jurisdiction.

Total Petroleum Hydrocarbons (TPH): the measurable amount of petroleum-based hydrocarbon in an environmental media in which it is found.

Transmissivity: the ability of geologic material to transmit water.

Underground Storage Tank (UST): any one or combination of tanks (including underground pipes connected thereto) that is used to contain an accumulation of regulated substances, and the volume of which (including the volume of underground pipes connected thereto) is 10 percent or more beneath the surface of the ground (Refer to full definition in 15A NCAC 2N .0203.).

Used Oil: means any oil that has been refined from crude oil, or any synthetic oil, that has been used and as a result of such use is contaminated by physical or chemical impurities.

UST System: an underground storage tank, connected underground piping, underground ancillary equipment, and containment system, if any.

Waste Oil: a generic term for oil that has been contaminated with substances that may or may not be hazardous. Any used oil or waste oil spill from a non-UST stored generator, transporter, recycler, etc. would fall under the jurisdiction of the Hazardous Waste Section if determined to be a hazardous waste.

Water Table: the surface of the saturated zone below which all interconnected voids are filled with water and at which the pressure is atmospheric.

Acronyms

<u>AFVR</u>	Aggressive Fluid - Vapor Recovery
<u>AST</u>	Aboveground Storage Tank
<u>ASTM</u>	American Society for Testing and Materials
<u>CAP</u>	Corrective Action Plan
<u>CAS</u>	Chemical Abstracts Service Number
<u>CERCLA</u>	Comprehensive Environmental Response, Compensation and Liability Act
<u>CFR</u>	Code of Federal Regulations
<u>CSA</u>	Comprehensive Site Assessment
<u>DEQ</u>	Department of Environmental Quality
<u>DWR</u>	Division of Water Resources
<u>DWM</u>	Division of Waste Management
<u>EDB</u>	Ethylene Dibromide (1,2 Dibromoethane)
<u>EPA</u>	The Environmental Protection Agency
<u>FID</u>	Flame Ionization Detector
<u>GCL</u>	Gross Contamination Level
<u>HCl</u>	Hydrochloric Acid
<u>HNO₃</u>	Nitric Acid
<u>IAA</u>	Initial Abatement Action
<u>IAR</u>	Initial Site Assessment Report
<u>IATA</u>	International Air Transport Association
<u>IDW</u>	Investigation Derived Waste
<u>L.G.</u>	Licensed Geologist
<u>LSA</u>	Limited Site Assessment
<u>MADEP</u>	Massachusetts Department of Environmental Protection
<u>MDL</u>	Method Detection Limit
<u>MMPE</u>	Mobile Multi-phase Extraction
<u>MRL</u>	Minimum Reporting Limit
<u>MSCC</u>	Maximum Soil Contaminant Concentration
<u>NC</u>	North Carolina
<u>NCAC</u>	North Carolina Administrative Code
<u>NCDA&CS</u>	North Carolina Department of Agriculture & Consumer Services

<u>NCGS</u>	North Carolina General Statutes
<u>NCS</u>	Notice of Contaminated Site
<u>NFA</u>	No Further Action
<u>NORR</u>	Notice of Regulatory Requirements
<u>NOV</u>	Notice of Violation
<u>NPDES</u>	National Pollutant Discharge Elimination System
<u>NRP</u>	Notice of Residual Petroleum
<u>OPHSCA</u>	Oil Pollution and Hazardous Substances Control Act of 1978
<u>PAH</u>	Polycyclic Aromatic Hydrocarbon
<u>PCB</u>	Polychlorinated Biphenyl
<u>P.E.</u>	Professional Engineer
<u>PID</u>	Photo Ionization Detector
<u>PFAS</u>	Per- and polyfluoroalkyl substances
<u>POTW</u>	Publicly Owned Treatment Works
<u>QA/QC</u>	Quality Assurance/Quality Control
<u>SAR</u>	Soil Assessment Report
<u>SCR/SCR</u>	Soil Cleanup Report/Site Closure Request
<u>SM</u>	Standard Method
<u>STF</u>	State Trust Fund
<u>SVE</u>	Soil Vapor Extraction
<u>SVOC</u>	Semi-volatile Organic Compounds
<u>SW</u>	Solid Waste
<u>TCLP</u>	Toxicity Characteristic Leaching Procedure (EPA Method SW-846 1311)
<u>TOC</u>	Total Organic Carbon
<u>TPH</u>	Total Petroleum Hydrocarbons
<u>TPH-DRO</u>	Total Petroleum Hydrocarbons - Diesel Range Organics
<u>TPH-GRO</u>	Total Petroleum Hydrocarbons - Gasoline Range Organics
<u>UST</u>	Underground Storage Tank
<u>UVF</u>	Ultraviolet Fluorescence
<u>USGS</u>	United States Geological Survey
<u>VOA</u>	Volatile Organic Analysis
<u>VOC</u>	Volatile Organic Compounds

1.0 Purpose and Application of the Guidelines

The purpose of this document is to provide guidance on the sampling process for environmental monitoring associated with petroleum releases and releases from underground storage tank (UST) systems. Questions concerning the information presented in this document should be directed to the UST Section central office at 919-707-8200. Questions concerning a specific site should be directed to the UST Section regional office that is responsible for the county in which the site is located. The address, telephone number, and jurisdiction of each regional office can be found on the UST website at <https://deq.nc.gov/about/divisions/waste-management/ust/corrective-action>.

Requirements for environmental monitoring are described in 15A NCAC 2B .0500, 15A NCAC 2T and 15A NCAC 2L .0110. In conjunction with the Guidelines for Sampling, the following specific guidance documents for each type of monitoring activity are to be used: *Guidelines for Site Check, Tank Closure, and Initial Response and Abatement Petroleum And Hazardous Substance UST Releases Petroleum Non-UST Releases; UST Section Assessment Guidelines; UST Section Corrective Action Guidelines; and the Guidelines for Ex Situ Petroleum Contaminated Soil Remediation*. Electronic versions of the guidelines are available for download from the Division of Waste Management web site at <https://deq.nc.gov/about/divisions/waste-management/ust>. Electronic versions of the rules can be found on the Office of Administrative Hearings web page at <http://reports.oah.state.nc.us/ncac.asp>

2.0 General Sampling Procedures

Sampling activities are associated with site checks; UST closures; initial release response and abatement activities; assessment and corrective actions; and soil remediation permitting. A systematic sampling approach must be used to assure that sample collection activities provide usable data. Sampling must begin with an evaluation of background information, historical data and site conditions. General sampling procedures are described in this section. The location, type and number of environmental samples for specific monitoring activities (e.g. closure, ex-situ soil remediation and assessment and corrective action) are described in the specific guideline documents for each type of activity.

2.1 Planning

Sampling activities should begin with planning and coordination. Analytical reporting requirements that must be communicated to the laboratory in the selection process to ensure the laboratory can meet project specific needs. The party contracting with the laboratory is responsible for effectively communicating reporting requirements and evaluating data usability as it relates to specific monitoring activities. Planning for sampling should also address equipment and sampling containers as follows (see Section 2.1.2).

2.1.1 Selecting a Laboratory

The NCDEQ DWR Laboratory Certification Program generates a list of certified commercial laboratories. The list includes laboratory contact information and the analytical methods they are certified to perform. A copy of the list may be obtained from the NCDEQ DWR Chemistry Laboratory at 4405 Reedy Creek Road, Raleigh, NC 27607 or the DWR Laboratory Certification List web page at <https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/certified-laboratory-listings> or by calling (919) 733-3908. Each responsible party, person or organization that uses laboratory services has certain responsibilities to ensure that the laboratory has the appropriate credentials and that the data are useable for the intended needs. These responsibilities include:

- Evaluate the laboratory: Ensure that the laboratory has the proper credentials. Ensure that the laboratory can produce data of a quality that will be acceptable to NCDEQ's DWM UST Section.
- Evaluate the reported data: Review the final laboratory reports against the original expectations and acceptable quality control measures.
- Ask questions: The user has the right to question laboratory results, quality data and data acceptability and receive a logical, clear response.

A. Identifying Laboratory Needs

The critical needs must be determined before considering any laboratory:

1. The purpose for which the data are needed.
 - a) Expectations must be determined for data quality in terms of the precision, accuracy and detection limit (reporting level or criteria) for each reported value.
 - b) Examples include: permit compliance at some specified concentration levels, compliance monitoring at specified reporting levels; and site cleanup to specified soil and water cleanup levels.

2. The benefits of using contracted or in-house analytical services.
3. The specific laboratory services that are required:
 - a) Are sample collection and sample analysis required, or just sample analysis?
 - b) Types of samples (groundwater, soils, air, etc.).
 - c) The number, frequency and types of samples to be analyzed.
 - d) The test methods that must be used (normally found in permits, guidance documents or relevant rules).
 - e) The expected quality based on regulatory requirements.
 - f) The expected turnaround time for laboratory analysis.
 - g) The deliverables including the report format.
 - h) Field related services such as sample collection.
4. Any required laboratory credentials, such as certification.
5. Identify key personnel that will interface with the laboratory.
6. Understand the current market price for the tests to be performed.

B. Evaluating the Laboratory

1. Laboratory Credentials - The laboratory must hold certification from the N.C. DEQ DWR Laboratory Certification Program.
2. On-site Visit - Conduct an on-site visit to verify the laboratory's capabilities and to determine if the laboratory has the equipment and personnel resources necessary for proposed services.
 - a) The laboratory must show a willingness to meet the client's needs.
 - b) The laboratory (both the analytical and administrative areas) should appear organized.
 - c) The analytical staff must be knowledgeable about the services to be provided.
 - d) The administrative staff must appear organized.
 - e) The laboratory must have the capacity to accommodate the proposed scope of work in terms of personnel and equipment.
3. Laboratory Performance Evaluation - Blind Check Samples: Before signing a contract or agreement, submit a set of blind check samples to the laboratory. A blind check sample is a sample in a real matrix (water, soil, sediment, etc.) that appears to be a real sample, except that the submitter has a list of the components and their known concentration values. Submit the sample(s) to the laboratory as a routine sample(s). Evaluate the results of the reported values against the certified values in the sample(s). The values must be within the laboratory's stated precision for the measurement.
4. Customer Satisfaction:
 - a) Obtain a list of current and previous clients.
 - b) Call several of the clients to determine their satisfaction with laboratory.

C. Contracting

1. Purpose:

- a) Provide a detailed list of the scope of services to be contracted.
- b) Include the purpose for which the data will be used (permit, compliance, etc.).

2. Key Contacts - Identify key contacts for both laboratory and client:

- a) Administrative: Dealing with billing, contract writing, invoicing, etc.
- b) Technical: Dealing with data, and quality control issues and problems.
- c) Sample Control: Dealing with scheduling, shipping supplies and sample receipt.

3. Anticipated Needs - Specify:

- a) The schedule of activities;
- b) The expected number of samples, matrices and tests; and
- c) Field support services, including containers, preservatives, cleaning and calibration services.

4. Expectations:

- a) Certification - The laboratory must maintain certification for the analyte, test methods and matrices to be performed. The laboratory must immediately notify clients if its certification status for any analytical method changes. The laboratory must flag and justify any results that were not generated in accordance with certification requirements.
- b) Analytical Expectations - Provide a copy of the permit, sampling plan or guidance document that outlines the regulatory agency's requirements, a list of approved analytical methods to be performed and the matrices for each method (included in Tables for specific sampling activities). Site and activity-specific information must be considered when deciding whether reporting down to MDL is needed. Highly contaminated samples will not be able to meet routine MDLs due to required dilution. Specify the expected turn-around time for the analyses. Specify the shipping schedule if sample containers or supplies are to be provided.
- c) Container/Equipment Services - State the scope of container and equipment services:
 - Pre-cleaned Containers: types and numbers
 - Preservatives
 - Equipment type and numbers.
 - Equipment calibration
- d) Quality Control - State adherence to both method and internal quality control requirements. Specify acceptable ranges for spikes, duplicates, surrogates and other QC measures if appropriate.
- e) Custody/Sample Tracking - State a time-period for retaining all records if greater than five years. Make arrangements for the transfer of records should the laboratory go out of business or transfer ownership before the records retention time period has lapsed. Specify the level of custody (routine, legal, etc.).
- f) Minimum Reporting Levels - Provide the laboratory with the minimum acceptable values to be reported. Describe contingencies if these levels cannot be met (i.e. explanation in case narrative).
- g) Reporting Format - All analytical reports issued by the laboratory must comply with N.C. DEQ DWM UST Section and DWR Laboratory Certification Program requirements. Specify whether the information must be provided as hardcopy or electronic or both. If electronic, specify the format for submission.

- h) Deliverables - Specify any deliverables needed over and above the basic report elements outlined in 2.6.1.
 - 1. Copies of all raw data and associated records, or
 - 2. Description of any modifications to methods.
- i) Subcontracting - The laboratory should inform the client **before** any analytical services are subcontracted to another laboratory. The laboratory **must** ensure that the subcontracted laboratory meets the same qualifications and requirements as the primary laboratory. A copy of the analytical report from subcontracted laboratories **must** be submitted to the client.
- j) Method Modifications - The laboratory must identify any modifications that have been made to the requested analytical methods. The client must be notified of any method modifications prior to use in the laboratory, and must provide written consent.
- k) Dilutions - Negotiate how multiple dilutions will be handled. They may be considered a separate analysis and therefore an additional cost. Agree to pay for the analysis of multiple dilutions only if:
 - 1. The sample concentration exceeds the calibration range and the laboratory was not aware of the expected sample concentration, or
 - 2. A dilution is required (and it is possible) to quantitate all contaminants of concern and/or achieve their routine laboratory lower reporting limits (i.e. where closure is possible).

NOTE: *Samples must never be diluted routinely or without cause. Dilutions may not provide the reporting limits necessary for compounds with cleanup standards near routine laboratory lower reporting concentrations or practical quantitation limits. However, the analysis of undiluted or less diluted samples in an attempt to obtain lower reporting limits may damage analytical instrumentation. If lower detection and reporting limits are needed, but are not possible due to interferences in the sample, an explanation in the case narrative with supporting documentation will be required.*

5. Penalties and Consequences

- a) Negotiate penalties or other consequences (no payment) for these problems:
 - 1. Failure to provide data or associated (expected) information,
 - 2. Failure to meet deadlines,
 - 3. Failure to provide acceptable data, and
 - 4. Failure to meet contract requirements.
- b) Consider these consequences:
 - 1. Costs of re-sampling,
 - 2. Fines incurred because of unacceptable data,
 - 3. Costs associated with having evaluated and/or processed unacceptable data, and
 - 4. Re-analysis costs (if re-analysis is due to laboratory error or failed QC).
- c) Reserve the right to reject data, however if any data are used, laboratory should be paid according to negotiated terms.

D. On-Going Evaluation

- 1. Monitor laboratory's performance against the specific contract requirements.
- 2. Continue to use blind QC samples as a measure of routine performance. Either submit vendor supplied samples, samples prepared to a known concentration, or split samples with another laboratory.

E. Data Review

The end user of the data must realize that the use of approved analytical methods by a certified laboratory cannot assure defensibility and data quality. The primary questions of data assessment should be, “Are the data effective for making the specified decisions, and are both the sampling and analytical documentation accompanying the data sufficient to establish that they are?” The end user of the data who has access to site specific information must follow the key to defensible environmental decision-making by openly acknowledging all underlying assumptions and managing all sources of uncertainty that can significantly impact the correctness of a decision to the degree feasible. Often a “weight of evidence” approach is needed because no single piece of information can provide definitive evidence given the complexities present in environmental systems. A number of questions must be asked to establish that data is of known quality and demonstrated as useful and reliable for the intended purpose. The concept of effective data embodies the principle that the information value of data (i.e., data quality) depends heavily upon the interaction between sampling design, analytical design, and the intended use of the data. Considering site-specific conditions, sample support, quality control, and data documentation assure the scientific defensibility of effective data.

1. Are the reported concentrations different from the routine (expected) levels?
2. Is the same value reported for the same analyte (except non-detects) in the same set of samples or over a historical period of time?
3. Do the parts add up to the total? Total values must be greater than or equal to dissolved values.
4. Are different but related analyses consistent? High turbidity and high total suspended solids. High turbidity and increased method detection limits for other tests.
5. Do results indicate a sample collection problem such as high dissolved oxygen in groundwater or high turbidity and elevated metals results?
6. Are the QC check samples within acceptable ranges and are the ranges reasonable?
7. Are non-detects reported correctly (should be a value with a qualifier code defined in a key)?
8. Over the history of laboratory use, were any QC problems reported?
9. Is there any laboratory or field blank contamination?
10. Do the reports contain all required information?

F. Ask questions if:

1. There are problems associated with the data review.
2. The QC check sample data are not acceptable.
3. The laboratory consistently reports the same QC failure.
4. The laboratory uses different methods than requested.
5. The laboratory subcontracts analyses without notifying the client.
6. The laboratory does not meet any of the contract requirements.
7. The laboratory misses holding times.
8. The laboratory fails to provide requested resource(s) (e.g., containers, calibration, etc.) in a timely manner.
9. There any doubts about the acceptability of the data.
10. Detection limits are above the expected values and the laboratory provides no reasonable explanation.

Note: There are two types of analytical lower limits: detection limits (DLs) and quantitation limits (QLs). The DL is the lowest concentration that can reliably be distinguished from zero, but is not

quantifiable with acceptable precision. At the DL, the analyte is proven to be present, but its reported concentration is an estimate. The QL is the lowest concentration that can be not only detected, but also quantified with a specified degree of precision. At the QL, the analyte is both proven present and measured reliably. Risk assessments often inappropriately report and handle data near the limits of detection. Common errors include (1) omission of detection limits, (2) failure to define detection limits that are reported, and (3) unjustified treatment of non-detects as zero. The practice of omitting information on DLs from risk assessments conceals important uncertainties about potential levels of undetected risk.

G. Scheduling Services

1. Notify the laboratory about the analytical and equipment needs at least a week in advance of the actual sampling trip.
2. Even if the trip is routine (monthly, weekly, quarterly compliance sampling), notify the laboratory of the number and type of samples to be collected, as well as any needs for specific reporting requirements. Also communicate expected contamination levels. This is important if a highly contaminated site is sampled.

2.1.2 Equipment and Sampling Containers

1. Equipment - Appropriate equipment must be selected based on the sampling source, the analytes of interest and the sampling procedure. The equipment construction must be consistent with the analytes or analyte groups to be collected (See Comprehensive Tables 13, 14, 15, 16 and 17). Equipment should be pre-cleaned before use in the field or equipment that has been certified clean by the vendor or laboratory should be used.
2. Dedicated Equipment Storage - All dedicated equipment (except dedicated pump systems or dedicated drop pipes) should be stored in a controlled environment. Equipment should be stored in an area that is located away from the sampling site. If equipment other than dedicated pumps or dedicated drop pipes are stored in monitoring wells, the equipment should be suspended above the formation water. The monitoring well should be securely sealed in order to prevent tampering between sampling events. All equipment (except dedicated pumps or drop pipes) should be decontaminated before use according to the applicable procedures outlined below.
3. Sample Containers - The analyses to be performed on the sample determine the construction of sample containers. (See Comprehensive Tables 13 and 14 for acceptable sample container, preservation and hold time options for approved analytical procedures). All containers and lids should be inspected for flaws (cracks, chips, etc.) before use. Sampling kits for sample collection and transport may be purchased from some commercial laboratories. The kits include all the items needed (sample containers, shipping cartons, etc.) for collection and shipment of samples.

The Interstate Technology and Regulatory Council (ITRC) has posted information about sampling and analytical methods for PFAS: <https://pfas-1.itrcweb.org/11-sampling-and-analytical-methods/> This is a developing area, please check with the regulators, laboratories and consultants for ongoing updates.

2.1.3 Decontamination of Field Equipment

Decontamination of sampling equipment and containers, before and after sampling, must be performed to ensure collection of representative samples and to prevent the potential spread of contamination. Decontamination of personal protective equipment prevents ingestion and absorption of contaminants. It must be done with a soap and water wash and deionized or distilled water rinse. Please note that sampling equipment and containers may be used that are certified pre-cleaned by the vendor or laboratory.

All previously used sampling equipment must be properly decontaminated before sampling and between sampling locations, to prevent the introduction of contamination into uncontaminated samples and to avoid cross-contamination of samples. Cross-contamination can be a significant problem when attempting to characterize extremely low concentrations of organic compounds or when working with soils that are highly contaminated.

Clean, solvent-resistant gloves and appropriate protective equipment must be worn by persons decontaminating tools and equipment.

2.1.3.1 General Requirements

1. Before using any equipment, clean/decontaminate all sampling equipment (pumps, tubing, cords, split spoons, etc.) that are exposed to the sample.
2. Before installing, clean (or obtain as certified pre-cleaned) all equipment that is dedicated to a single sampling point and remains in contact with the sample medium (e.g., permanently installed groundwater pump). If certified pre-cleaned equipment is used no cleaning is necessary.
 - a) Clean this equipment any time it is removed for maintenance or repair.
 - b) Replace dedicated tubing if discolored or damaged.
3. Clean all equipment in a designated area having a controlled environment (house, laboratory, or base of field operations) and transport it to the field, pre-cleaned and ready to use, unless otherwise justified.
4. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.
5. Whenever possible, transport sufficient clean equipment to the field so that an entire sampling event can be conducted without the need for cleaning equipment in the field.
6. Segregate equipment that is only used once (i.e., not cleaned in the field) from clean equipment and return to the in-house cleaning facility to be cleaned in a controlled environment.
7. Protect decontaminated field equipment from environmental contamination by securely wrapping and sealing with a suitable wrapping material, for example see the following:
 - a) Aluminum foil (commercial grade is acceptable),
 - b) Untreated butcher paper, or

- c) Clean, untreated disposable plastic bags. Plastic bags may be used for all analyte groups except volatile and extractable organics. Plastic bags may be used for volatile and extractable organics, if the equipment is first wrapped in foil or butcher paper, or if the equipment is completely dry.

2.1.3.2 Cleaning Reagents

Recommendations for the types and grades of various cleaning supplies are outlined below. The recommended reagent types or grades were selected to ensure that the cleaned equipment is free from any detectable contamination.

1. Detergents - Use Liquinox (or a non-phosphate equivalent) or Alconox (or equivalent). Liquinox (or equivalent) is recommended by EPA, although Alconox (or equivalent) may be substituted if the sampling equipment will not be used to collect phosphorus or phosphorus containing compounds.
2. Solvents
 - a) Use pesticide grade isopropanol as the rinse solvent in routine equipment cleaning procedures. This grade of alcohol must be purchased from a laboratory supply vendor. Rubbing alcohol or other commonly available sources of isopropanol **are not acceptable**.
 - b) Other solvents, such as acetone or methanol, may be used as the final rinse solvent if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics.
 1. **Do not use** acetone if volatile organics are of interest.
 2. Containerize all methanol wastes (including rinses) and dispose as a hazardous waste.
 - c) Pre-clean equipment that is heavily contaminated with organic analytes. Use reagent grade acetone and hexane or other suitable solvents. Use pesticide grade methylene chloride when cleaning sample containers. Store all solvents away from potential sources of contamination (gas, copier supplies, etc.).
3. Analyte-Free Water Sources
 - a) Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits. Maintain documentation (such as results from equipment blanks) to demonstrate the reliability and purity of analyte-free water source(s). The source of the water must meet the requirements of the analytical method and must be free from the analytes of interest. In general, the following water types are associated with specific analyte groups:
 1. Milli-Q (or equivalent polished water): suitable for all analyses.
 2. Organic-free: suitable for volatile and extractable organics.
 3. Deionized water: may not be suitable for volatile and extractable organics.
 4. Distilled water: not suitable for volatile and extractable organics, metals or ultra-trace metals.
 - b) Use analyte-free water for blank preparation and the final decontamination water rinse. In order to minimize long-term storage and potential leaching problems, obtain or purchase analyte-free water just prior to the sampling event. If obtained from a source (such as a laboratory), fill the transport containers and use the contents for a single sampling event. Empty the transport container(s) at the end of the sampling event. Discard any analyte-free

water that is transferred to a dispensing container (such as a wash bottle or pump sprayer) at the end of each sampling day.

4. Acids

- a) Reagent Grade Nitric Acid: 10 - 15% (one volume concentrated nitric acid and five volumes deionized water). Use for the acid rinse unless nitrogen components (e.g., nitrate, nitrite, etc.) are to be sampled. If sampling for ultra-trace levels of metals, use an ultra-pure grade acid.
- b) Reagent Grade Hydrochloric Acid: 10% hydrochloric acid (one volume concentrated hydrochloric and three volumes deionized water). Use when nitrogen components are to be sampled.
- c) If samples for both metals and the nitrogen-containing components are collected with the equipment, use the hydrochloric acid rinse, or thoroughly rinse with hydrochloric acid after a nitric acid rinse. If sampling for ultra-trace levels of metals, use an ultra-pure grade acid.
- d) Freshly prepared acid solutions may be recycled during the sampling event or cleaning process. Dispose of any unused acids according to local ordinances.

5. Reagent Storage Containers

The contents of all containers must be clearly marked.

- a) Detergents: Store in the original container or in a high density polyethylene (HDPE) or polypropylene container.
- b) Solvents
 - i. Store solvents to be used for cleaning or decontamination in the original container until use in the field. If transferred to another container for field use, use either a glass or Teflon (with the exception of PFAS contamination) container.
 - ii. Use dispensing containers constructed of glass, Teflon (with the exception of PFAS contamination) or stainless steel. Note: if stainless steel sprayers are used, any gaskets that contact the solvents must be constructed of inert materials.
- c) Analyte-Free Water:
 - i. Transport in containers appropriate for the type of water stored. If the water is commercially purchased (e.g., grocery store), use the original containers when transporting the water to the field. Containers made of glass, Teflon, polypropylene or HDPE are acceptable.
 - ii. Use glass or Teflon (with the exception of PFAS contamination) to transport organic-free sources of water on the site. Polypropylene or HDPE may be used, but are not recommended.
 - iii. Dispense water from containers made of glass, Teflon (with the exception of PFAS contamination), high density polyethylene (HDPE) or polypropylene.
 - iv. Do not store water in transport containers for more than three days before beginning a sampling event.
 - v. If working on a project that has oversight from EPA Region 4, use glass containers for the transport and storage of all water.
 - vi. Store and dispense acids using containers made of glass, Teflon (with the exception of PFAS contamination) or plastic.

2.1.3.3 Cleaning Sample Collection Equipment

1. On-Site/In-Field Cleaning:

- a) Cleaning equipment on site is not recommended because:
 - i. Environmental conditions cannot be controlled.
 - ii. Wastes (including solvents and acids) must be containerized for proper disposal.
- b) If equipment must be cleaned on the site or in the field, follow the appropriate cleaning procedure as outlined below in item 5 of this section. Ambient temperature water may be substituted in the hot, sudsy water bath and hot water rinses.

NOTE: *Properly dispose of all solvents and acids.*

- c) Rinse all equipment with water after use, even if it is to be field cleaned for other sites. Immediately rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples with water.

2. Heavily Contaminated Equipment

In order to avoid contaminating other samples, isolate heavily contaminated equipment from other equipment and thoroughly decontaminate the equipment before further use. Equipment is considered heavily contaminated if it:

- a) Has been used to collect samples from a source known to contain significantly higher levels than background,
- b) Has been used to collect free product, or
- c) Has been used to collect industrial products (e.g., pesticides or solvents) or their byproducts.

NOTE: *Cleaning heavily contaminated equipment in the field is not recommended.*

3. On-Site Procedures

- a) Protect all other equipment, personnel and samples from exposure by isolating the equipment immediately after use.
- b) At a minimum, place the equipment in a tightly sealed, untreated, plastic bag.
- c) Do not store or ship the contaminated equipment next to clean, decontaminated equipment, unused sample containers, or filled sample containers.
- d) Transport the equipment back to the base of operations for thorough decontamination.
- e) If cleaning must occur in the field, document the effectiveness of the procedure, collect and analyze blanks on the cleaned equipment.

4. Cleaning Procedures

- a) If organic contamination cannot be readily removed with scrubbing and a detergent solution, pre-rinse equipment by thoroughly rinsing or soaking the equipment in acetone.
- b) Use hexane only if preceded and followed by acetone.
- c) In extreme cases, it may be necessary to steam clean the field equipment before proceeding with routine cleaning procedures.
- d) After the solvent rinses (and/or steam cleaning), use the appropriate cleaning procedure.
 - i. Scrub, rather than soak, all equipment with sudsy water.
 - ii. If high levels of metals are suspected and the equipment cannot be cleaned without acid rinsing, soak the equipment in the appropriate acid. Since stainless steel equipment should not be exposed to acid rinses, do not use stainless steel equipment when heavy metal contamination is suspected or present.

- e) If the field equipment cannot be cleaned utilizing these procedures, discard unless further cleaning with stronger solvents and/or oxidizing solutions is effective as evidenced by visual observation and blanks.
- f) Clearly mark or disable all discarded equipment to discourage use.

5. General Cleaning in a Controlled Environment

Follow these procedures when cleaning equipment under controlled conditions. Check manufacturer's instructions for cleaning restrictions and/or recommendations.

- a) Procedure for Teflon, stainless steel and glass sampling equipment - This procedure must be used when sampling for ALL analyte groups: extractable organics, metals, nutrients, etc. or if a single decontamination protocol is desired to clean all Teflon, stainless steel and glass equipment.
 - i. Rinse equipment with hot tap water.
 - ii. Soak equipment in a hot, sudsy water solution (Liquinox or equivalent).
 - iii. If necessary, use a brush to remove particulate matter or surface film.
 - iv. Rinse thoroughly with hot tap water.
 - v. If samples for trace metals or inorganic analytes will be collected with the equipment that is not stainless steel, thoroughly rinse (wet all surfaces) with the appropriate acid solution.
 - vi. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water.
 - vii. If samples for volatile or extractable organics will be collected, rinse with isopropanol. Wet equipment surfaces thoroughly with free-flowing solvent. Rinse thoroughly with analyte-free water.
 - viii. Allow to air dry. Wrap and seal as soon as the equipment is air-dried.
 - ix. If isopropanol is used, the equipment may be air-dried without the final analyte-free water rinse; however, the equipment must be completely dry before wrapping or use.
 - x. Wrap clean sampling equipment according to the procedure described above.
- b) General Cleaning Procedure for Plastic Sampling Equipment
 - i. Rinse equipment with hot tap water.
 - ii. Soak equipment in a hot, sudsy water solution (Liquinox or equivalent).
 - iii. If necessary, use a brush to remove particulate matter or surface film.
 - iv. Rinse thoroughly with hot tap water.
 - v. Thoroughly rinse (wet all surfaces) with the appropriate acid solution.
 - vi. Check manufacturer's instructions for cleaning restrictions and/or recommendations.
 - vii. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water. Allow to air dry as long as possible.
 - viii. Wrap clean sampling equipment according to the procedure described above.

2.1.4 Calibration and Maintenance of Field Equipment

Preventive maintenance activities are necessary to ensure that the equipment can be used to obtain the expected results and to avoid unusable or broken equipment while in the field.

Equipment is properly maintained when:

- It functions as expected during mobilization, and
- It is not a source of sample contamination (e.g., dust).

1. Follow the manufacturer's suggested maintenance activities and document all maintenance.
2. Assign equipment a unique ID code (may be the name of the item, if there is only one).
3. Document the following information on each piece of equipment or instrumentation:
 - a) Identity (unique identifier code) and description (including software if used),
 - b) Manufacturer's name, model number and serial number (if applicable),
 - c) Calibration checks or other tasks that demonstrate that the equipment performs as expected,
 - d) Manufacturer's operating and maintenance instructions,
 - e) Written preventive maintenance schedule that includes the activity, and the frequency of each activity,
 - f) Date(s) of any preventive maintenance, repairs, malfunctions, etc., and
 - g) Name of person(s) performing the task(s).

2.1.4.1 Calibration

The calibration of field instruments is critical to obtain acceptable data. Improper calibration or failure of an instrument in the field might result in improper choice of sample locations, failure to detect contamination, and inefficient and inadequate segregation of clean soils from contaminated soils. Potentially much higher disposal or treatment costs may result.

To ensure that field instruments will be properly calibrated and remain operable in the field, the procedures set out in this section must be used.

1. If PID and FID field instruments are used, instruments must be calibrated before each testing session to yield "total organic vapors" in parts per million to a benzene equivalent. The PID instrument must be operated with a lamp source that is able to detect the contaminants of concern, operates at a minimum of 10.6 eV, and is capable of ionizing those contaminants of concern.
2. Field instruments must be calibrated onsite.
3. All standards used to calibrate field instruments must meet the minimum requirements for source and purity recommended in the equipment's operation manual.
4. If the instrument's operation manual recommends specific calibration requirements for other criteria in calibrating the instrument (such as pH, conductivity, temperature, etc.), those criteria must be adhered to.
5. Acceptance criteria for calibration must be determined depending on the potential contaminant(s). Criteria must be within the limits set in the manufacturer's operations manual.
6. The dates, times and results of all calibrations and repairs to field instruments must be recorded in the field record and the instrument's log.
7. All users of the instrument must be trained to properly calibrate and operate the instrument. Equipment users must read the operation manual before initial use.

2.1.4.2 Maintenance

1. At a minimum, operation, maintenance, and calibration must be performed in accordance with the instrument manufacturer's specifications.
2. All users of the instrument must be trained in routine maintenance, including battery and lamp replacement, lamp and sensor cleaning, and battery charging.

3. Each instrument's operation and maintenance manual must be present at the site.
4. All field instruments must be inspected before departure for the site and on site.
5. Instrument battery charges must be inspected far enough ahead of time to bring the instrument up to full charge before departure for the site.
6. At a minimum, a source of extra batteries and lamps (if applicable) must be readily available.

2.1.5. Preparation of Methanol Preserved Vials

1. Preparation by Sampler

- a) Add 5 ml of **purge and trap grade methanol** to 40-mL VOA vials with open top screw caps and Teflon-coated septa. Add 25 ml if using 60-mL VOA vials. Include a methanol trip blank and a glass jar, or other appropriate container per sample set, for dry weight determination.
- b) Seal the vials with the screw caps and septum seals.
- c) Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label, (The weight of any markings added to the label in the field is negligible.)
- d) Calibrate an electronic balance and weigh each prepared sample vial to within 0.01 gram. Record the tare weight and the date on the label. Store the prepared vials at 4°C and protect them with sealable plastic bags for transport to the field.

2. Laboratory prepared methanol preserved bottles - It is recommended that sample containers be ordered and received immediately prior to each job. It is not recommended that a supply of sample containers with methanol be stored unused for long periods of time. The concern with stockpiling sample containers containing methanol is the volatilization of the methanol. This may magnify the contamination levels in the sample and subsequently result in higher cleanup costs. It is recommended that samples collected in sample containers that contain methanol within 14 days of the date the vial was prepared and weighed in the laboratory. When the laboratories weigh the sample and record the weight on the vials, they should also record the date on the vials.

NOTE: *Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of ≥ 0.01 g, if weighed in the laboratory or ≥ 0.2 g, if weighed in the field) should not be used to collect samples. An alternative to field weighing is to mark the meniscus of the methanol after addition to the VOA vial for field personnel to visually inspect and note any apparent loss during shipment. If field personnel are concerned with methanol loss, during shipment to the field and return, individual vials can be re-weighed.*

3. Alternate Storage of Laboratory prepared methanol preserved bottles - All sample containers containing methanol should be on ice when shipped from the laboratory, before sample collection. The sample containers may be kept in a refrigerator when they are received from the laboratory until they are taken to the field for sample collection. In other words, all sample containers containing methanol should be kept cold from the time they leave the laboratory until the time they return to the laboratory. The reason for keeping the methanol cold at all times is to reduce methanol vaporization, which may magnify the concentration of contaminants in the samples and subsequently increase cleanup costs.

NOTE: *An alternative to shipping on ice and/or field weighing is to mark the meniscus of the methanol after addition to the VOA vial for field personnel to visually inspect and note any apparent loss during shipment. If sample containers containing methanol are not shipped from*

the laboratory on ice, or if the meniscus of the methanol is not marked for field personnel to visually inspect for apparent loss, then they must be re-weighed to ensure vials have not lost methanol. If field personnel are concerned with methanol loss, individual vials can be re-weighed. Vials with reduction in weight of >0.01 g, if weighed in the laboratory, or > 0.2 g, if weighed in the field, should not be used to collect samples.

The collection devices which are not shipped with methanol (i.e., specially-designed, approved, airtight sampling devices and pre-weighed empty VOAs) do not need to be shipped on ice from the laboratory. They should be put on ice prior to sample collection and after collection for shipping to the laboratory. Collecting the sample in a chilled collection device will reduce volatilization and will provide a more accurate result of the amount of contamination in the soil.

2.2 Health and Safety

All local, state and federal requirements relating to health and safety should be implemented. All local, state and federal requirements pertaining to the storage and disposal of any hazardous or investigation-derived wastes should be complied with. (Investigation-derived waste is defined as water, soil, drilling mud, decontamination wastes, discarded personal protective equipment, etc. from site investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, abandonment and other investigative activities.)

The UST Section recommends wearing protective gloves when conducting all sampling activities. Gloves serve to protect the sample collector from potential exposure to sample constituents; minimize accidental contamination of samples by the collector; and preserve accurate tare weights on pre-weighed sample containers. They must be worn unless:

- The sample source is considered to be non-hazardous.
- The samples will not be analyzed for trace constituents.
- The part of the sampling equipment that is handled without gloves does not contact the sample source.

Gloves must not come into contact with the sample or with the interior or lip of the sample container. Clean, new, unpowdered and disposable gloves must be used. Various types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection. Materials potentially present in concentrated effluent can pass through certain glove types and be absorbed through the skin. Vendor catalogs provide information about the permeability of different gloves and the circumstances under which the glove material might be applicable. The powder in powdered gloves can contribute significant contamination. Powdered gloves are not recommended unless it can be demonstrated that the powder does not interfere with the sample analysis.

Gloves should be changed after preliminary activities, such as pump placement; after collecting all the samples at a single sampling point; or if torn or used to handle extremely dirty or highly contaminated surfaces. All used gloves should be properly disposed of as investigation derived wastes.

All investigation-derived waste (IDW) should be properly managed so contamination is not spread into previously uncontaminated areas. Investigation-derived waste includes all water, soil, drilling mud, decontamination wastes, discarded personal protective equipment, etc. from site

investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, abandonment, and other investigative activities. All investigation-derived waste that is determined to be RCRA-regulated hazardous waste must be managed according to the local, state and federal requirements.

IDW that is not a RCRA-regulated hazardous waste but is contaminated above the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for groundwater quality must be properly disposed of.

The "*UST Section Assessment Guidelines*" or "*UST Section Corrective Action Guidelines*" should be consulted for information regarding the disposal of drill cuttings/mud and purged well water as a result of field environmental investigations. Under 15A NCAC 2T .0113(a)(10) [Waste Not Discharged to Surface Waters – Permitting by Regulation], "drilling muds, cuttings and well water from the development of wells" are deemed permitted in accordance with NCGS 143-215.1(d), and thus no individual Division permit need be issued. If the drill cuttings/mud or purged well water is contaminated with hazardous waste, contact the DWM Hazardous Waste Section (919 707-8200) for disposal options. Section 2.4.4 includes decision flow diagrams with guidance on the proper disposal for drill cuttings/mud and purged well water that result from field investigations and/or cleanup operations.

All containers holding IDW should be maintained in good condition and inspected periodically for damage. All containers holding IDW must have all required labeling (DOT, RCRA, etc.) clearly visible.

2.3 Preservation, Holding Times and Container Types

1. Samples must be preserved by one of the options indicated in Section 2.4.4 for soils, 2.4.5 or 2.4.6 for water and 2.4.9 for air. (See Table 8 for soils preservation and Table 9 for water preservation.) The holding times and preservation options listed in the above-referenced tables supersede those in individual analytical methods.
2. The preservation protocols in the referenced tables require immediate preservation. "Immediate" is defined as "within 15 minutes of sample collection." All preservation must be performed onsite unless samples can be transported to the laboratory within 15 minutes of collecting the sample. The preservation options for volatiles soil samples for the inhibition of biodegradation are an exception. These options are detailed in Tables 8 and in Section 2.4.4.13.
3. 24-hour composite water samples are the exception to the "15-minute" criterion. If the sample requires thermal preservation, the automatic sampler must be able to maintain the required temperature by packed ice or refrigeration. When chemical preservation is also required, the preservation process must begin within 15 minutes of the last collected sample.

4. The pH of samples must be checked at these recommended intervals:
 - a) During the first sampling event at a particular site, all samples must be checked that are pH adjusted except volatile organics. and
 - b) During subsequent visits to a particular site, at least one sample must be checked per parameter group that must be pH-adjusted.

2.4 Sample Collection

2.4.1. Contamination Prevention

- a) Cross contamination or environmental contamination when collecting samples should be prevented.
 - i. If possible, samples should be collected in sequence from the least contaminated sampling location (or background sampling location, if applicable) to the most contaminated sampling location.
 - ii. The ambient or background samples should be collected first, and stored in separate ice chests or separate shipping containers within the same ice chest (i.e. untreated plastic bags).
 - iii. Samples from flowing water should be collected in sequence from least contaminated to most contaminated, unless sedimentation will be an issue.
- b) Highly contaminated samples (concentrated wastes, free product, etc.) or samples suspected of containing high concentrations of contaminants should not be stored or shipped in the same ice chest or shipping container with other environmental samples.
 - i. These sample containers should be isolated by sealing them in separate, untreated plastic bags immediately after collecting, preserving, labeling, etc.
 - ii. A clean, untreated plastic bag should be used to line the ice chest or shipping container.
- c) All sampling equipment should be thoroughly decontaminated and transported in a manner that does not allow it to become contaminated. Arrangements should be made ahead of time to decontaminate any sampling or measuring equipment that will be reused when taking samples from more than one well. Field decontamination of sampling equipment will be necessary before sampling each well to minimize the risk of cross contamination. Decontamination procedures should be included in reports as necessary. (See Section 2.1.3 for decontamination procedures.) Sampling equipment and containers may be used that are certified pre-cleaned by the vendor or laboratory.

When collecting aqueous samples, the sample collection equipment should be rinsed with a portion of the sample water before taking the actual sample. Sample containers do not need to be rinsed.

Sample containers with pre-measured preservatives must not be rinsed. Also, sample containers used when collecting samples of petroleum hydrocarbons and oil and grease must not be rinsed

- d) All fuel-powered equipment should be placed away from, and downwind of, any site activities (e.g., purging, sampling, decontamination).
 - i. If field conditions preclude such placement, the fuel source(s) should be placed as far away as possible from the sampling activities and the conditions described in the field notes.
 - ii. Fuel for vehicles and equipment should be handled prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves.
 - iii. All fuels should be dispensed downwind and well away from the sampling activities. Gloves and other protective equipment should be disposed of downwind, and well away from the sampling activities.
 - iv. If sampling at active gas stations, sample collection activities should stop during fuel deliveries.

2.4.2 Sample Labels

At a minimum, the label or tag must identify the sample with the sample identification (sample ID), date of collection, method of analysis requested, collector, and preservative(s). Additional information (i.e., a location identification code) may be included on the tag or label. The label should be filled out before placing it on the vial/bottle. The label should be placed on the bottle before collecting the sample. The following information should be printed legibly on the label with indelible ink:

- a) Date and Time of sampling
- b) Method of Analysis required [i.e., VOCs (EPA 8260), Metals]
- c) Sample collector
- d) Preservative used, if any (i.e., HCl, Na₂S₂O₃, HNO₃, ice, etc.)
- e) Sampling location and site name

2.4.3 Sample Collection Order

Unless field conditions justify other sampling sequences, collect samples in the following order:

- a) Volatile Organics and Volatile Inorganics
- b) Extractable Organics, Petroleum Hydrocarbons, Aggregate Organics and Oil and Grease
- c) Total Metals
- d) Inorganic Non-metallic, Physical and Aggregate Property, and Biological samples
- e) Microbiological samples

NOTE: *If the pump used to collect groundwater samples cannot be used to collect volatile or extractable organic samples then the volatile and extractable organics should be collected after withdrawing the pump and tubing.*

2.4.4 Collecting Soil Samples

Soil samples are collected for a variety of purposes. A systematic sampling approach must be used to assure that sample collection activities provide usable data. Sampling must begin with an evaluation of background information, historical data and site conditions. There are three major activities requiring the collection of soil samples: closure, soil remediation permitting and assessment and corrective action.

2.4.4.1. Equipment to Collect Soil Samples

Equipment and materials that may be used to collect soil samples include disposable plastic syringes and other “industry-standard” equipment and materials that are contaminant-free. Non-disposable sampling equipment must be decontaminated between each sample location.

2.4.4.2 Equipment for Reaching the Appropriate Soil Sampling Depth

Samples may be collected using a hollow stem soil auger, Shelby tube or split-spoon sampler. These sampling devices may be used as long as an effort is made to reduce the loss of contaminants through volatilization. Obtain a sufficient volume to ensure the samples are collected without volatilization and disturbance to the internal structure of the samples. Samples should be collected from cores of the soil. Non-disposable sampling equipment must be decontaminated between each sample location.

- a) Shovels and Diggers - Used for soils from approximately 12 inches bgs until impractical
 - i. Dig a hole or trench to the required depth.
 - ii. Follow the general sample collection procedures outlined above.
- b) Backhoe - Used for soils from approximately 12 inches bgs until impractical.
 - i. Dig a trench to the appropriate depth.
 - ii. Expose the sample, in the trench, by using a pre-cleaned spoon, spatula or equivalent to clean away the soil that came in contact with the backhoe bucket.
 - iii. Use a second pre-cleaned utensil to actually collect the sample from the trench.
 - iv. Follow the general sample collection procedures outlined above.
- c) Hand Augers and Corers - Suitable to reach soils from approximately 12 inches bgs until impractical.
 - i. Push and rotate the auger into the soil until the auger is filled.
 - ii. Addition of a non-contaminating sleeve may allow an undisturbed soil sample to be obtained. The device consists of a standard auger head with a removable sleeve, which is inserted into the auger barrel.
 - iii. Remove the sleeve from the auger and cap.
 - iv. If the auger hole is prone to collapse due to low soil cohesion, insert a temporary, rigid PVC casing into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced. After collecting the samples, remove the temporary casing (if used) and fill the hole.
 - v. Remove the sample from the sampler by pushing or scraping the soil with an appropriate, pre-cleaned utensil into an appropriately pre-cleaned tray or aluminum foil.
 - vi. Remove any portion of the sample that has been disturbed and discard.
 - vii. Follow the general sample collection procedures outlined above.

NOTE: *If a confining layer has been breached during sampling, grout the hole to land surface with Type-1 Portland cement.*

- d) Split Spoon Sampler - Suitable for reaching soils from approximately 12 inches to depths greater than 10 feet bgs. A split spoon sampler, useful for sampling unconsolidated soil, consists of two half cylinders (spoons) that fit together to form a tube approximately two feet in length and two inches in diameter. The cylindrical arrangement is maintained by a

retaining head and bit rings that screw on at each end of the split spoon. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.

- i. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.
 - ii. Insert a catcher device in the head ring to prevent the loss of unconsolidated sample material during recovery.
 - iii. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.
 - iv. If the recovery is enough to accommodate discarding a portion of the sample, discard the top and bottom two to three inches of the sample.
 - v. For volatile organic compounds, collect the sample immediately from the center portion of the split spoon using the procedures described below in Section E.
 - vi. For other analyses, slice the sample from the center portion of the split spoon. Use a clean, decontaminated utensil.
 - vii. Select an appropriate, pre-cleaned sampling device and collect the sample.
 - viii. Transfer the sample to the appropriate sample container.
 - ix. Clean the outside of the sample container to remove excess soil.
 - x. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}$ and complete the field notes.
- e) Direct Push Rigs – These may be used for depths greater than 10 feet bgs. The clear liners are used with direct push rigs. This method is appropriate only for unconsolidated materials. The sampling depth that can be achieved varies depending on the rig and the lithologies that are encountered.
- i. Place the liner inside the metal probe rod.
 - ii. Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod.
 - iii. Advance the rod a full rod length.
 - iv. Retrieve the rod.
 - v. Remove the point holder.
 - vi. Remove the liner.
 - vii. Slice the liner to expose the soil.
 - viii. After the liner has been sliced, follow the procedures outlined above for the split spoon sampler. If needed collect volatile organic samples immediately after the liner is sliced. If samples for organic vapor analysis screening are required, collect them by slicing the sample(s). Use a clean, decontaminated utensil and place the samples in 8-ounce (preferred) or 16-ounce jars. Immediately cover the opening with aluminum foil and screw on the lid ring. If the contamination is derived from petroleum products, it is acceptable to use a clean, gloved hand to transfer the sample(s) to the sample container(s).
 - ix. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.
 - x. Select an appropriate, pre-cleaned sampling device and collect the sample.
 - xi. Transfer the sample to the appropriate sample container.
 - xii. Clean the outside of the sample container to remove excess soil.

- xiii. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}$, and complete the field notes.
- f) Shelby Tube Sampler - The Shelby tube sampler is used to sample unconsolidated soil. It consists of a tube approximately 30 inches long and two inches (or larger) in diameter. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter, which allows attachment to the drilling rig assembly. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly from the borehole.
- i. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or push manually with a sledge hammer.
 - ii. Remove the tube from the sampler head.
 - iii. Extrude the sample from the Shelby tube.
 - iv. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.
 - v. Collect samples for volatile organics immediately from the center portion of the Shelby tube.
 - vi. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.
 - vii. Transfer the sample to the appropriate sample container.
 - viii. Clean the outside of the sample container to remove excess soil.
 - ix. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}$, and complete the field notes.
- g) Core Barrel - A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be collected. The core barrel is a cylinder approximately three feet long and two inches in diameter. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soil as the drilling rods are rotated.
- i. Retrieve the sample core by unscrewing the head ring. Slide the sample into a pre-cleaned container.
 - ii. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.
 - iii. Remove the sample from the sampler (corer) with a pre-cleaned tool.
 - iv. Transfer the sample to the appropriate sample container.
 - v. Clean the outside of the sample container to remove excess soil.
 - vi. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}$ C and complete the field notes.

2.4.4.3 Soil Sample Collection Procedures for Laboratory Samples

The number and type of laboratory samples collected depends on the purpose of the sampling activity. The three major activities are closure, soil remediation permitting and assessment and corrective action. There are different requirements associated with initial characterization, routine monitoring and permit completion for soil remediation permitting and assessment and corrective action. The soil remediation technology employed and the contamination source material also affect the number and type of laboratory samples needed. Samples analyzed with field screening devices may not be substituted for required laboratory samples.

General Sample Collection - When collecting samples from potentially contaminated soil, care should be taken to reduce contact with skin or other parts of the body. Disposable gloves should be worn by the sample collector and should be changed between samples to avoid cross-contamination. Soil samples should be collected in a manner that causes the least disturbance to the internal structure of the sample and reduces its exposure to heat, sunlight and open air. Likewise, care should be taken to keep the samples from being contaminated by other material at the site or by other samples collected at the site. When sampling occurs over an extended period of time, it is necessary to insure that the samples are collected in a comparable manner.

- a) All samples must be collected with disposable or clean tools that have been decontaminated as outlined in Section 2.1.3.
- b) Disposable gloves must be worn and changed between sample collections.
- c) Sample containers must be filled quickly.
- d) Soil samples must be placed in containers in the order of volatility, for example, volatile organic aromatic samples must be taken first, gasoline range organics next, then heavier range organics, and finally soil classification samples.
- e) Containers must be quickly and adequately sealed, and rims must be cleaned before tightening lids. Tape may be used only if known not to affect sample analysis.
- f) Sample containers must be clearly labeled.
- g) Samples must have the appropriate laboratory specified preservative present in the container prior to addition of the sample. Unless specified otherwise, at a minimum, the samples must be immediately cooled to $4^{\circ} \pm 2^{\circ} \text{C}$ and this temperature must be maintained throughout delivery to the laboratory.

2.4.4.4. Composite Sample Collection Procedures

Characterizing stockpiled soil is necessary for several reasons. It is used to determine whether treatment or disposal of the soil is needed, to assist with selection of treatment or disposal methods and to establish baseline data for use in evaluating the effectiveness of treatment.

Composite sampling is used for soil samples collected to characterize stock piles, for soil remediation permitting and soil disposal. Composite samples are prepared differently depending on the analysis required and the relative tendency of the contaminant analyzed to vaporize.

Soil samples taken to characterize stock piles, monitor land application or containment and treatment sites, must be obtained from freshly uncovered soil at different depths from each of two randomly selected soil borings made to the maximum depth of waste incorporation.

Soil samples collected from excavation equipment buckets for field screening must be obtained away from the bucket edges. At least six inches of soil must be removed immediately before collection.

If soil samples are collected from a soil boring, the drill hole must be advanced to the desired depth. Then the center rods of the auger must be withdrawn from the drill hole and the plug and pilot bit must be removed from the center rods. The sampler must also be attached to the correct length of drill rod and driven ahead of the auger flights in order to collect a relatively undisturbed sample. After the split spoon or Shelby tube has been retrieved back out of the boring, the desired sample section must be immediately removed from the sampling device. Only soil from the middle portion of the spoon may be used for samples. Soil from the very ends of the spoon must be

discarded as it often contains disturbed soils. A clean sampling tool must be used to quickly collect the sample from the undisturbed portion with a minimum of disturbance.

- a) Container(s) should be labeled as unmixed composite (i.e. for TPH DRO, SW-846 8270 or metals), or as primary samples to be composited by the laboratory (i.e. for TPH GRO or SW-846 8260).
- b) Sampling points from which to collect each aliquot should be selected.
- c) Using the appropriate sampling technique, equal aliquots (same sample size) should be collected from each location and placed in an approved sample container (See Table 8).
- d) An appropriate preservation option should be used and selected in coordination with the analytical laboratory.
- e) The laboratory should be notified that the sample is a composite sample.

Volatile Samples

- a) Samples collected for volatile organic analysis cannot be dried, ground or mixed if they are to reflect the concentrations found in the soil. Since methanol preserved samples lend themselves to composite sampling techniques, the methanol preservation technique will be required to preserve primary samples that are to be composited and analyzed for volatiles analysis (i.e. TPH GRO or SW-846 8260).
- b) The six primary samples collected for volatiles analysis shall be collected and preserved in **separate** VOA vials. The primary samples are methanol extracted. Representative portions of the methanol extracts must be composited by the analytical laboratory using methods that minimize volatile organic loss. See below for details on Volatiles Soil Sample Preservation.

Non-volatile Samples

- a) Immediately upon removal from the ground, each soil sample must be placed in the appropriate container (See Table 8). Once placed in the container, each sample should be immediately capped and sealed. Be sure that the sample containers are labeled in accordance with Section 4.2, and in such a manner that prevents the labels from peeling away from the containers during transport.
- b) Collect six non-volatile representative portions of soil at the same time the six volatile primary samples are collected. The non-volatile representative portions are to be added to a single, unpreserved container. A separate portion may be required for the determination of soil moisture content and dry weight correction factors.
- c) Aliquots of soil samples for semi-volatile (i.e. TPH DRO or SW-846 8270D) or metals analysis should not be mixed before containerizing. The laboratory should be notified that the sample is an unmixed composite sample, and should be requested to thoroughly mix the sample before sample preparation or analysis.

2.4.4.5 Characterization of Stock-pile or Excavation Sampling

Characterizing stockpiled or excavated soil is necessary for several reasons. It is used to determine whether treatment or disposal of the soil is needed, to assist with selection of treatment or disposal methods and to establish baseline data for use in evaluating the effectiveness of treatment. Soil samples for laboratory analysis must be collected from each stockpile or as soil is excavated.

- a) One composite sample shall be collected per 200 cubic yards for initial characterization of petroleum contaminated soil. For sites containing less than 200 cubic yards, a minimum of one composite sample shall be taken. Results should be secured prior to acceptance at

treatment-sites. These composite samples must be analyzed in accordance with the rule as outlined in “Guidelines for Ex Situ Petroleum Contaminated Soil Remediation,” to provide a complete chemical analysis of the typical petroleum contaminated soil to be remediated. Where required, samples must also be analyzed for a determination of hazardous waste constituents using the TCLP described in 40 CFR 261.24 (EPA SW-846/Method 1311 (TCLP) metals). TCLP analysis will be required for all permit applications to dispose of petroleum contaminated soil unless the criteria that relate to the determination of hazardous waste characterization can be met.

- b) For a stock-pile sample, each composite sample must be collected from two soil cores composed of a vertical column of soil collected using a soil auger, Shelby tube or split-spoon sampler. Sample collection and preservation must meet the guidelines in Table 8.
- c) For excavation sampling, a composite sample must be collected as described above.

2.4.4.6 Routine Monitoring and Permit Completion Sampling

The number of soil samples collected from the treated soil are determined below and as required by 15A NCAC 2L .0106 (f).

a) Soil Sampling for Land Application

- i. Two composite soil samples must be collected from each acre. If the site is less than one acre, collect from each application area following application. For routine monitoring samples the purpose is to monitor the progress of treatment or for dedicated sites the availability for reapplication of petroleum-contaminated soil. For permit completion samples the purpose is to determine if soil has been adequately treated and to document that the treatment goals have been reached.
- ii. Each composite sample must be collected from two soil cores composed of a vertical column of soil. The cores must extend from land surface to the maximum depth of waste incorporation. Collect samples using a soil auger, Shelby tube or split-spoon sampler. Sample collection and preservation must meet the guidelines in Table 8.
- iii. A composite sample is comprised of six primary soil samples, three from core A and three from core B. Primary samples are taken at different depths from each of the two randomly selected soil borings. Each primary sample shall be collected in the field and be analyzed as a composite by a DWR-certified laboratory.

b) Soil Sampling for Containment and Treatment Technologies

The following describes the minimum sampling requirements, however the permit may require more extensive sampling.

- i. One composite sample shall be collected per 200 cubic yards or per source (whichever is smaller), at the application site, at a minimum of six month intervals. For sites containing less than 200 cubic yards, a minimum of one sample shall be taken every six months. For routine monitoring samples, the purpose is to monitor the progress of treatment. For permit completion samples, the purpose is to determine if soil has been adequately treated and to document that the treatment goals have been reached.
- ii. Each composite sample must be collected from two soil cores composed of a vertical column of soil. Collected the cores using a soil auger, Shelby tube or split-spoon sampler. Sample collection and preservation must meet guidelines in Table 8.
- iii. A composite sample is comprised of six primary soil samples. Three from core A and three from core B. Primary samples are taken at different depths from each of the two, randomly

selected soil borings. Each primary sample shall be collected in the field and be analyzed as a composite by a DWR-certified laboratory.

2.4.4.7 Soil Field Screening Procedures

Field screening is the use of portable devices capable of detecting petroleum contaminants on a real-time basis or by rapid field analytical technique. Field screening should be used to help assess locations where contamination is most likely to be present.

When possible, field-screening samples should be collected directly from the excavation or from the excavation equipment's bucket. If field screening is conducted only from the equipment's bucket, then a minimum of one field screening sample should be collected from each 10 cubic yards of excavated soil (See Figures 2 and 3). If instruments or other observations indicate contamination, soil should be separated into stockpiles based on apparent degrees of contamination. At a minimum, soil suspected of contamination must be segregated from soil determined to be free of contamination.

1. Field screening devices

Many field-screening instruments are available for detecting petroleum contaminants in the field on a rapid or real-time basis. Acceptable field screening instruments must be suitable for the contaminant being screened. The procedure for field screening using photoionization detectors (PIDs) and flame ionization detectors (FIDs) is described below. If other instruments are used, a description of the instrument or method and its intended use must be provided to the UST Section. Whichever field screening method is chosen, its accuracy must be verified throughout the sampling process. Use of appropriate standards that match the use intended for the data. **Unless the UST Section indicates otherwise (such as noncommercial, nonregulated heating oil USTs), wherever field screening is recommended in this document, instrumental or analytical methods of detection must be used, not olfactory or visual screening methods.**

2. Headspace analytical screening procedure for field screening (semi-quantitative field screening)

The most commonly used field instruments for UST site assessments are FIDs and PIDs. When using FIDs and PIDs, use the following headspace screening procedure to obtain and analyze field-screening samples;

- a) Partially fill (one-third to one-half) a clean jar or clean resealable plastic bag with the sample to be analyzed. The total capacity of the jar or bag may not be less than eight ounces (app. 250 ml), but the container should not be so large as to allow vapor diffusion and stratification effects to significantly affect the sample;
- b) If the sample is collected from a split spoon or a direct push sleeve, it must be transferred to the jar or bag for headspace analysis immediately after opening the split-spoon. If the sample is collected from an excavation or soil pile, it must be collected from freshly uncovered soil.
- c) If a jar is used, its top must be quickly covered with clean aluminum foil or a jar lid; screw tops or thick rubber bands must be used to tightly seal the jar. If a resealable plastic bag is used, it must be quickly sealed shut.
- d) Headspace vapors must be allowed to develop in the container for at least 10 minutes but no longer than one hour. Containers must be shaken or agitated for 15 seconds at the beginning and end of the headspace development period to assist volatilization. Temperatures of the headspace must be warmed to at least 40° F (approximately 5° C) with instruments calibrated for the temperature used.

- e) After headspace development, the instrument sampling probe must be inserted to a point about one-half the headspace depth. The container opening must be minimized and care must be taken to avoid the uptake of water droplets and soil particulates.
- f) After probe insertion, the highest meter reading must be taken and recorded. This will normally occur between two and five seconds after probe insertion. If erratic meter response occurs at high organic vapor concentrations or conditions of elevated headspace moisture, a note to that effect must accompany the headspace data.
- g) Calibration of PID and FID field instruments must follow the procedures outlined in 2.1.4.
- h) All field-screening results must be documented in the field record or log book.

2.4.4.8 Soil Background Sampling

This guidance is primarily designed to assist technical staff in the UST Section to evaluate naturally occurring inorganics (Lead and Chromium) in soils at sites with concentrations above currently established MSCCs. One of the most essential issues for remediating soil contaminated with naturally occurring compounds is the determination of remediation standards for those compounds. A remediation standard for each naturally occurring contaminant in soil may be adjusted based on background conditions. There are some national databases, (e.g., USGS studies), which can provide a sense of the likely background ranges of element concentrations in soils unaffected by most man-made activities. However, local variations and analytical method differences make site-specific sample data collection preferable, and required, in some situations. Whether remediation is required for the site in question is determined by comparison to either naturally occurring background conditions or through risk assessment. In most cases, a sufficient number of samples will not be available to conduct a statistical analysis.

In some situations, non-statistical approaches may be considered more appropriate to compare site contaminant levels to background constituent levels when selecting potential chemicals of concern. There are two basic applications for non-statistical twice background criterion. One requires the collection of a minimum of four site-specific background samples. The other allows the use of historical data to establish the background concentration for the specific soil type in question. The application allowed depends on whether there is evidence of a release of contamination in addition to the naturally occurring inorganics. For sites with no evidence of a release other than the naturally occurring inorganics, may use historical data. Other sites require site-specific background samples before the background levels can be established.

Both non-statistical approaches are easily used and easily reviewed methods for background screening. Generally, statistical analysis requires more extensive sampling and accompanying expense. The final decision of whether to accept statistical comparison of site sampling data with background concentrations for the purpose of selecting chemicals of concern is at the discretion of the UST Section regional office supervisor.

1. Data Evaluation and Collection for Background Concentrations of Naturally Occurring Inorganics in Soils.

- a) Evaluate the available data to determine which of the two non-statistical approaches may be used. If soil characterization or previous knowledge of the site indicates that the area of potential contamination is located in an area containing fill material, comparison to historical data is not recommended. In this case a minimum of four site specific background samples are recommended for initial evaluation. If background sample results are highly variable, additional samples will be required to evaluate background levels and

non-statistical approaches may not be used. The UST Section should be consulted before using any type of statistical approach for comparison to background.

- b) If there is no evidence of a release other than naturally occurring inorganics above MSCCs, comparison to background ranges from historical databases may be used. To obtain comparable historical data to establish the “background” concentration, characterize soil type and use the depth of the samples of concern.
- c) If there is evidence of a release other than the naturally occurring inorganics, a minimum of four samples must be used to establish "background" in soils. Background samples must be collected in an area that has not been impacted by environmental contamination from the site and from the same depth as site samples to which the background samples will be compared.
- d) Background soil should be the same type of soil horizon material as the comparison sample. Multiple soil horizons should have "background" established separately (e.g., a minimum of four samples per each soil unit). This will not be necessary unless more than one soil horizon has shown potential impact by contamination at or above relevant MSCCs. Evaluate the soil texture (percent silt, sand, clay), soil pH and cation exchange capacity to confirm that the background samples are from comparable soil types. Many of these soil parameters can be obtained by contacting the local Natural Resources Conservation Service Office (NRCS) and requesting a soil survey report for the County where the site is located. By using the soil survey report, field personnel can evaluate how the soils were originally classified and gain access to average values for the soil series located at the site. Background samples must be analyzed using total constituent analysis.

Soil Texture Classification Chart

Texture Class	% Sand	% Silt	% Clay
Sands	> 85	<15	0
Loamy Sands	70 - 85	< 30 (silt + clay)	< 30 (silt + clay)
Sandy Loams	50 - 70	< 50	< 20
Loam	<52	28-50	7 - 27
Silt Loam	Trace	50 - 80	12 - 27
Silt	Trace	> 80	<12
Sandy Clay Loams	45 - 72	< 28	20 - 35
Clay Loams	20 - 45	15 - 40	27 - 40
Silty Clay Loams	< 20	60 - 73	27 - 40
Sandy Clays	45-65	0	35 - 55
Silt Clays	0	40 - 60	40 - 60
Clays	<15	< 40	> 40

NOTE: *In order to obtain appropriate background samples, the sampler may be required to collect samples from a nearby, offsite location. The soil in the area under investigation and the soil in the background area may have both been similarly affected by a source unrelated to the UST unit (e.g. air emissions, wastewater sludge operations, etc.). Concentrations found in the background soil may still be acceptable in this situation. Situations will exist where the surrounding area has historically been affected by sources outside of the site under investigation. **Specific guidelines cannot be outlined for every site condition encountered; therefore, evaluations must be made on a site-by-site basis.***

- e) In general, background samples should be eliminated and replaced with a like number of samples from uncontaminated areas if:
 - i. the background samples are taken in areas known or suspected to be contaminated by a source which did not similarly affect the area under investigation, or
 - ii. the background samples have possibly been affected by activities conducted in the area undergoing investigation.

- f) Areas to avoid for background sampling include but are not limited to:
 - i. past waste management areas where solid and/or hazardous wastes or wastewater may have been placed on the ground, areas of concentrated air pollutant deposition (from a definable localized source), or areas affected by the runoff;
 - ii. roads, roadsides, parking lots, areas surrounding parking lots or other paved areas, railroad tracks, railway areas or other areas affected by their runoff;
 - iii. storm drains or ditches presently or historically receiving industrial or urban runoff;
 - iv. spill areas, material handling areas, such as truck or rail car loading areas, or near pipelines, fill areas and other areas as determined by the UST Section.

- g) Detection Limits - Detection limits should be reviewed before the sampling and analysis is completed to ensure that they do not exceed preliminary remediation goals. This should not be a problem for chromium and lead.

2. Evaluation of Potential Chemicals of Concern

Potential chemicals of concern are chemicals that are carried through the risk assessment process. EPA Region 4 has designed a screening process to identify chemicals of concern, which are most likely to contribute to an unacceptable risk.

NOTE: This selection process is not designed to eliminate any naturally occurring substance in the subsurface soils as a chemical of potential concern to protect groundwater. The potential for chemicals in subsurface soils to leach to the groundwater must be considered for high or intermediate risk sites as required by title 15A NCAC 2L .0408 and .0508. Based on review of site specific information, limited site assessment or interim corrective actions, the Department may determine that the discharge or release poses no significant risk to human health or the environment and reclassify the site as low risk. If additional information becomes available, the Department may reclassify the risk posed by a release.

- 3. Selection Process for Chemicals of Concern - The process of selecting chemicals of concern includes screening that utilizes risk-based concentrations. For naturally occurring substances that exceed the established standard:
 - a) The data for each chemical should be sorted by medium. For this purpose, surface soil and subsurface soil should be considered as separate media. Surface soil is considered the top 12 inches. Identify the background data for each medium.
 - b) For any data which have qualifiers, decide if the qualified data should be retained. Do not eliminate data based on estimated qualifiers.

- 4. Summarize the following parameters for the naturally occurring substance under review for both the closure and background samples.
 - a) Frequency of detection
 - b) Range of detection limits
 - c) Arithmetic average background concentration

- d) Arithmetic average of detected concentrations
 - e) Range of detected concentrations
 - f) Risk-based screening value
 - g) Basis for elimination or selection as a chemical of concern
5. Eliminate chemicals as chemicals of concern based on comparison to blanks. See Section 2.6.3 for an explanation of blank evaluation criteria.
 6. Compare maximum detected concentrations in surface soils to the appropriate maximum soil contaminant concentration (MSCC).
 - a) Eliminate the chemical as a chemical of concern for human exposures if the concentration is less than the appropriate residential or industrial/commercial MSCCs.
 - b) Eliminate the chemical as a chemical of concern for protection of groundwater if the concentration is less than the soil to groundwater MSCC.
 7. For naturally occurring inorganics, compare the on-site maximum detected concentration to the average site-specific background concentration. Eliminate the chemical as a chemical of concern if it is less than the background level. One background sample, if elevated, is usually not adequate for comparison or elimination purposes.
 - a) Specific guidelines as to the number of background samples cannot be outlined for every site; therefore, evaluations must be made on a site by site basis.
 - b) A minimum of four background samples is recommended. Additional samples may be needed if background concentrations are highly variable or if sampling locations are determined to be inappropriate.
 - c) To use the background criterion, the maximum detected concentration on site is compared to the average background concentration. If the maximum detected concentration is greater than the average background concentration, the chemical is included as a chemical of concern.

NOTE: *If a background sample result is not detected at or above the limit of quantitation, half the limit of quantitation is substituted when averaging background concentrations.*

2.4.4.9 Soil Sample Preparation for Laboratory Samples

The type of sample container used depends on the type of analysis performed. First, determine the type of sampling activity, type(s) of contaminants expected and the proper analytical method(s) established in Tables 3, 4, 6, and 8. Approved sample containers and preservation requirements for the specified methods sampling activities are included in Table 8. Be sure to consult with the selected laboratory for specific needs and requirements prior to sampling.

1. Sampling kits for sample collection and transport may be requested from commercial laboratories. The kits include all the items needed (sample containers, shipping cartons, etc.) for the collection and shipment of samples. If these services are used, carefully follow the instructions provided and do not discard any preservative that may have been added to the containers. If a customized kit provided by the laboratory is not used, only new containers of the appropriate sampling/analytical method should be used. Check with the laboratory that will be running the analysis about appropriate sample containers and preservation requirements for each method. **If proper sampling and QA/QC protocols are not followed, the Department may consider the results invalid.**

2. Select sampling equipment based on the type of sample to be collected and the analytes of interest. Choose soil sampling locations so that a representative portion of the soil is collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.
3. If background sampling is warranted and feasible, select an upgradient, undisturbed location to obtain the background samples. See 2.2.5.2 Soil Background Sampling above for additional guidance.
4. Do not collect samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, unless they are used to characterize waste for disposal.
5. Do not use samples that are collected for geological/lithological or vapor meter determinations for chemical analyses.
6. Equipment and Supplies:
 - a) All equipment must be constructed of materials consistent with the analytes of interest. Refer to Table 17 for selection of appropriate equipment.
 - b) For information on sampling equipment cleaning requirements, see Section 2.1.3.
 - c) For information on preservation and holding time requirements, see Table 8.

2.4.4.10 Surface Soil Samples

Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches, however the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12-inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical. Stainless steel spoons may be used for surface soil sampling to depths of approximately 6-inches below ground surface where conditions are generally soft and non-indurated and there is no problematic vegetative layer to penetrate. The following EPA webpage presents more detailed information regarding sample collection and various drilling techniques:

<https://www.epa.gov/quality/soil-sampling>, Effective date June 11, 2020 and Review date June 11, 2024

2.4.4.11 Surface Soil Sampling Procedures

1. Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.
2. Remove leaves, grass and surface debris from the area to be sampled.
3. Collect samples for volatile organic analyses as described below.
4. Select an appropriate, pre-cleaned sampling device and collect the sample.
5. Transfer the sample to the appropriate sample container.
6. Clean the outside of the sample container to remove excess soil.
7. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}\text{C}$.

2.4.4.12 Subsurface Soil Sampling

The interval begins at approximately 12 inches below ground surface.

1. Collect samples for volatile organic analyses as described below.

2. For other analyses, select an appropriate, pre-cleaned sampling device and collect the sample.
3. Transfer the sample to the appropriate sample container.
4. Clean the outside of the sample container to remove excess soil.
5. Label the sample container, place on wet ice to preserve to $4^{\circ} \pm 2^{\circ}\text{C}$.

2.4.4.13 Collection and Preservation of Volatiles Soil Samples

SW-846 Method 5035 outlines a variety of options to collect and preserve volatiles soil samples. The method describes a closed-system purge and trap process for the analysis of low level volatile organic compounds (VOC) in soils. The method also describes preparation techniques for high level VOC soil samples to be purged using method 5030 and analyzed by appropriate determinative methods. Additional collection and preservation techniques have also been shown to effectively reduce VOC losses attributable to volatilization and biodegradation. These additional collection and preservation options are acceptable, in addition to the preservation options outlined in Method 5035 and MADEP VPH, for samples analyzed and reported to the N.C. DEQ Division of Waste Management UST Section.

1. Low and High Level Methods - Here is a brief description of the low and high level methods, with hold times for various preservation options.

a) Low Level ($\leq 200 \mu\text{g}/\text{kg}$ volatile organics) - SW-846 method 5035 includes a low-level closed-system purge and trap method with a preservation option using sodium bisulfate. However, soils high in carbonate minerals may effervesce on contact with the acidic sodium bisulfate preservation solution. Samples suspected to contain high levels of carbonates require a test sample to check for effervescence. If a rapid or vigorous reaction occurs, discard the sample. Then collect the samples in vials that do not contain the sodium bisulfate preservative solution. Preservation options and acceptable hold times include:

- i. A sample field preserved with sodium bisulfate has a 14-day holding time.
- ii. An unpreserved sample (no acid) must be analyzed within 48 hours.
- iii. An unpreserved sample in a vial with premeasured analyte-free water must be analyzed within 48 hours.
- iv. Holding times for unpreserved samples in vials with premeasured analyte-free water may be extended to 14 days if the laboratory freezes the samples to $-12 \pm 2^{\circ}\text{C}$ within 48 hours of sample collection.
- v. If transported to the laboratory in a sealed coring device or pre-weighed VOA vial without chemical preservative, the samples must be analyzed within 48 hours.
- vi. Holding times for sealed coring device samples may be extended to 14 days if the laboratory extrudes the sample into sodium bisulfate, or freezes it to $-12 \pm 2^{\circ}\text{C}$ within 48 hours.

b) High Level ($> 200 \mu\text{g}/\text{kg}$ volatile organics) - SW-846 method 5035 includes a high level soil method for samples where the closed-system purge and trap sample introduction technique is not appropriate. For reporting to the N.C. DEQ Division of Waste Management UST Section, the high level method will apply to soil samples that require volatiles analysis for TPH or VPH and composite samples required by soil remediation permits. The exact weight of the soil samples and the volume of the methanol used for extraction must be known by the laboratory. Preservation options and acceptable hold times include:

- i. A sample field preserved with methanol has a 14-day holding time.
- ii. An unpreserved sample (no methanol) must be analyzed within 48 hours.

- iii. Holding times for unpreserved samples in a vial may be extended to 14 days if the laboratory adds methanol within 48 hours and refrigerates at 4°C + 2°C.
- iv. Holding times for unpreserved samples in vials may be extended to 14 days if the laboratory freezes the samples to -12 + 2°C within 48 hours of sample collection.
- v. If transported to the laboratory in a sealed coring device, the samples must be analyzed within 48 hours.
- vi. Holding times for sealed coring device samples may be extended to 14 days if the laboratory either freezes the device at -12 + 2°C within 48 hours of sample collection or extrudes the sample into methanol, and refrigerates it at 4°C.

2. Basic Options for Collection and Preservation - Acceptable options to collect and preserve volatiles soil samples to be analyzed and reported to the NCDEQ Division of Waste Management UST Section are detailed below. Coordination between sampling personnel and the laboratory is essential prior to selection of the sampling containers and preservation options. *Each option involves collection of a small soil plug with a coring device, followed by a single transfer to a pre-weighed VOA vial.* See Tables 6 and 8 for a summary of approved sample containers preservation options and hold times specific to the type of sampling activity.

For closure or assessment and corrective action sampling activities, collect duplicate samples for TPH Gasoline or MADEP VPH aromatics/aliphatics analysis (both for high level purge and trap). Collect three replicate samples for constituent specific EPA 5035/8260B analysis (two for low level closed-system purge and trap and one for high level purge and trap). For soil remediation sampling activities, collect six primary samples for each composite sample required for a soil remediation permit. Composite volatiles soil samples must be methanol extracted and therefore require one of the preservation options for the high level method above. The primary samples are to be composited by the laboratory after the extraction procedure. Portions of methanol extract from the six primary soil samples are to be composited by a DWR certified laboratory using methods that minimize volatile organic loss. Label containers as primary samples for composite and notify the laboratory that the six primary samples are to be composited.

Be sure to collect an additional aliquot of sample for dry weight determination. This sample may also be used for soil characterization (i.e., effervescence check) for low concentration samples suspected to contain carbonate minerals. Here are the sample collection and preservation options.

a) Field Preservation with Sodium Bisulfate or Methanol

PERFORMANCE STANDARD: Obtain and store an undisturbed soil sample by collecting a small soil plug with a coring device. Follow collection with a single transfer to a pre-weighed VOA vial that contains chemical preservative to inhibit biodegradation.

- i. Choose appropriate sampling containers that are labeled, pre-weighed and preserved with sodium bisulfate or purge and trap (or equivalent) grade methanol. All sampling containers should have an open-top screw cap with Teflon-coated silicone rubber septa or equivalent. Sodium bisulfate preserved VOA vials must also contain a stirring device.
- ii. A 10-30 ml disposable syringe with the end cut off is recommended to obtain an undisturbed soil sample from freshly exposed soils. Sample coring devices designed to estimate the appropriate weight of soil by volume may be used. Extrude the soil into sample container and avoid splashing liquid out of the container.

- iii. Use a clean brush or paper towel to remove soil particles from the threads of the sample container and screw cap. Tightly apply and secure the screw cap. Gently swirl the sample to break up soil aggregate, if necessary, until soil is covered with sodium bisulfate or methanol. DO NOT SHAKE.
- iv. Immediately place containers in a cooler with ice for storage in an upright position. Sample containers can be placed in separate zip-lock bags in case of leakage during transport. Transport samples to the analytical laboratory using the appropriate chain-of-custody procedures and forms.

b) Use of Pre-weighed VOA Vials

PERFORMANCE STANDARD: Obtain and store an undisturbed soil sample by collecting a small soil plug with a coring device. Follow collection with a single transfer to a pre-weighed VOA vial. Immediately seal the airtight VOA vial and analyze it within 48 hours or preserve it within 48 hours to inhibit biodegradation.

- i. Obtain labeled, pre-weighed VOA vials for the collection and air-tight storage of at least five grams of soil. Pre-weighed vials for analysis by low level, closed system, purge and trap must include a stirring device (i.e., magnetic stir bar).
- ii. In the field, obtain an undisturbed sample from a freshly exposed soil. Collect soil core samples with disposable cut off syringes or other coring devices and extrude them immediately into pre-weighed VOA vials. Immediately seal the VOA vial, and place it in a cooler. Transport samples to the analytical laboratory using the appropriate chain-of-custody procedures and forms.
- iii. Samples must either be analyzed or be preserved in the laboratory within 48 hours of collection to inhibit biodegradation. To do this, samples must be either frozen to $-12 \pm 2^{\circ}\text{C}$ or preserved with an appropriate chemical preservative (sodium bisulfate or purge and trap (or equivalent) grade methanol) at the laboratory within 48 hours of sampling. The laboratory may add sodium bisulfate or purge and trap (or equivalent) grade methanol to the sample after it has been enclosed in a pre-weighed VOA vial. It may be added by using a syringe and puncturing the septum with a 23 or smaller gauge needle. Samples immersed in methanol should not be stored for more than two days in VOA vials that have punctured septa. When methanol is introduced through the septum via a needle, the septum should be replaced if the sample is archived. A ratio of 1 ml methanol to 1 gram soil will minimize the dilution factor. In no case, however, shall the level of soil in the laboratory container exceed the level of methanol (i.e., the soil must be completely immersed in methanol).

c) Use of a Sealed-Tube Sampling/Storage Device

PERFORMANCE STANDARD: Obtain an undisturbed soil sample and immediately seal it in an airtight container for shipment to a laboratory. Follow collection with a single transfer to a pre-weighed VOA vial. Either analyze or preserve to inhibit biodegradation within 48 hours.

- i. Obtain pre-cleaned and/or disposable samplers/containers that allow the collection and air-tight storage of at least five grams of soil.
- ii. In the field obtain the appropriate number (two or three) of undisturbed, co-located samples from a freshly exposed soil. Immediately seal containers and place them in a cooler. Obtain an extra sample in an empty VOA vial to determine soil moisture

content. Transport samples to the analytical laboratory using the appropriate chain-of-custody procedures and forms.

- iii. Samples must either be analyzed or be preserved in the laboratory to inhibit biodegradation within 48 hours of collection. To do this, samples must be either frozen to $-12 \pm 2^{\circ}\text{C}$ or preserved chemically at the laboratory within 48 hours of sampling. Samples are extruded and immersed in an appropriate chemical preservative (sodium bisulfate or purge and trap (or equivalent) grade methanol) at the laboratory within 48 hours of sampling, at a ratio of 1 mL preservative to 1-gram soil. In no case, however, shall the level of soil in the laboratory container exceed the level of liquid (i.e., the soil must be completely immersed).

NOTE: *Documentation must be provided/available on the ability of the sampler/container to provide an air-tight seal in a manner that results in no statistically significant loss of volatile hydrocarbons for at least 48 hours. Check with the laboratory before collecting samples to make sure that the necessary equipment is available to open the devices and to ensure that the 48-hour holding time can be met.*

Regardless of which sampling option is used, the desired ratio of methanol-to-soil should be 1-ml methanol to 1-gram soil, $\pm 25\%$. A soil sample with a minimum weight of four grams is required. The exact weight of the soil sample and the volume of methanol must be determined by the laboratory when calculating and reporting soil concentration data.

NOTE: *The 1:1 soil to solvent ratio appears to work well for solid samples (e.g., sandy soil) that do not expand to soak up the methanol when it is added. On the other hand, many samples, such as those with a high organic content, may expand and soak up the methanol, making it impossible to remove the methanol extract from the sample container for purging purposes. If the solvent does not cover all of the soil, volatile analytes will escape into the headspace and not be captured in the aliquot of solvent removed from the vial for analysis. **In all cases, the soil sample in the vial must be completely covered by methanol.***

3. Container Preparation

- a) All containers must be cleaned using the appropriate sample container cleaning procedures for volatile organics **or be certified as pre-cleaned** (See Section 2.1.3).
- b) Sample Vials - If samples are to be field preserved, vials must be provided with all reagents, stirring devices, labels and vial caps to be used during sample analysis. These vials must be pre-weighed and records must be maintained so that there is an unambiguous link between the tare weight and the filled sample vial.
- c) Pre-weighed vials should be handled with gloves. Protect the sample containers and labels from moisture by using sealable plastic bags.

4. Collection Tips

- a) The pre-weighed sample vials (when used), may contain a pre-measured amount of liquid. The laboratory must weigh the vials before sending them into the field, and then again after receipt. To collect useable samples:
 - i. Do not lose any of the liquid either through evaporation or spillage.
 - ii. Do not use a vial if some of the liquid has spilled, or if it appears that some has leaked during transport.
 - iii. Use the laboratory-supplied container label for identification information.

- iv. Do not apply any additional labels or chain of custody seals to the pre-weighed container.
 - v. Do not interchange vial caps or septa.
 - vi. Protect the sample containers and labels from moisture by using sealable plastic bags.
- b) Transport the pre-labeled and weighed vials, either empty or containing the appropriate volume of chemical preservative, to the field in a cooler with ice. Take precautions to avoid exposing the vials to exhaust fumes or other known airborne contaminants at all times. Use disposable gloves while handling pre-weighed vials and collecting samples.
- c) The sampler must be proficient in estimating the 5-gram weight necessary for each sample. Use sample coring devices designed to estimate the appropriate weight of soil by volume. If an accurate estimate of the 5-gram sample size is desired before sampling begins. Use a balance with a sensitivity of 0.1 gram. Calibrate an electronic balance to weigh 5.0 grams of soil, to the nearest 0.1 gram, for a 40-mL vial. The calibration check weight should approximate the expected combined weight of the closed sample vial that contains methanol and the soil sample. Check the balance calibration before each day's use. Use a set of weights that have been calibrated against NIST-traceable weights at least annually. Use a 10-30 mL disposable plastic syringe with the end cut off or a special soil sampler to obtain an undisturbed soil sample from freshly exposed soil. Collect trial samples with the syringe. Weigh and note the length of the soil column in the syringe to determine the length of soil corresponding to 5.0 ± 0.5 grams. Discard all trial samples.
- d) Minimize exposure to air by obtaining the sample directly from the sample source. Use a coring device or a commercially designed sampling tool. Collect soil samples within a few minutes, at most, from the time when the surface of the soil has been exposed to the atmosphere.
- i. The sample collection device must be designed to fit tightly against the mouth of the vial or be small enough to be inserted into the vial.
 - ii. EnCore or equivalent sampling devices may be used. If the sampling device is transported to the laboratory with a sample, make sure the seals are intact, especially if collecting samples from sandy soils.
 - iii. Disposable "industry standard" coring devices or plastic syringes with the syringe end cut off prior to sampling may only be used once per sampling location.
- e) Collect a soil sample, open a sample vial, and immediately extrude the soil sample into the vial. Avoid splashing methanol if using the methanol preserved VOAs. Use a clean brush or paper towel to quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw cap. Wipe off any soil adhering to the outside of the vial. For methanol preserved vials, gently swirl the sample to break up any soil clumps, if necessary, but do so only until the soil is covered with methanol. **The soil sample must be completely covered by methanol.**
- f) Optionally, weigh the sealed vial containing the soil sample to ensure that 5.0 ± 0.5 grams of sample were added. Obtain a duplicate sample in the same manner. If too little soil is collected, detection limits may be increased. If too much soil is collected in methanol preserved vials, the soil may not be adequately covered with methanol.

- g) Collect a bulk soil sample in a vial without preservatives so that the laboratory can calculate moisture content and dry weight. A portion of this may also be used for soil characterization if field preservation was not performed.

NOTE: *Because water is completely miscible with methanol, naturally occurring moisture contained in the soil sample may result in under-reporting of the true, dry weight VOC concentrations. In general, every percent of moisture (by weight) present in a soil sample will result in a negative bias of about 1 percent. It is the responsibility of the data user to determine the significance of this effect on a site-specific basis. However, moisture contents less than 25% by weight are generally not considered a significant concern. No data adjustments are to be done by the laboratory relative to this issue, although laboratories may reference this phenomenon as a reason for low surrogate recoveries in the case narrative, when appropriate.*

- h) Place each vial in a separate sealable plastic bag immediately after collection. Then, quickly place each sample container in a cooler with plenty of ice. Transport the samples to the analytical laboratory as soon as possible. Use chain-of-custody procedures and forms.

2.4.5 Surface Water Samples

Surface water samples should be collected at the point of discharge, upstream of the point of discharge, and downstream of the point of discharge. The order of sample collection should be based on the potential for downstream sedimentation due to wading and the potential for cross contamination. Surface water for VOCs should be collected first. The following EPA webpage presents more detailed information regarding surface water sample collection:

<https://www.epa.gov/quality/surface-water-sampling>, February 28, 2013

2.4.5.1 Collecting Surface Water Samples

The following topics include acceptable equipment selection and equipment construction materials; and standard grab, depth-specific and depth-composited surface water sampling techniques.

1. When using watercraft, take samples near the bow, away and upwind from any gasoline outboard engine. Orient watercraft so that bow is positioned in the upstream direction. When wading, collect samples upstream from the body. Avoid disturbing sediments in the immediate area of sample collection. Collect water samples prior to taking sediment samples when obtaining both from the same area (site). Unless dictated by permit, program or order, sampling at or near man-made structures (e.g., dams, weirs or bridges) may not provide representative data because of unnatural flow patterns. Collect surface water samples from downstream towards upstream.
2. Equipment and Supplies - Use sampling equipment constructed of materials consistent with the analytes of interest. Refer to Tables 13 and 15 for material selection. Select equipment based on the analytes of interest, the specific equipment use and the available equipment. Refer to Table 14 for selection of appropriate equipment. For information on sample containers, preservation and holding time requirements, see Table 9. For information on sampling equipment cleaning requirements, see Section 2.4.7. Sampling events will most frequently employ the suction lift sample gathering system.

2.4.5.2 Surface Water Sampling Techniques

Use the following protocols when collecting surface water samples. Adhere to all general protocols applicable to aqueous sampling when following the surface water sampling procedures addressed below.

1. Manual Sampling: Use manual sampling for collecting grab samples for immediate in-situ field analyses. Use manual sampling in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to observe and/or note unusual conditions.
 - a) Surface Grab Samples - Do not use sample containers containing premeasured amounts of preservatives to collect grab samples. If the sample matrix is homogeneous, then the grab method is a simple and effective technique for collection purposes. If homogeneity is not apparent, based on flow or vertical variations (and should never be assumed), then use other collection protocols. Where practical, use the actual sample container submitted to the laboratory for collecting samples to be analyzed for oil & grease, volatile organic compounds (VOCs) and microbiological samples. This procedure eliminates the possibility of contaminating the sample with an intermediate collection container. The use of unpreserved sample containers as direct grab samplers is encouraged since the same container can be submitted for laboratory analysis after appropriate preservation. This procedure reduces sample handling and eliminates potential contamination from other sources (e.g., additional sampling equipment, environment, etc.).
 - i. Grab directly into sample container
 - ii. Slowly submerge the container, opening neck first, into the water.
 - iii. Invert the bottle so the neck is upright and pointing towards the direction of water flow (if applicable). Allow water to run slowly into the container until filled.
 - iv. Return the filled container quickly to the surface.
 - v. Pour out a few mL of sample away from and downstream of the sampling location. This procedure allows for the addition of preservatives and sample expansion. Do not use this step for volatile organics or other analytes where headspace is not allowed in the sample container.
 - vi. Add preservatives, securely cap container, label and complete field notes.
 - iv. If sample containers are attached to a pole via a clamp, submerge the container and follow steps 3 – 5 but omit steps 1 and 2.
 - b) Sampling with an Intermediate Vessel or Container: If the sample cannot be collected directly into the sample container to be submitted to the laboratory, or if the laboratory provides pre-preserved sample containers, use an unpreserved sample container or an intermediate vessel (e.g., beakers, buckets or dippers) to obtain the sample. These vessels must be constructed appropriately, including any poles or extension arms used to access the sample location.
 - i. Rinse the intermediate vessel with ample amounts of site water prior to collecting the first sample.
 - ii. Collect the sample as outlined above using the intermediate vessel.
 - iii. Use pole mounted containers of appropriate construction to sample at distances away from shore, boat, etc. Follow the protocols above to collect samples.

- c) **Peristaltic Pump and Tubing:** The most portable pump for this technique is a 12-volt peristaltic pump. Use appropriately pre-cleaned, silastic tubing in the pump head and attach polyethylene, Tygon, etc. tubing to the pump (see restrictions listed in Tables 14 and 16). This technique is not acceptable for Oil and Grease, EPH, VPH or VOCs. Extractable organics can be collected through the pump if flexible interior-wall Teflon, polyethylene or polypropylene tubing is used in the pump head or if used with an organic trap setup.
- i. Lower appropriately pre-cleaned tubing to a depth of 6 – 12 inches below water surface, where possible.
 - ii. Pump 3 – 5 tube volumes through the system to acclimate the tubing before collecting the first sample.
 - iii. Fill individual sample bottles via the discharge tubing. Be careful not to remove the inlet tubing from the water.
 - iv. Add preservatives, securely cap container, label and complete field notes.
- d) **Mid-Depth Grab Samples:** Mid-depth samples or samples taken at a specific depth can approximate the conditions throughout the entire water column. The equipment that may be used for this type of sampling consists of the following depth-specific sampling devices: Kemmerer, Niskin, Van Dorn type, etc. Pumps with tubing or double check-valve bailers may also be used. Certain construction material details may preclude its use for certain analytes (see Tables 14 and 16). These are acceptable for all analyte groups without restriction.
- i. Measure the water column to determine maximum depth and sampling depth prior to lowering the sampling device.
 - ii. Mark the line attached to the sampler with depth increments so that the sampling depth can be accurately recorded.
 - iii. Lower the sampler slowly to the appropriate sampling depth, taking care not to disturb the sediments.
 - iv. At the desired depth, send the messenger weight down to trip the closure mechanism.
 - v. Retrieve the sampler slowly.
 - vi. Rinse the sampling device with ample amounts of site water prior to collecting the first sample. Discard rinsate away from and downstream of the sampling location.
 - vii. Fill the individual sample bottles via the discharge tube.
- e) **Double Check-Valve Bailers:** Collect samples using double check-valve bailers if the data requirements do not necessitate a sample from a strictly discrete interval of the water column. Bailers with an upper and lower check-valve can be lowered through the water column. Water will continually be displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. Sampling with this type of bailer must follow the same protocols outlined above, except that a messenger weight is not applicable. Although not designed specifically for this kind of sampling, a bailer is acceptable when a mid-depth sample is required. This sampler does not perform as well as the devices described above or the pump and tubing described in the next section.
- i. As the bailer is dropped through the water column, water is displaced through the body of the bailer. The degree of displacement depends upon the check-valve ball movement to allow water to flow freely through the bailer body.
 - ii. Slowly lower the bailer to the appropriate depth. Upon retrieval, the two check-valves seat, preventing water from escaping or entering the bailer.

- iii. Rinse the sampling device with ample amounts of site water prior to collecting the first sample.
 - iv. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described above.
- f) Peristaltic Pump and Tubing: The most portable pump for this technique is a peristaltic pump. Use appropriately pre-cleaned, silastic tubing in the pump head and attach high density polyethylene (HDPE), or other appropriate tubing to the pump (see restrictions listed in Tables 15 and 16). Use of peristaltic pumps should be limited to aquifers with shallow groundwater depths, and should not be used when the groundwater depth exceeds 25 feet below ground surface.
- i. Measure the water column to determine the maximum depth and the sampling depth.
 - ii. Tubing will need to be tied to a stiff pole or be weighted down so the tubing placement will be secure. Do not use a lead weight. Any dense, non-contaminating, non-interfering material will work (brick, stainless steel weight, etc.). Tie the weight with a cord (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.
 - iii. Turn the pump on and allow several tubing volumes of water to be discharged before collecting the first sample.
 - iv. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described above.

2.4.6 Groundwater Samples

Groundwater samples are collected to identify, investigate, assess and monitor the concentration of dissolved contaminant constituents. To properly assess groundwater contamination, first install sampling points (monitoring wells, etc.) to collect groundwater samples and then perform specific laboratory analyses. All monitoring wells should be constructed in accordance with 15A NCAC 2C .0100 and sampled as outlined in this section.

Groundwater samples should be collected from the least contaminated well to the most contaminated well. Depending upon site conditions, several different groundwater techniques may be considered (i.e., bailing, low flow sampling, and passive samplers). Passive sampling techniques require approval from the appropriate regional office incident manager who will likely request a comparison of passive sampling analytical results to active sampling analytical results.

Field parameters must be collected during well development to ensure formation stabilization prior to the collection of groundwater samples. The applicable parameters may vary depending on the constituents of concern and any required monitoring for active remediation systems as defined in Title 15A NCAC 2C .0225.

The groundwater samples should be collected in the order of analyses presented in Section 2.4.6.

Groundwater samples may be collected from a number of different well types and locations. Sampling equipment requirements and techniques vary according to well construction and scope of work.

- Wells without Plumbing: These wells require equipment to be brought to the well to purge and sample unless dedicated equipment is placed in the well.
- Wells with In-Place Plumbing: Wells with in-place plumbing do not require equipment to be brought to the well to purge and sample. In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply wells. They are generally found at wellfields, industrial facilities and private residences.

The following website presents more detailed information regarding groundwater sample collection:

https://www.epa.gov/sites/production/files/2017-07/documents/groundwater_sampling301_af.r4.pdf,

2.4.6.1 Groundwater Sampling Techniques

1. Purge wells using the techniques outlined in Section 2.4.6.4.
2. Replace the protective covering around the well if it is soiled or torn after completing the purging operations.

3. Equipment Considerations

- a) Some pumps may be used to sample groundwater. Follow all notes and restrictions as defined in Tables 13 and 14 and discussed in Section A when using pumps to collect samples.

NOTE: *The only pumps that are currently approved for use to collect volatile organic samples through the pump are: stainless steel and Teflon variable speed submersible pumps, stainless steel and Teflon or polyethylene variable speed bladder pumps, and permanently installed PVC bodied pumps, as long as the pump remains in contact with the water in the well at all times. Pumps with Teflon are not suitable for assessing PFAS contamination.*

- b) Collect the sample into the sample container from the sampling device. Do not use intermediate containers.

- c) In order to avoid contaminating the sample or loss of analytes from the sample:

- i. Handle the sampling equipment as little as possible.
- ii. Minimize the equipment that is exposed to the sample.

- d) Dedicated Sampling Equipment

- i. Whenever possible, use dedicated equipment. It significantly reduces the chance of cross-contamination.
- ii. Dedicated equipment is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).
- iii. All material construction and restrictions from Tables 15 and 16 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.

iv. **Cleaning/Decontamination**

- Clean or make sure dedicated pumps are clean before installation. They do not need to be cleaned prior to each use, but must be cleaned if they are withdrawn for repair or servicing.
- Clean or make sure any permanently mounted tubing is clean before installation.
- Change or clean tubing when the pump is withdrawn for servicing.
- Clean any replaceable or temporary parts as specified in Section 2.1.3.
- Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.
- Clean or make sure dedicated bailers are clean before placing them into the well.
- Collect an equipment blank on dedicated bailers before introducing them into the water column.
- Suspend dedicated bailers above the water column if they are stored in the well.

2.4.6.2 Water Level Collection

Water levels should be collected at the beginning of groundwater sampling activities prior to purging. Monitoring well caps should be removed and the groundwater allowed to equilibrate beneath the site. Groundwater level measurements should be collected from all existing site monitoring within a total of 2 hours to ensure that the resulting groundwater elevation map represents a “snap shot” in time.

2.4.6.3 Water Level and Purge Volume Determination

Collect groundwater samples from fresh water from the aquifer. The amount of water that must be purged from a well is determined by the volume of water and/or field parameter stabilization.

1. General Equipment Considerations - Selection of appropriate purging equipment depends on the analytes of interest, the well diameter, transmissivity of the aquifer, the depth to groundwater and other site conditions.
 - a) Use of a pump to purge the well is recommended unless no other equipment can be used or there is non-aqueous phase liquid in the well, or non-aqueous phase liquid is suspected to be in the well.
 - b) Bailers must be used with caution since improper bailing:
 - i. Introduces atmospheric oxygen, which may precipitate metals (e.g., iron) or cause other changes in the chemistry of the water in the sample (e.g, pH, redox, TDS).
 - ii. Agitates groundwater, which volatilize dissolved-phase hydrocarbons thus introducing a bias to laboratory analyses.
 - iii. Increases turbidity by agitating the water column, re-suspending fine particulate matter, and loosening particulate matter in the annular space around the well screen.

NOTE: *It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during the final stages of purging, to minimize turbidity and disturbance of volatile organic constituents.*

2. Initial Inspection

- a) Remove the well cover and remove all standing water around the top of the well casing (manhole) before opening the well. **DO NOT ALLOW WATER FROM SURFACE RUN-OFF TO ENTER THE WELL CASING AT ANY TIME.**
- b) Inspect the exterior protective casing of the monitoring well for damage. Document the results of the inspection if there is a problem.
- c) It is recommended that a protective covering be placed around the well head. Replace the covering if it becomes soiled or ripped.
- d) Inspect the well lock and determine whether the cap fits tightly. Replace the cap if necessary.

3. Water Level Measurements - Use an electronic probe to determine the water level. Decontaminate all equipment before use. Measure the depth to groundwater from the top of the well casing to the nearest 0.01 foot. Always measure from the same reference point or survey mark on the well casing.

4. Water Column Determination - To determine the length of the water column, subtract the depth to the top of the water column from the total well depth (or gauged well depth if silting has occurred). The total well depth depends on the well construction. If gauged well depth is used due to silting, report total well depth also. Some wells may be drilled in areas of sinkhole, karst formations or rock leaving an open borehole. Attempt to find the total borehole depth in cases where there is an open borehole below the cased portion.

5. Well Water Volume - Calculate the total volume of water, in gallons, in the well using the following equation:

$$V = (0.041)d \times d \times h$$

Where: V = volume in gallons
d = well diameter in inches
h = height of the water column in feet

The total volume of water in the well may also be determined with the following equation by using a casing volume per foot factor (Gallons per Foot of Water) for the appropriate diameter well:

$$V = [\text{Gallons per Foot of Water}] \times h$$

Where: V = volume in gallons
h = height of the water column in feet

Casing Internal Diameter	Approximate Gallons per Foot of Water
0.75"	0.02
1"	0.04
1.25"	0.06
2"	0.16
3"	0.37
4"	0.65
5"	1.02
6"	1.47
12"	5.88

Record all measurements and calculations in the field records.

6. Purging Equipment Volume - Calculate the total volume of the pump, associated tubing and flow cell (if used), using the following equation:

$$V = p + ((0.041)d \times d \times l) + fc$$

Where: V = volume in gallons
p = volume of pump in gallons
d = tubing diameter in inches
l = length of tubing in feet
fc = volume of flow cell in gallons

7. If the groundwater elevation data are to be used to construct groundwater elevation contour maps, all water level measurements **must be taken within the same 24-hour time interval** when collecting samples from multiple wells on a site, **unless a shorter time period is required**. If the site is tidally influenced, complete the water level measurements within the time frame of an incoming or outgoing tide.

2.4.6.4 Well Purging Techniques

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. Equipment selection must comply with the construction and configuration requirements specified in Table 14.

1. Measuring the Purge Volume - The volume of water that is removed during purging must be recorded.
 - a) Collect the water in a graduated container and multiply the number of times the container was emptied by the volume of the container, or
 - b) Estimate the volume based on pumping rate. This technique may be used only if the pumping rate is constant. Determine the pumping rate by measuring the amount of water that is pumped for a fixed period of time, or use a flow meter.

- i. Calculate the amount of water that is discharged per minute:

$$D = \frac{\text{Measured amount}}{\text{Total time in minutes}}$$

- ii. Calculate the time needed to purge one (1) well volume or one (1) purging equipment volume:

$$\text{Time} = \frac{V}{D}$$

Where: V = well volume or purging equipment volume
D = discharge rate

- iii. Make new measurements each time the pumping rate is changed.

- c) Use a totalizing flow meter.

- i. Record the reading on the totalizer prior to purging.
- ii. Record the reading on the totalizer at the end of purging.
- iii. To obtain the volume purged, subtract the reading on the totalizer prior to purging from the reading on the totalizer at the end of purging.
- iv. Record the times that purging begins and ends in the field records.

2. Purging Measurement Frequency

- a) When purging a well that has the well screen fully submerged and the pump or intake tubing is placed within the well casing above the well screen or open hole, purge a minimum of one (1) well volume prior to collecting measurements of the field parameters. Allow at least one quarter (1/4) well volume to purge between subsequent measurements.
- b) When purging a well that has the pump or intake tubing placed within a fully submerged well screen or open hole, purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) volume of the pump, associated tubing and flow cell (if used) prior to collecting measurements of the field parameters. Take measurements of the field parameters no sooner than two (2) to three (3) minutes apart. Purge at least three (3) volumes of the pump, associated tubing and flow cell, if used, prior to collecting a sample.
- c) When purging a well that has a partially submerged well screen, purge a minimum of one (1) well volume prior to collecting measurements of the field parameters. Take measurements of the field parameters no sooner than two (2) to three (3) minutes apart.

3. Purging Completion - Wells must be adequately purged prior to sample collection to ensure representation of the aquifer formation water, rather than stagnant well water. This may be achieved by purging three volumes from the well or by satisfying any one of the following three purge completion criteria:

- a) Three (3) consecutive measurements in which the three (3) parameters listed below are within the stated limits, dissolved oxygen is no greater than 20 percent of saturation at the field measured temperature, and turbidity is no greater than 20 Nephelometric Turbidity Units (NTUs).

1. Temperature: $\pm 0.2^{\circ} \text{C}$

2. pH: ± 0.2 Standard Units
3. Specific Conductance: $\pm 5.0\%$ of reading

Document and report the following, as applicable. The last four items only need to be submitted once:

- i. Purging rate.
 - ii. Drawdown in the well, if any.
 - iii. A description of the process and the data used to design the well.
 - iv. The equipment and procedure used to install the well.
 - v. The well development procedure.
 - vi. Pertinent lithologic or hydrogeologic information.
- b) If it is impossible to get dissolved oxygen at or below 20 percent of saturation at the field measured temperature or turbidity at or below 20 NTUs, then three (3) consecutive measurements of temperature, pH, specific conductance and the parameter(s) dissolved oxygen and/or turbidity that do not meet the requirements above must be within the limits below. The measurements are:
- i. Temperature: $+ 0.2^{\circ}$ C
 - ii. pH: $+ 0.2$ Standard Units
 - iii. Specific Conductance: $+ 5.0\%$ of reading
 - iv. Dissolved Oxygen: $+ 0.2$ mg/L or 10%, whichever is greater
 - v. Turbidity: $+ 5$ NTUs or 10%, whichever is greater

Additionally, document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- i. Purging rate.
- ii. Drawdown in the well, if any.
- iii. A description of conditions at the site that may cause the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open hole portion of the well with a downhole dissolved oxygen probe.
- iv. A description of conditions at the site that may cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- v. A description of the process and the data used to design the well.
- vi. The equipment and procedure used to install the well.
- vii. The well development procedure.
- viii. Pertinent lithologic or hydrogeologic information.

If the Department's review of the submitted data determines that both the elevated dissolved oxygen and turbidity measurements are due to naturally occurring conditions, then only the first two (2) items are required to be submitted in future reports. However, if the Department cannot determine if the dissolved oxygen or turbidity are elevated due to naturally occurring conditions, more data are required. In addition to the first two (2) items, a description of the conditions at the site that may have caused the affected parameter(s) to be high will be required in future reports.

- c) If after five (5) well volumes, three (3) consecutive measurements of the field parameters temperature, pH, specific conductance, dissolved oxygen, and turbidity are not within the

limits stated above, check the instrument condition and calibration, purging flow rate and all tubing connections to determine if they might be affecting the ability to achieve stable measurements. It is at the discretion of the consultant/contractor whether or not to collect a sample or to continue purging. Further, the report in which the data are submitted must include the following, as applicable. The last four (4) items only need to be submitted once.

- i. Purging rate.
- ii. Drawdown in the well, if any.
- iii. A description of conditions at the site that may cause the Dissolved Oxygen to be high and/or Dissolved Oxygen measurements made within the screened or open hole portion of the well with a downhole dissolved oxygen probe.
- iv. A description of conditions at the site that may cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- v. A description of the process and the data used to design the well.
- vi. The equipment and procedure used to install the well.
- vii. The well development procedure.
- viii. Pertinent lithologic or hydrogeologic information.

If a review of the data shows that both the elevated dissolved oxygen and turbidity measurements are due to naturally occurring conditions, then only the first two (2) items are required to be submitted in future reports. However, if it cannot be determined that the dissolved oxygen or turbidity are elevated due to naturally occurring conditions, more data is required. In addition to the first two (2) items, a description of the conditions at the site that may have caused the affected parameter(s) to be high will be required in future reports.

- d) One fully dry purge (not recommended). This criterion applies only if purging was attempted, and if it was impossible to balance the pumping rate with the rate of recharge at very low pumping rates (< 100 mL/minute). If wells have previously and consistently purged dry, and the current depth to groundwater indicates that the well will purge dry during the current sampling event, minimize the amount of water removed from the well by using the same pump to purge and collect the sample:
 - i. Place the pump or tubing intake within the well screened interval.
 - ii. Use very small diameter Teflon, polyethylene or polypropylene tubing and the smallest possible pump chamber volume. This will minimize the total volume of water pumped from the well and reduce drawdown.
 - iii. Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.
 - iv. Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.
 - v. Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).
 - vi. Measure pH, specific conductance, temperature, dissolved oxygen and turbidity, then begin to collect the samples.

4. Collect samples immediately after the purging cycle is complete. (The purging cycle is complete when the well is fully recharged.) If sample collection does not occur within one hour of purging completion, re-measure the five field parameters: temperature, pH, specific conductance, dissolved oxygen and turbidity, just prior to collecting the sample. If the measured values are not within 10

percent of the previous measurements, re-purge the well. The exception is wells that are slow to recharge (“dry”).

5. Should a monitoring well fail to adequately recharge after removal of the original standing water volume such that the required criteria cannot be met, then groundwater sample may be collected and submitted for laboratory analyses using the subsequent groundwater recharge within 24 hours or purging.

ALL SAMPLING ACTIVITIES MUST BE COMPLETELY AND ACCURATELY DOCUMENTED IN FIELD NOTES AND SAMPLING FORMS

2.4.6.5 Groundwater Sample Preparation

The type of sample containers used depends on the type of analysis performed. First, determine the type(s) of contaminants expected and the proper analytical method(s) established in Tables 5 or 7. Table 9 lists approved sample containers, preservation and hold times for the specified methods. Be sure to consult the selected laboratory for its specific needs and requirements prior to sampling.

Prepare the storage and transport containers (ice chest, etc.) before taking any samples so that each sample can be placed in a chilled environment immediately after collection.

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. In selecting groundwater purging and sampling equipment, give consideration to the depth of the well, the depth to groundwater, the volume of water to be evacuated, the sampling and purging technique, and the analytes of interest. Refer to Tables 13, 14, 15, 16 and 17. for selection of appropriate equipment. Additional supplies, such as reagents and preservatives, may be necessary.

All sampling equipment (bailers, tubing, containers, etc.) must be selected based on its chemical compatibility with the source being sampled (e.g., water supply well, monitoring well) and the contaminants potentially present.

1. Pumps - All pumps or pump tubing must be lowered and retrieved from the well slowly and carefully to minimize disturbance to the formation water. This is especially critical at the air/water interface.

a) Above-Ground Pumps

1. Variable Speed Peristaltic Pump: Use a variable speed peristaltic pump to purge groundwater from wells when the static water level in the well is no greater than 20-25 feet below land surface. If the water levels are deeper than 18-20 feet below land surface, the pumping velocity will decrease.

- a) A variable speed peristaltic pump can be used for normal purging and sampling, and sampling low permeability aquifers or formations.
- b) Most analyte groups can be sampled with a peristaltic pump if the tubing and pump configurations are appropriate. See Table 14 for proper tubing selection and pump configurations.

2. **Variable Speed Centrifugal Pump:** A variable speed centrifugal pump can be used to purge groundwater from 2-inch and larger internal diameter wells. **Do not use** this type of pump to collect groundwater samples.

- a) When purging is complete, do not allow the water that remains in the tubing to fall back into the well. Install a check valve at the end of the purge tubing.
- b) See Table 14 for proper tubing selection and allowable analyte groups.

b) Submersible Pumps

1. Variable Speed Electric Submersible Pump: A variable speed submersible pump can be used to purge and sample groundwater from 2-inch and larger internal diameter wells.

- a) A variable speed submersible pump can be used for normal purging and
- b) sampling, and sampling low permeability aquifers or formations.
- c) Make sure that the pump housing, fittings, check valves and associated hardware are constructed of stainless steel. Make sure that any other materials are compatible with the analytes of interest. See Table 14 for restrictions.
- d) Install a check valve at the output side of the pump to prevent backflow.
- e) If purging and sampling for organics:
 - i. The entire length of the delivery tube must be Teflon, polyethylene or polypropylene tubing.
 - ii. The electrical cord must be sealed in Teflon, polyethylene or polypropylene and any cabling must be sealed in Teflon, polyethylene or polypropylene, or be constructed of stainless steel.
 - iii. All interior components that contact the sample water (impeller, seals, gaskets, etc.) must be constructed of stainless steel or Teflon.

2. Variable Speed Bladder Pump: A variable speed, positive displacement, bladder pump (no-gas contact) can be used to purge and sample groundwater from 3/4-inch and larger internal diameter wells.

- a) A variable speed bladder pump can be used for normal purging and sampling, and sampling low permeability aquifers or formations.
- b) The bladder pump system is composed of the pump, the compressed air tubing, the water discharge tubing, the controller and a compressor or compressed gas supply.
- c) The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder and two (2) check valves. These parts can be composed of various materials, usually combinations of polyvinyl chloride (PVC), Teflon, polyethylene, polypropylene and stainless steel. Other materials must be compatible with the analytes of interest. See Table 14 for restrictions.
- d) If purging and sampling for organics:
 - i. The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, polyethylene or polypropylene.
 - ii. The entire length of the delivery tube must be Teflon, polyethylene or polypropylene.
 - iii. Any cabling must be sealed in Teflon, polyethylene or polypropylene, or be constructed of stainless steel.
- e) Permanently installed pumps may have a PVC pump body as long as the pump remains in contact with the water in the well.

2. Bailers

- a) Purging: Bailers must be used with caution because improper bailing can cause changes in the chemistry of the water due to aeration and loosening particulate matter in the space around the well screen. Use a bailer if there is non-aqueous phase liquid (free product) in the well or if non-aqueous phase liquid is suspected to be in the well. If a bailer is used, follow the procedures outlined in Section 2.4.6.4.
- b) Sampling: Bailers must be used with caution following the procedures outlined in 2.4.6.3.
- c) Construction and Type:
 1. Bailers must be constructed of materials compatible with the analytes of interest. See Table 14 for restrictions. Stainless steel, Teflon, rigid medical grade PVC, polyethylene and polypropylene bailers may be used to sample all analytes.
 2. Use disposable bailers when sampling grossly contaminated sample sources.
 3. NCDEQ recommends using dual check valve bailers when collecting samples.
 4. Use bailers with a controlled flow bottom to collect volatile organic samples.
- d) Contamination Prevention:
 1. Keep the bailer wrapped (foil, butcher paper, etc.) until just before use.
 2. Use protective gloves to handle the bailer once it is removed from its wrapping.
 3. Handle the bailer by the bailing cord to minimize contact with the bailer surface.

3. Bailing Cord

- a) Bailing cord must be made of non-reactive, non-leachable material. They may be cotton twine, nylon, stainless steel, or may be coated with Teflon, polyethylene or polypropylene.
- b) Discard cotton twine, nylon, and non-stainless steel braided cord after sampling each monitoring well.
- c) Decontaminate stainless steel, coated Teflon, polyethylene and polypropylene bailing cords between monitoring wells. They do not need to be decontaminated between purging and sampling operations.

2.4.6.6 Collecting Samples from Wells Without Plumbing

1. Sampling with Pumps

The following may be used to sample for organics: Variable speed stainless steel and Teflon (with the exception of PFAS contamination) submersible pumps; and stainless steel, Teflon (with the exception of PFAS contamination) or polyethylene bladder pumps; and permanently installed PVC bodied pumps, as long as the pump remains in contact with the water in the well at all times. The delivery tubing must be Teflon (with the exception of PFAS contamination), polyethylene or polypropylene. Extractable organics may be collected through a peristaltic pump if flexible interior-wall Teflon (with the exception of PFAS contamination), polyethylene or polypropylene tubing is used in the pump head. Or if the flexible tubing used in the pump head is other than the types listed, through a peristaltic pump with a vacuum trap. Tubing coming in contact with samples must be one of the types listed. Follow all notes and restrictions as defined in Tables 13 and 14 and discussed in this section when using pumps to collect samples.

2. Tubing/Pump Placement

- a) If attempting to minimize the volume of purge water, the pump will be used for both purging and sampling, the well screen interval is less than or equal to 10 feet, and the well screen is fully submerged, position the intake hose or pump at the midpoint of the screened or open hole interval.
- b) If monitoring well conditions do not allow minimizing of the purge water volume, or if samples will be collected with equipment different than that used to purge, position the

pump or intake hose near the top of the water column. This will ensure that all stagnant water in the casing is removed.

- c) If the well screen or borehole is partially submerged, and the pump will be used for both purging and sampling, position the pump midway between the measured water level and the bottom of the screen. Otherwise, position the pump or intake hose near the top of the water column.

3. Non-dedicated (portable) pumps

a) Variable Speed Peristaltic Pump

- i. Wear sampling gloves to position the decontaminated pump and tubing.
- ii. Attach a short section of tubing to the discharge side of the pump and into a graduated container.
- iii. Attach one end of a length of new or pre-cleaned tubing to the pump head flexible hose.
- iv. Place the tubing as described in one of the options listed above.
- v. Change gloves before beginning to purge.
- vi. Measure the depth to groundwater at frequent intervals.
- vii. Record these measurements.
- viii. Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
- ix. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- x. If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that water is removed from the top of the water column.
- xi. Record the purging rate each time the rate changes.
- xii. Measure the purge volume.
- xiii. Record this measurement.
- xiv. Decontaminate the pump and tubing between wells or if precleaned tubing is used for each well, only the pump.

b) Peristaltic Pump for sampling of Volatile Organics:

- i. Remove the drop tubing from the inlet side of the pump.
- ii. Submerge the drop tubing into the water column.
- iii. Prevent the water in the tubing from flowing back into the well.
- iv. Remove the drop tubing from the well.
- v. Carefully allow the groundwater to gravity drain into the sample vials. Avoid turbulence. Do not aerate the sample.
- vi. Repeat steps ii through v until enough vials are filled.

Or:

- vii. Use the pump to fill the drop tubing.
- viii. Quickly remove the tubing from the pump.
- ix. Prevent the water in the tubing from flowing back into the well.
- x. Remove the drop tubing from the well.
- xi. Carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample.
- xii. Repeat steps vii through xi until enough vials are filled.

Or:

- xiii. Use the pump to fill the drop tubing
- xiv. Withdraw the tubing from the well.
- xv. Reverse the flow on the peristaltic pumps to deliver the sample into the vials at a slow, steady rate.
- xvi. Repeat steps xiii through xv until enough vials are filled.

c) Peristaltic Pump for sampling of Extractable Organics

- 1) If the tubing in the pump head is polyethylene or polypropylene, or is Teflon lined (with the exception of PFAS analyses), the samples may be collected through the pump.
- 2) If the tubing in the pump head is not polyethylene or polypropylene, or is not Teflon lined (with the exception of PFAS analyses), use the pump and vacuum trap method.
 - i. Assemble the components of the pump and trap.
 - ii. The sample container should be the trap.
 - iii. All equipment that contacts the groundwater before the sample container must be constructed of Teflon, polyethylene, polypropylene, stainless steel or glass, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. Do not use a rubber stopper as a cap.
 - iv. Connect the outflow tubing from the container to the influent side of the peristaltic pump.
 - v. Turn the pump on and reduce the flow rate to a smooth and even flow.
 - vi. Discard a small portion of the sample to allow an air space.
 - vii. Preserve (if required), label and complete the field notes.

d) Peristaltic Pump for Sampling of Inorganics

- a) Inorganic samples may be collected from the effluent tubing. There are a few restrictions on tubing type (see Table 14).
- b) If samples are collected from the pump, decontaminate all tubing (including the tubing in the head) or change it between wells.
- c) Preserve (if required), label and complete field notes.

e) Variable Speed Centrifugal Pump

- i. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- ii. Wear sampling gloves to position the decontaminated pump and tubing.
- iii. Place the decontaminated suction hose so that water is always pumped from the top of the water column.
- iv. Change gloves before beginning to purge.
- v. Equip the suction hose with a foot valve to prevent purge water from re-entering the well.
- vi. Measure the depth to groundwater at frequent intervals.
- vii. Record these measurements.

- viii. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
- ix. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- x. If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.
- xi. Record the purging rate each time the rate changes.
- xii. Measure the purge volume.
- xiii. Record this measurement.
- xiv. Decontaminate the pump and tubing between wells or if pre-cleaned tubing is used for each well, only decontaminate the pump.

f) Variable Speed Electric Submersible Pump

- i. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- ii. Wear sampling gloves to position the decontaminated pump and tubing.
- iii. Carefully position the decontaminated pump as described in one of the options in Section D 1 above.
- iv. Change gloves before beginning to purge.
- v. Measure the depth to groundwater at frequent intervals.
- vi. Record these measurements.
- vii. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
- viii. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- ix. If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that water is removed from the top of the water column.
- x. Record the purging rate each time the rate changes.
- xi. Measure the purge volume.
- xii. Record this measurement.
- xiii. Decontaminate the pump and tubing between wells or only the pump if pre-cleaned tubing is used for each well.

g) Variable Speed Submersible Pump for sampling of organics

- i. The housing must be stainless steel or other non-reactive material.
- ii. If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon (with the exception of PFAS analyses), polyethylene or polypropylene. The delivery tubing must be Teflon (with the exception of PFAS analyses), polyethylene or polypropylene; the electrical cord must be sealed in Teflon (with the exception of PFAS analyses; and any cabling must be sealed in Teflon (with the exception of PFAS analyses) or constructed of stainless steel.
- iii. After purging to a smooth even flow, reduce the flow rate.
- iv. When sampling for volatile organic compounds, reduce the flow rate to 100-200 mL/minute, if possible.

h) Variable Speed Bladder Pump

- i. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- ii. Wear sampling gloves to position the decontaminated pump and tubing.
- iii. Attach the tubing and carefully position the pump.
- iv. Change gloves before beginning purging.
- v. Measure the depth to groundwater at frequent intervals.
- vi. Record these measurements.
- vii. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
- viii. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- ix. If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that water is removed from the top of the water column.
- x. Record the purging rate each time the rate changes.
- xi. Measure the purge volume.
- xii. Record this measurement.
- xiii. Decontaminate the pump and tubing between wells or if pre-cleaned tubing is used for each well, decontaminate the pump between wells.

i) Variable Speed Bladder Pump for sampling of organics

- i. If sampling for organics, the pump body must be constructed of stainless steel or non-reactive material and the valves and bladder must be Teflon (with the exception of PFAS analyses). All tubing must be Teflon (with the exception of PFAS analyses), polyethylene, or polypropylene and any cabling must be sealed in Teflon (with the exception of PFAS analyses), polyethylene or polypropylene, or made of stainless steel.
- ii. After purging to a smooth even flow, reduce the flow rate.
- iii. When sampling for volatile organic compounds, reduce the flow rate to 100-200 mL/minute, if possible.

Other types of pumps may be suitable for sampling. If using pumps or pumping devices not listed in this document, a document detailing sampling procedures must be provided for review.

4. Bailers

Using bailers for purging is not recommended unless care is taken to use proper bailing technique, or if free product is present in the well or suspected to be in the well. A high degree of skill and coordination are necessary to collect representative samples with a bailer.

- i. Minimize handling the bailer as much as possible.
- ii. Wear sampling gloves.
- iii. Remove the bailer from its protective wrapping just before use.
- iv. Attach a bailing cord of appropriate material.
- v. Use the bailing cord to move and position the bailer.
- vi. Do not allow the bailer or cord to touch the ground.
- vii. Lower and retrieve the bailer slowly and smoothly.
- viii. Lower the bailer carefully into the well to a depth approximately a foot above the water column.

- ix. When the bailer is in position, lower the bailer into the water column at a rate of 2 cm/sec until the desired depth is reached.
- x. Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column.
- xi. Allow time for the bailer to fill with aquifer water as it descends into the water column.
- xii. Carefully raise the bailer. Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.
- xiii. Measure the purge volume.
- xiv. Record the volume of the bailer.
- xv. Continue to carefully lower and retrieve the bailer as described above until the purging is considered complete, based on either the removal of 3 well volumes or after meeting any of the other purge completion criteria listed above in Section B.
- xvi. Remove at least one (1) well volume before collecting measurements of the field parameters. Take each subsequent set of measurements after removing at least one-quarter (1/4) well volume between measurements.

Rinsing Bailers

- a) If the bailer is certified pre-cleaned, no rinsing is necessary.
- b) If both a pump and a bailer are to be used to collect samples, rinse the exterior and interior of the bailer with sample water from the pump before removing the pump.
- c) If the purge pump is not appropriate for collecting samples (e.g., non-inert components), rinse the bailer by collecting a single bailer of the groundwater to be sampled.
- d) Discard the water appropriately.
- e) Do not rinse the bailer if Oil and Grease samples are to be collected.

2.4.6.7 Collecting Samples from Wells with In-Place Plumbing

In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply. They are generally found at wellfields, industrial facilities and private residences.

1. Air Strippers or Remedial Systems

These types of systems are installed as remediation devices. Collect influent and effluent samples from air stripping units as described below.

- a) Remove any tubing from the sampling port and flush for one to two minutes.
- b) Remove all hoses, aerators and filters (if possible).
- c) Open the spigot and purge sufficient volume to flush the spigot and lines and until the purging completion criteria have been met.
- d) Reduce the flow rate to approximately 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples.
- e) Follow procedures for collecting samples from water supply wells as outlined below.

2. Water Supply Wells

Water supply wells with in-place plumbing do not require equipment to be brought to the well to purge and sample. Water supply wells at UST facilities must be sampled for volatile organic compounds (VOCs) and semivolatile compounds (SVOCs). If a waste oil tank is present, the well should also be sampled for lead and chromium. In accordance with 15A NCAC 2N .0304(b)(5),

water supply wells may also be sampled if there is suspicion of a release from a UST facility. Water supply well samples (potable or non-potable wells) are required to be collected when a well is potentially contaminated, or at risk for becoming contaminated, in response to concerns by well user, and/or to determine the risk ranking of a release. Water supply well samples must be collected directly from a non-aerated spigot (not a garden hose) connected to the water supply well in question. Water from the spigot must be allowed to flow for approximately 15 minutes before sample collection. Samples should be collected in the order of analyses presented above in Section 2.4.1. The following website presents more detailed information regarding water supply well sample collection:

<https://www.epa.gov/quality/potable-water-supply-sampling>, June 11, 2019

Procedures for Sampling Water Supply Wells

1. Label sample containers prior to sample collection.
2. Prepare the storage and transport containers (ice chest, etc.) before taking any samples so that each collected sample can be placed in a chilled environment immediately after collection.
3. Selecting the Sampling Location
 - a) Use the tap closest to the well, preferably at the wellhead. The tap must be before any holding or pressurization tank, water softener, ion exchange, disinfection process or before the water line enters the residence, office or building. If no tap fits the above conditions, a new tap that does must be installed.
 - b) The well pump must not be lubricated with oil, as that may contaminate the samples.
 - c) The sampling tap must be protected from exterior contamination associated with being too close to a sink bottom or to the ground. If the tap is too close to the ground for direct collection into the appropriate container, it is acceptable to use a smaller (clean) container to transfer the sample to a larger container.
 - d) Leaking taps that allow water to discharge from around the valve stem handle and down the outside of the faucet, or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations.
4. Disconnect any hoses, filters, or aerators attached to the tap before sampling.
5. Do not sample from a tap close to a gas pump. The gas fumes could contaminate the sample.

Collecting Volatile Organic Samples from Water Supply Wells

Equipment Needed:

- 1) VOC sample vials [40 milliliters, glass, may contain 3 to 4 drops of hydrochloric acid (HCl) as preservative]
- 2) Disposable gloves and protective goggles
- 3) Ice chest/cooler
- 4) Ice
- 5) Packing materials (sealable plastic bags, bubble wrap, etc.)
- 6) Lab forms

Sampling Procedure:

- 1) Run the water from the well for at least 15 minutes. If the well is deep the water should be run for a longer period of time (purging three well volumes is best). If the tap or spigot is located directly before a holding tank it is a good idea to leave a tap after the holding tank

running to prevent any backflow into the tap where the sample will be collected. This will ensure that the water sample collected is representative of water from the aquifer and not from the holding tank.

- 2) After running the water for at least 15 minutes, reduce the flow of water to make collecting the samples easier and more accurate.
- 3) Remove the cap of a VOC vial and hold the vial under the stream of water to fill it. Be careful not to spill any acid that is in the vial.
- 4) For best results use a low flow of water and angle the vial slightly so that the water runs down the inside of the vial. This will help keep the sample from being agitated, aerated or splashed out of the vial. It will also increase the accuracy of the sample. As the vial fills and is almost full, turn the vial until it is straight up and down so the water won't spill out.
- 5) Fill the vial until the water is just about to spill over the lip of the vial. The surface of the water sample should become mounded. It is a good idea not to overfill the vial, especially if an acid preservative is present in the vial.
- 6) Carefully replace and screw the cap onto the vial. Some water may overflow as the cap is put on.
- 7) After the cap is secure, turn the vial upside down and gently tap the vial to see if any bubbles are present. If bubbles are present in the vial, remove the cap, add more water and check again to see if bubbles are present. Repeat as necessary.
- 8) After the appropriate number of vials without bubbles have been collected, the samples should be labeled and prepared for shipment. The sample will need to be kept at a temperature of 4° C.

Collecting Extractable Organic and/or Metals Samples from Water Supply Wells

Equipment Needed

- 1) SVOC sample bottle [1 liter, amber glass] and/or Metals sample bottle [0.5 liter, polyethylene or glass, 5 milliliters of nitric acid (HNO₃) preservative]
- 2) Disposable gloves and protective goggles
- 3) Ice Chest/Cooler
- 4) Ice
- 5) Packing materials (sealable plastic bags, bubble wrap, etc.)
- 6) Lab forms

Sampling Procedure

- 1) Run the water from the well for at least 15 minutes. If the well is deep the water should be run for a longer period of time (purging three well volumes is best). If the tap or spigot is located directly before a holding tank it is a good idea to leave a tap after the holding tank running to prevent any backflow into the tap where the sample will be collected. This will ensure that the water sample collected is representative of water from the aquifer and not from the holding tank.
- 2) After running the water for at least 15 minutes, reduce the flow of water to make collecting the samples easier and more accurate to reduce turbidity.
- 3) Remove the cap of a SVOC or metals bottle and hold it under the stream of water to fill it.
- 4) The bottle does not have to be completely filled (i.e., an inch or so of headspace in the bottle is permissible unless indicated otherwise by the laboratory).
- 5) After filling, screw-on the cap, label the bottle and prepare for shipment. The sample will need to be kept at a temperature of 4° C.

2.4.6.8 Wells with Floating Non-Aqueous Phase Liquid

Sampling Wells with Floating Non-Aqueous Phase Liquid

NCDEQ does not recommend the sampling of wells with floating non-aqueous phase liquid for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g., solvent) that floats on the water table. Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, these wells may never provide legitimate data as they may have become permanently chemically damaged by the product being in contact with the well casing for an extended period of time. NCDEQ does reserve the right to require sampling of these wells - not for levels of trace contaminants - but for confirmation of an appropriate remediation technique. This type of sampling is performed below the non-aqueous phase layer.

Monitoring Wells with non-aqueous phase liquid

Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.

Non-aqueous phase liquid is normally sampled for two reasons:

- 1) Documentation for its existence and thickness; and
- 2) Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases, as it may not be possible to identify weathered product.
 - i. If a non-aqueous phase liquid is identified in a monitoring well during the water level measurement, measure its thickness in the well. If the thickness of the non-aqueous phase liquid is greater than 0.01 foot, or if product globules are present, collect a sample using a pre-cleaned, disposable bailer.
 - ii. Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.
 - iii. Pour a portion of the product into a glass sample container.
 - iv. This sample is considered a concentrated waste. Therefore, package the container in protective wrapping to prevent breakage, isolate it from other samples, and ice to 4° C.

2.4.6.9 Sampling Low Permeability Aquifers or Wells that have Purged Dry

- 1) Collect the sample(s) after the well has been purged. Minimize the amount of water removed from the well by using the same pump to purge and collect the sample. If the well has purged dry, collect samples as soon as sufficient sample water is available.
- 2) Measure the five field parameters temperature, pH, specific conductance, dissolved oxygen and turbidity at the time of sample collection.
- 3) Advise the analytical laboratory and the client that the usual amount of sample for analysis may not be available.

2.4.7 Equipment Decontamination and Quality Control Samples

General and specific procedures, methods and considerations must be used and observed when cleaning and decontaminating sampling equipment during the course of field investigations. The following website presents a more detailed information regarding equipment decontamination and quality control samples:

<https://www.epa.gov/quality/field-equipment-cleaning-and-decontamination>, June 22, 2020

2.4.8 Product (Tank) Sampling and Drum Sampling (See Waste Sampling)

The US EPA document, **Waste Sampling** (https://www.epa.gov/sites/production/files/2020-11/documents/waste_sampling_lsasdproc-302-r4_051520.pdf) describes general and specific procedures, methods and considerations to be used and observed when collecting waste/container samples for field screening or laboratory analysis. The procedures contained in this document are to be used by field personnel when collecting and handling waste/container samples in the field. On the occasion that field personnel determine that any of the procedures described in this document are inappropriate, inadequate or impractical and that another procedure must be used to obtain a waste sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

2.4.9 Collecting Air Samples from vapor extraction units

The following topics include acceptable equipment selection and equipment construction materials, and standard grab sampling techniques for the collection of air samples from vapor extraction units by the EPA Method 18 Bag Procedure (See Figure 4). Analysis includes benzene, toluene, ethylbenzene, xylene and total petroleum hydrocarbons as isooctane. Other EPA approved comparable methods, which have similar costs, the same constituents, and equivalent or lower detection limits, may be used.

1. General Cautions - When preparing to collect air samples, determine whether the sampling site is in a potentially explosive atmosphere. Follow all guidelines in the health and safety plan for the test. Use appropriate safety equipment as required by conditions at the sampling site.
2. Equipment and Supplies - Use sampling equipment constructed of materials consistent with the analytes of interest. Refer to Tables 13 and 14 for material selection. Select equipment based on the analytes of interest, the specific equipment use and the available equipment. For information on sampling equipment cleaning requirements, see 2.1.4. Sampling events will most frequently employ the Tedlar bag sampling system.
3. Air Sampling Techniques - Adhere to all general protocols applicable to air sampling when following sampling procedures. Alternate sampling procedures may be considered an adequate alternative to the Tedlar bag sample collection, if the recovery study for the alternate sample collection procedure meets the recovery criteria of between 70 and 130 percent recovery for all target compounds listed above.

2.4.10 Vapor Intrusion (VI) Sampling

The Interstate Regulatory Technology Council (IRTC) has developed extensive guidance to assist decision makers in addressing and mitigating risks from vapor intrusion. The **ITRC Petroleum Vapor Intrusion Guidance, Appendix G** provides information on sampling types and processes to determine the amounts and presence of vapor at a site. (<https://www.itrcweb.org/PetroleumVI-Guidance>) Contaminated soil and groundwater can release vapors which have the potential to migrate into buildings. This process is known as vapor intrusion (VI). Petroleum vapor intrusion (PVI) is a subset of VI and describes the process by which volatilized hydrocarbons from petroleum-contaminated soils, groundwater, and light nonaqueous phase liquids (LNAPL) diffuse through the vadose zone and into overlying buildings. Volatile chemicals released from contaminated soil and groundwater can accumulate in soil gas and migrate through unsaturated soils of the vadose zone.

Additional sampling guidance can be found at the following EPA website:

<https://www.epa.gov/quality/quality-system-and-technical-procedures-lsasd-field-branches>

Vapor Sampling Methods

Tedlar bags:

With the flexible bag collection technique, the bags are filled by evacuating the rigid air-tight container holding the bags in a direct interface system. Collect triplicate samples from each sample location.

- a) Assemble the sample train as required in EPA Method 18.
- b) Leak-check both the bag and the container.
- c) Connect the vacuum line from the needle valve to the Teflon (with the exception of PFAS contamination sample line from the probe).
- d) Place the end of the probe in the sample collection port, and start the pump.
- e) Set the flow rate so that the final volume of the sample is approximately 80 percent of the bag capacity.
- f) After allowing sufficient time to purge the line several times, connect the vacuum line to the bag, and evacuate until the rotameter indicates no flow.
- g) Position the sample and vacuum lines for sampling and begin the actual sampling.
- h) At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container.
- i) Record the source temperature, sampling flow rate, and initial and final sampling time.
- j) Protect the Tedlar bags and containers from sunlight.

Other Modified Bag Sampling Procedures:

Sampling with an alternative method may be necessary if condensation is observed in the bag while collecting the sample. See EPA Method 18 for additional details.

- a) Heated sample collection requires the box that contains the sample bag to be heated to 120° C (+ 5° C), followed by transport to the laboratory while maintaining the heating or by insulating the box.
- b) Sample collection in Tedlar bags pre-filled with a known quantity of inert gas.

Adsorption Tube Procedure:

This sampling procedure must be justified and shown to be an adequate alternative to grab sampling with Tedlar bags. Any commercially available adsorbent is allowed for the purposes of EPA Method 18 sample collection as long as the recovery criteria of between 70 and 130 percent recovery for all target compounds are met. Reimbursement by the State Trust Fund will be no greater than the maximum amount allowed for air sampling with the EPA Method 18 bag procedure included in the price list of the Reasonable Rates Document. However, solid adsorbent tubes have some limitations in that most analytical instruments, Gas Chromatographs and Gas Chromatograph/Mass Spectrometers with volatile organic concentrators, have limited dynamic ranges (approximately 1-200 ppb for ambient air). If the sample is not in this range, and the entire sample tube has been used for the first analysis, the option of taking a portion of the sample or diluting the sample is not available. High level samples can cause the systems to become contaminated.

Summa®/Silcosteel® Canister Procedure:

This sampling procedure must be justified and shown to be an adequate alternative to grab sampling with Tedlar bags. Any commercially available pre-cleaned canister is allowed for the purposes of the current version of EPA Method TO-15. Check with the State Trust Fund for allowed reimbursement for air sampling. When collected in canisters, samples can be screened for high levels prior to low level analysis (See Figure 5).

2.5 Sample Storage and Transport

1. Samples for transport must be stored carefully to prevent samples from breaking and to maintain a temperature of approximately 4 degrees Celsius (°C). Samples must be placed on ice immediately and transported to a N.C. DWR-certified laboratory as soon as possible. Unnecessary handling of sample containers should be avoided. Heating (room temperature or above, including exposure to sunlight) or freezing of the sample containers should be avoided. The time between sample collection and delivery to a laboratory should be minimized. The collector must insure that the analytical holding times of samples can be met by the laboratory (See Tables 8 or 9). If samples are field preserved with methanol, no additional preservation measures are necessary other than $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ storage. If samples are not field preserved, additional preservation to inhibit biodegradation besides refrigerated (4°C) storage must be applied the same day samples are received by the laboratory and within 48 hours of sample collection. The maximum hold time may be extended to a total of 14 days from sample collection if the laboratory applies measures to inhibit biodegradation within 48 hours of sample collection. Freezing samples to $-12 \pm 2^{\circ}\text{C}$ in empty VOA vials or specially-designed, approved airtight sampling devices is an acceptable alternative to chemical preservation as a means to inhibit biodegradation.
2. A complete chain-of-custody (COC) form must be maintained to document all transfers and receipts of the samples. Be sure that the sample containers must be labeled with the sample location, site name and/or well number, sample identification, the date and time of collection, the method of analysis to be performed, the preservative added, the sampler's initials and any other pertinent information for sample identification. The labels should contain a unique identifier (i.e., unique well numbers) that can be traced to the COC form. The details of sample collection must be documented on the COC. The COC must include the following:

- a. A description of each sample (including QA/QC samples) and the number of containers (sample location and identification);
 - b. Signature of the sampler;
 - c. The date and time of sample collection;
 - d. The analytical method to be performed;
 - e. The sample type (i.e., water or soil);
 - f. The regulatory agency (i.e., NCDEQ/DWM – UST Section);
 - g. Signatures of all persons relinquishing and receiving custody of the samples; and dates and times of custody transfers.
3. Samples should be packed so that they are segregated by site, sampling location or by sample analysis type. Samples should be segregated in coolers by site as much as possible. If samples from multiple sites fit in one cooler, they may be packed in the same cooler with the associated field sheets and a single COC form for all. For safety, coolers should be packed so that a maximum weight of 50 pounds is not exceeded. Additional coolers should be used as necessary. All sample containers should be placed in plastic bags (segregated by analysis and location) and then completely surrounded by ice. Good packing practices include:
- a. Prepare and place trip blanks in an ice filled cooler before leaving for the field.
 - b. Segregate samples by analysis and place in sealed plastic bags.
 - c. Pack samples carefully in the cooler placing ice around the samples.
 - d. Review the COC. The COC form must accompany the samples to the laboratory.
The trip blank(s) must also be recorded on the COC form.
 - e. Place completed COC form in a waterproof bag, sealed and taped under the lid of the cooler. Secure shipping containers with strapping tape to avoid accidental opening.
 - f. For COC samples, a tamper-proof seal may also be placed over the cooler lid or over a bag or container containing the samples inside the shipping cooler. "COC" or "EMERG" shall be written in indelible ink on the cooler seal to alert sample receipt technicians to priority or special handling samples. The date and sample handler's signature must also be written on the COC seal.
 - g. Deliver the samples to the laboratory or ship by commercial courier.

NOTE: *If transport time to the laboratory is not long enough to allow samples to be cooled to $\leq 6^{\circ}\text{C}$, a temperature reading of the sample source must be documented as the field temperature on the COC form. A downward trend in temperature will be adequate even if cooling to $\leq 6^{\circ}\text{C}$ is not achieved. The field temperature should always be documented if there is any question as to whether samples will have time to cool to $\leq 6^{\circ}\text{C}$ during shipment. Thermometers must be calibrated annually against an NIST traceable thermometer and documentation must be retained.*

2.5.1 Shipping Methanol Preserved Samples

Prepared vials and samples must be shipped according to U.S. Department of Transportation Regulations. Because methanol is a toxic and flammable liquid it must be handled with appropriate care. Use in a well-vented area and avoid inhaling methanol vapors. The use of protective gloves is recommended when handling or transferring methanol. Vials of methanol should always be stored in a cooler with ice away from sources of ignition, such as extreme heat or open flames.

Shipping Hazardous Materials:

Methanol is considered a hazardous material by the US Department of Transportation (DOT) and the International Air Transport Association (IATA). Shipments of methanol between the field and the laboratory must conform to the rules established in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and the most current edition of the IATA Dangerous Goods Regulations. Review these documents or consult with the shipping company for complete details.

Small Quantity Exemption:

The volumes of methanol recommended in the VPH method fall under the small quantity exemption of 49 CFR Section 173.4. To qualify for this exemption, all of the following conditions must be met:

- a) The maximum volume of methanol in each sample container must not exceed 30 ml.
- b) The sample container must not be full of methanol.
- c) The sample container must be securely packed and cushioned in an upright position. It must be surrounded by a sorbent material capable of absorbing spills from leaks or broken sample containers.
- d) The package weight must not exceed 64 pounds.
- e) The volume of methanol per shipping container must not exceed 500 ml.
- f) The packaging and shipping container must be strong enough to hold up to the intended use.
- g) The package must not be opened or altered while in transit.
- h) The shipper must mark the shipping container in accordance with the requirements for shipping dangerous goods in acceptable quantities. They must provide the statement, "This package conforms to conditions and limitations specified in 49 CFR 173.4."

Shipping Papers:

All shipments must be accompanied by shipping papers which include the following:

- a) Proper Shipping Name: Methyl Alcohol
- b) Hazardous Class: Flammable Liquid
- c) Identification Number: UN1230
- d) Total Quantity: (mL methanol/container x the number of containers)
- e) Emergency Response Info: Methanol MSDS attached
- f) Emergency Response Phone: provide appropriate number
- g) Shipping Exemption: DOT-E 173.118, Limited Quantity

Labeling & Placarding:

Labeling and placarding is not required for valid small quantity exemptions (per 49 CFR Section 173.118).

2.6 Laboratory Reports

Results of analyses must be included in the laboratory analytical report. (See Section 2.1.1 for details on Selecting a Laboratory.) All compounds analyzed using a certified method must be reported. All soil sample analytical results must be reported on a dry weight basis. Analytical reporting requirements are also included as an attachment to soil remediation permits. This information should be provided to the laboratory selected to analyze these samples. The laboratory report must include the seven required report elements outlined below.

To ensure reporting requirements are met, the responsible party or the consultant must verify that the person responsible for collection of samples and the N.C. DWR-certified Laboratory selected to perform analyses on samples have complied with the requirements relevant to sampling and analysis.

2.6.1 Required Report Elements

1. NC DWQ-certified Laboratory name, address, certification number, contact and phone number
2. Client/Facility name & address, incident number and name
3. Date of report preparation
4. Chain-of-Custody form including:
 - a) A description of each sample (including QA/QC samples) and the number of containers (sample location, sample preservation and sample identification);
 - b) Signature of the sampler;
 - c) The date and time of sample collection;
 - d) The analytical method to be performed;
 - e) The sample type (i.e., water or soil);
 - f) The regulatory agency (i.e., N.C. DEQ/DWM – UST Section);
 - g) Signatures of all persons relinquishing and receiving custody of the samples, and dates and times of custody transfers.
5. Case Narrative (written on laboratory letterhead or analytical report and signed by the laboratory supervisor or his/her designee): The case narrative should include a detailed description of all problems encountered in the analysis and a discussion of possible reasons for any QA/QC criteria outside acceptance limits.
6. Summary of Analytical Results including:
 - a) Client's sample identification and the corresponding laboratory identification
 - b) Sample matrix,
 - c) Dates of and methods of analysis, preparation and/or extraction,
 - d) Weight or volume of sample used for analysis/extraction/digestion,
 - e) Dilution or concentration factor for the samples,
 - f) Percentage of moisture in the soil samples,
 - g) Definitions of any data qualifiers,
 - h) Method Detection Limit or Limit of Detection,
 - i) Minimum Reporting Limit (established by UST Section for each target analyte),
 - j) Reporting Limit (achieved by a given laboratory for each target analyte)(optional),
 - k) Analytical results with units of measure,
 - l) Signature of Laboratory Supervisor.
7. Summary of QA/QC Results including:
 - a) Laboratory (method, instrument, and storage) blank results and equipment, field, and trip blank results,
 - b) Laboratory QC Check sample results with percent recoveries and control limits,
 - c) Laboratory duplicate results with relative percent difference and control limits,
 - d) Batch Matrix spike/matrix spike duplicate results (where required by method or permit),
 - e) Surrogate recoveries and control limits.

2.6.2 Required Document Retention Criteria

The following items must be retained on file by the laboratory for at least five years after the analysis and must be made available to NCDEQ upon request:

1. The NCDEQ DWR laboratory certification number.
2. Copies of all gas chromatogram traces (with the attached integration report) and of reconstructed ion chromatograms (RICs), if the analysis was performed by mass spectroscopy. (Chromatograms must be provided for all samples, blanks, and daily calibration standards and must be marked with sample identification and the time and date of analysis.)
3. A document reporting the date and time for the initial calibration, the standards used to verify instrument settings for the data, and the composition and concentration range of standards used to establish and verify maintenance of instrument calibration.
4. A document describing laboratory quality control procedures, including information about surrogates, standards, column performance, matrix spike and matrix spike duplicate samples, blank data, and reference samples.
5. A document supporting laboratory reporting limits and method detection limits.

2.6.3 Required Blank Evaluation Criteria

The analysis of blanks and the evaluation of blank results are required in order to reveal the existence and magnitude of contamination resulting from laboratory or field activities. The criteria for the evaluation of blanks apply to any blank associated with the set of samples (e.g., any method, instrument, storage, equipment, field, and trip blank). If any blank in a sample set is discovered to be contaminated, all associated data in the set must be carefully evaluated to determine if the samples also were contaminated or if the contamination in the blank was an isolated occurrence that did not affect the samples.

The action which should be taken when a blank result shows contamination depends on the origin of the contamination, as follows:

Contamination resulting from laboratory activities. For contaminants which are suspected to have originated in the laboratory, the analytical results must be reported and flagged; the case narrative in such instances should include an explanation of possible sources of laboratory contamination.

When laboratory contamination is determined in blanks, quality control samples, or samples in an analytical set, the analytical results must be qualified in the subsequent report.

Every effort must be made by the laboratory to minimize contamination.

Contamination resulting from field activities. It is the responsibility of sample collector to ensure that sampling is performed correctly. Therefore, if a trip blank is determined on laboratory analysis to be contaminated, the sample analytical data should not be corrected by the lab. The analytical results must be flagged, and the possible source of field contamination must be explained by the sample collector in any subsequent report. However, in most instances, the sample collector will be required by the UST Section to resample to obtain contaminant-free samples.

2.6.4 Required Target Analytes for Approved Methods

Methods EPA 8260 and EPA 8270 are approved by the UST Section for petroleum-contaminated soil, and SM 6200, EPA 625, and EPA 602 are approved for petroleum-contaminated water. Except for EPA 602, these methods include extensive lists of compounds, which may be selected as target analytes depending on the use of preparation techniques. Tables with the target analytes required by the UST Section for each of these approved methods are included in the *Tables for STIRA, Sampling Guidelines, Assessment Guidelines and Corrective Action Guidelines* (Tables 1-7). These lists are subsets of the extensive list of compounds amenable to analysis by these methods. Analysis for all of the target analytes in the standardized lists may be required, but the UST Section may approve a less comprehensive list on a site specific basis. Analysis for additional target analytes may be required if site specific conditions indicate that such compounds may be present. To comply with 15A NCAC 2L, the target analytes on the standardized lists must be reported whenever the approved methods are required for samples reported to the UST Section.

Methods other than those discussed above are approved for specific uses, e.g., analysis for non-petroleum (hazardous substance) contaminants in groundwater; target analyte lists for such methods should be requested from the UST Section when required.

2.6.5 Required Reporting Limits

A detection limit is indicated for each of the required target analytes in the method description. However, not all of the NC-certified laboratories are able to achieve this detection limit. Therefore, a minimum Reporting Limit (RL), which is equal to or greater than the detection limit indicated in method, is set by the UST Section for each of the target analytes. The RL is listed for each of the required volatile and semi-volatile target analytes, along with the Groundwater Quality Standard and most restrictive Maximum Soil Contaminant Concentration for each analyte, in Tables 1-7.

The Method Detection Limit (MDL) for each required target analyte must be calculated annually, at a minimum. The MDLs for all required target analytes should be retained by the laboratory.

3.0 References

- Alaska Department of Environmental Conservation. "Underground Storage Tanks Procedures Manual." March 22, 2017. Available at <https://dec.alaska.gov/spar/csp/tanks>.
- Arizona Department of Health Services. *Information Update #52*. "EPA 5035 Implementation". November 1998. Available on the Internet at <https://www.azdeq.gov/USTProgram>
- Bartha, R. and Bossert, I. 1981. "The Treatment and Disposal of Petroleum Wastes," *Petroleum Microbiology*. McMillan Publishing Co., NY.
- Federal Register 40 CFR Part 60 Appendix A "EPA Method 18 –Measurement of Gaseous Organic Compound Emissions by Gas Chromatography," Promulgated February 2000. Available at <https://www.epa.gov/emc/method-18-volatile-organic-compounds-gas-chromatography>
- Florida Department of Environmental Protection, "DEP Standard Operating Procedures for Field Activities," DEP SOP-001/01, January 2001. Available at <https://floridadep.gov/dear/quality-assurance/content/dep-sops>
- International Air Transport Association. "Dangerous Goods Regulations." Issued annually. Available at <https://www.iata.org/en/publications/dgr/>
- Massachusetts Department of Environmental Protection. "Method for the Determination of Extractable Petroleum Hydrocarbons." May 2004. Available on the Internet at <https://www.mass.gov/lists/policies-guidance-technical-support-for-site-cleanup>
- Massachusetts Department of Environmental Protection. "Preservation Techniques for Volatile Organic Compound (VOC) Soil Sample Analyses." Available at <https://www.mass.gov/lists/policies-guidance-technical-support-for-site-cleanup> and <https://www.mass.gov/doc/wsc-99-415-preservation-techniques-for-volatile-organic-compound-voc-soil-sample-analyses-0/download>
- State of California Leaking Underground Fuel Tank Task Force "Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure." October 1989. https://cfpub.epa.gov/ols/catalog/advanced_full_record.cfm?&FIELD1=SUBJECT&INPUT1=gasoline&TYPE1=EXACT&LOGIC1=AND&COLL=&SORT_TYPE=MTIC&item_count=629&item_accn=371458
- U.S. EPA "Current Perspectives in Site Remediation and Monitoring: Applying the Concept of Effective Data to Environmental Analysis for Contaminated Sites." October 2001. EPA542/R/01/013. <https://www.epa.gov/nscep>
- U.S. EPA. "Data Collection and Evaluation," Region IV Office of Technical Services, Human Health Risk Assessment Bulletins – Supplement to RAGS, 2000. Available on Internet at <http://www.epa.gov/region04/waste/ots/healthbul.htm>.
- U.S. EPA. "Data Quality Objectives Process for Superfund - Interim Final Guidance," OSWER, EPA 540-R-93-071, PB94-963203. 1993. <https://www.epa.gov/nscep>
- U.S. EPA. "Data Quality Objectives for Remedial Response Activities," EPA 540/G-87-003 OSWER 9355.07. 1987. <https://www.epa.gov/nscep>
- U.S. EPA. "Determination of Background Levels of Inorganics in soil and Sediments at Hazardous Waste Sites," Engineering Forum Issue Paper. 1995. EPA/540/S-96/500. <https://www.epa.gov/nscep>
- U.S. EPA. "Field Equipment Cleaning and Decontamination" <https://www.epa.gov/quality/field-equipment-cleaning-and-decontamination>
- U.S. EPA. Guidance for the Data Quality Objectives Process EPA QA/G-4, EPA/600/R-96/055, 1994. Office of Research and Development, available at <https://www.epa.gov/nscep>

U.S. EPA. "Groundwater sampling." https://www.epa.gov/sites/production/files/2017-07/documents/groundwater_sampling301_af.r4.pdf

U.S. EPA. "Interim Draft Statistical Tests for Background Comparison at Hazardous Waste Sites." 1998.

U.S. EPA. "National Functional Guidelines for Inorganic Superfund Methods Data Review." OLEM 9355.0-135, January 2017. EPA-540-R-2017-001. <https://www.epa.gov/clp/national-functional-guidelines-inorganic-superfund-methods-data-review-ism024>

U.S. EPA. "National Functional Guidelines for Organic Superfund Methods Data Review." OLEM 9355.0-136, January 2017. EPA-540-R-2017-002. <https://www.epa.gov/clp/national-functional-guidelines-organic-superfund-methods-data-review-som024>

U.S. EPA. Office of Solid Waste. Memorandum. "Clarification Regarding Use of SW-846 Methods." August 1998. <http://www.epa.gov/sw-846/news.htm>.

U.S. EPA. "Potable Water Supply Sampling." <https://www.epa.gov/quality/potable-water-supply-sampling>

U.S. EPA. "Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies." Preissue copy. July 1992. EPA/600/R-92/128. <https://www.epa.gov/nscep>

U.S. EPA Region 3, Risk Assessment: Technical Guidance Manual, "EPA Region III Guidance on Handling Chemical Concentration Data Near the Detection Limit in Risk Assessment," November 4, 1991. <http://www.epa.gov/reg3hwmd/risk/guide3.htm>.

U.S. EPA. Region 4, "Environmental Investigations Standard Operating Procedures and Quality Assurance Manual," November, 2001. <http://www.epa.gov/region4/sesd/eisopqam/eisopqam.html>.

U.S. EPA. Region 4, "Operating Procedure: Groundwater Sampling," SESDPROC-301-R4, April 26, 2017. https://www.epa.gov/sites/production/files/2017-07/documents/groundwater_sampling301_af.r4.pdf

U.S. EPA. "Risk Assessment Guidance for Superfund," Volume I, Human Health Evaluation Manual (Part A) Interim Final (RAGS). 1989. EPA/540/1-89/002, p. 4-8 <https://www.epa.gov/nscep>

U.S. EPA. "Soil Sampling Quality Assurance User's Guide." Second Edition, March 1989. EPA/6008-89/046. <https://www.epa.gov/nscep>

U.S. EPA. "Surface Water Sampling." February 28, 2013. <https://www.epa.gov/quality/surface-water-sampling>

U.S. EPA. "Soil Sampling." Effective date June 11, 2020 and Review date June 11, 2024. <https://www.epa.gov/quality/soil-sampling>

U.S. EPA. "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods," U.S. EPA publication number SW-846. Third Edition, June 1997. <https://www.epa.gov/nscep>

U.S. EPA. "Waste Sampling." https://www.epa.gov/sites/production/files/2020-11/documents/waste_sampling_lsasdproc-302-r4_051520.pdf

U.S. Geological Survey, "National Field Manual for the Collection of Water - Quality Data; Techniques of Water-Resources Investigations," Book 9, Chapters A-1 - A-9. 1997.

Wisconsin Department of Natural Resources. "Analytical Detection Limit Guidance and Laboratory Guide for Calculating Analytical Detection Limits." April 1996. [WDNR PUBL-TS-056; PDF, 337K] Available at <https://dnr.wi.gov/regulations/labcert/documents/guidance/-LODguide.pdf>

4.0 Figures

Figure 1	Regional Office Locations and Map	15
Figure 2	Volumes of Stockpiles	16
Figure 3	Soil Stockpile Sampling Map (Example)	17
Figure 4	EPA Method 18 Integrated Bag Sampling Train	18
Figure 5	EPA Method 15 Summa Canister	19

DRAFT

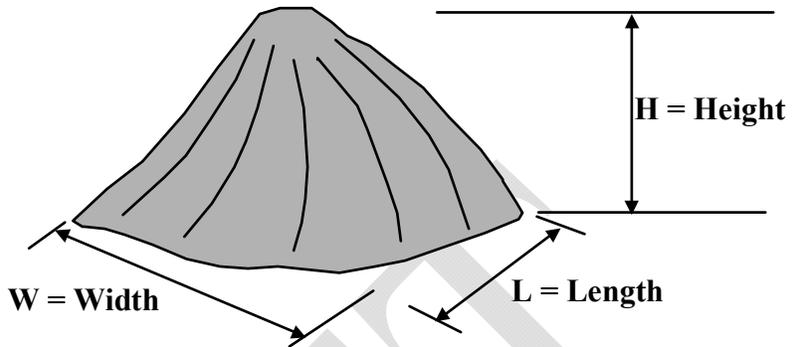
Figure 1 - Regional Office Locations and Map

<https://files.nc.gov/ncdeq/Waste%20Management/DWM/UST/Corrective%20Action/Regional%20Office%20Contact%20Map.pdf>

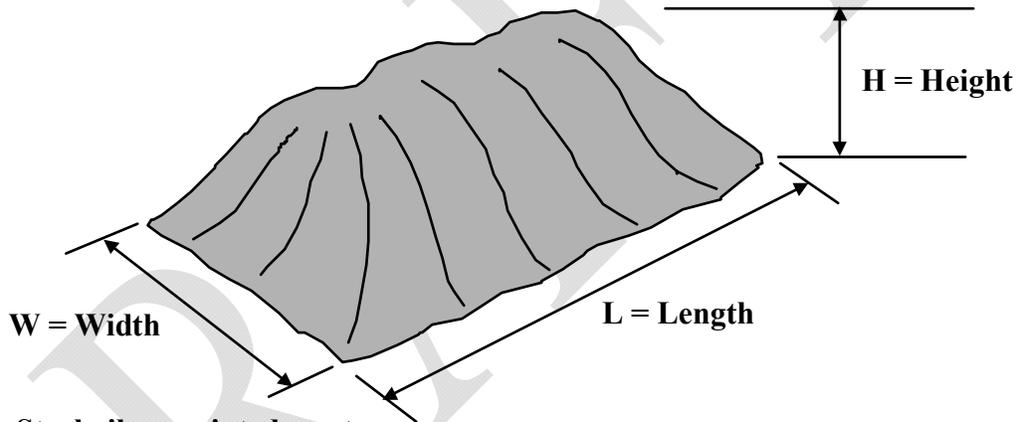
DRAFT

Figure 2 - Volumes of Stockpiles

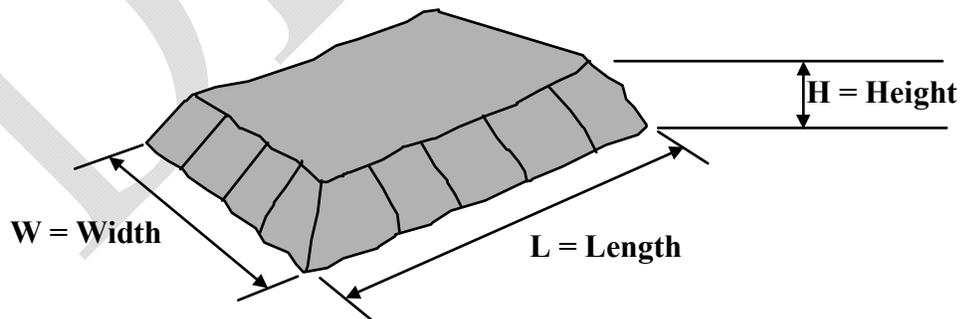
$L > W > H$



Conical Stockpiles:
Volume = $(H \times L \times W)/3$



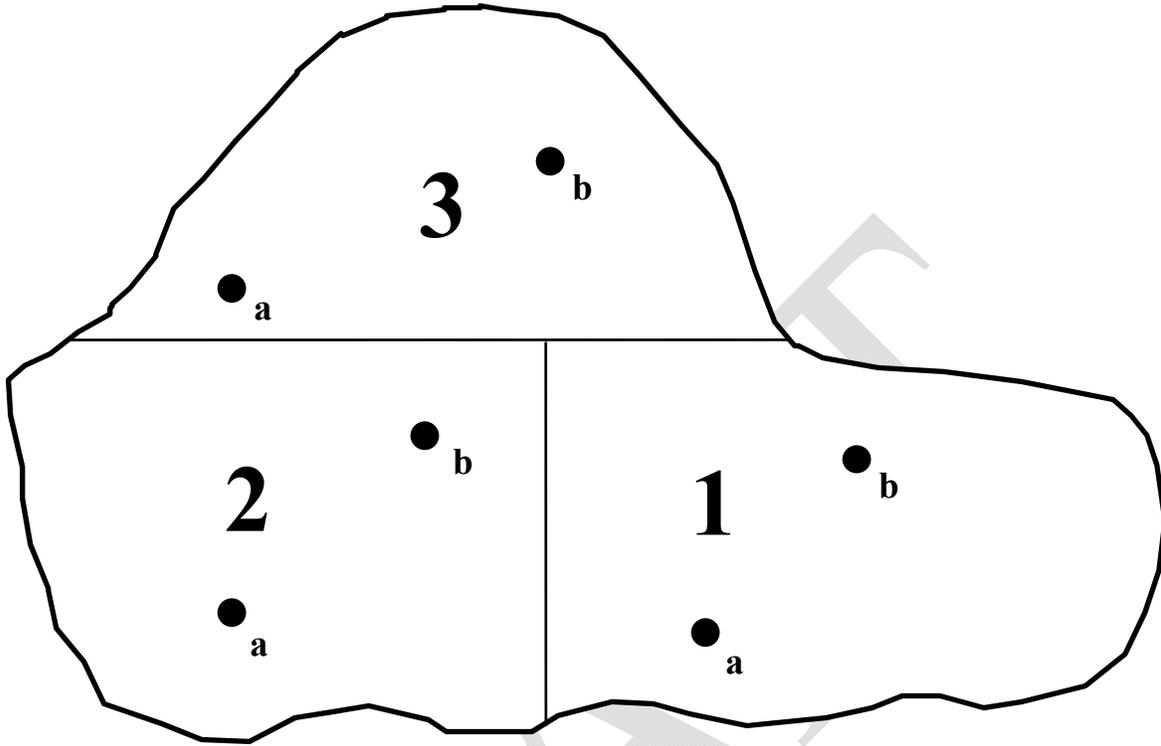
Rectangular Stockpiles - pointed crest:
Volume = $(H \times L \times W)/2$



Rectangular Stockpiles - flat topped:
Volume = $H \times [(L \times W) - (2 \times H \times W)]$

Note: These equations have been simplified for ease of calculation.

Figure 3 - Soil Stockpile Sampling Map (Example)



Stockpile Type: Rectangle, Flat-topped

Volume = 4 x [(21 x 11) - (2 x 4 x 11)] = 572 cubic yards

where: Height (maximum) = 12 feet = 4 yards
 Length (maximum) = 63 feet = 21 yards
 Width (maximum) = 33 feet = 11 yards

Composite Samples	3 Primary Samples per core	Sample Depths
1	core 1a	1', 4', 7'
	core 1b	1', 5', 8'
2	core 2a	2', 6', 10'
	core 2b	1', 4', 7'
3	core 3a	1', 3', 5'
	core 3b	2', 5', 8'

NOTE: Each composite sample contains six primary samples, three from core "a" and three from core "b."

Figure 4 - EPA Method 18 Integrated Bag Sampling Train

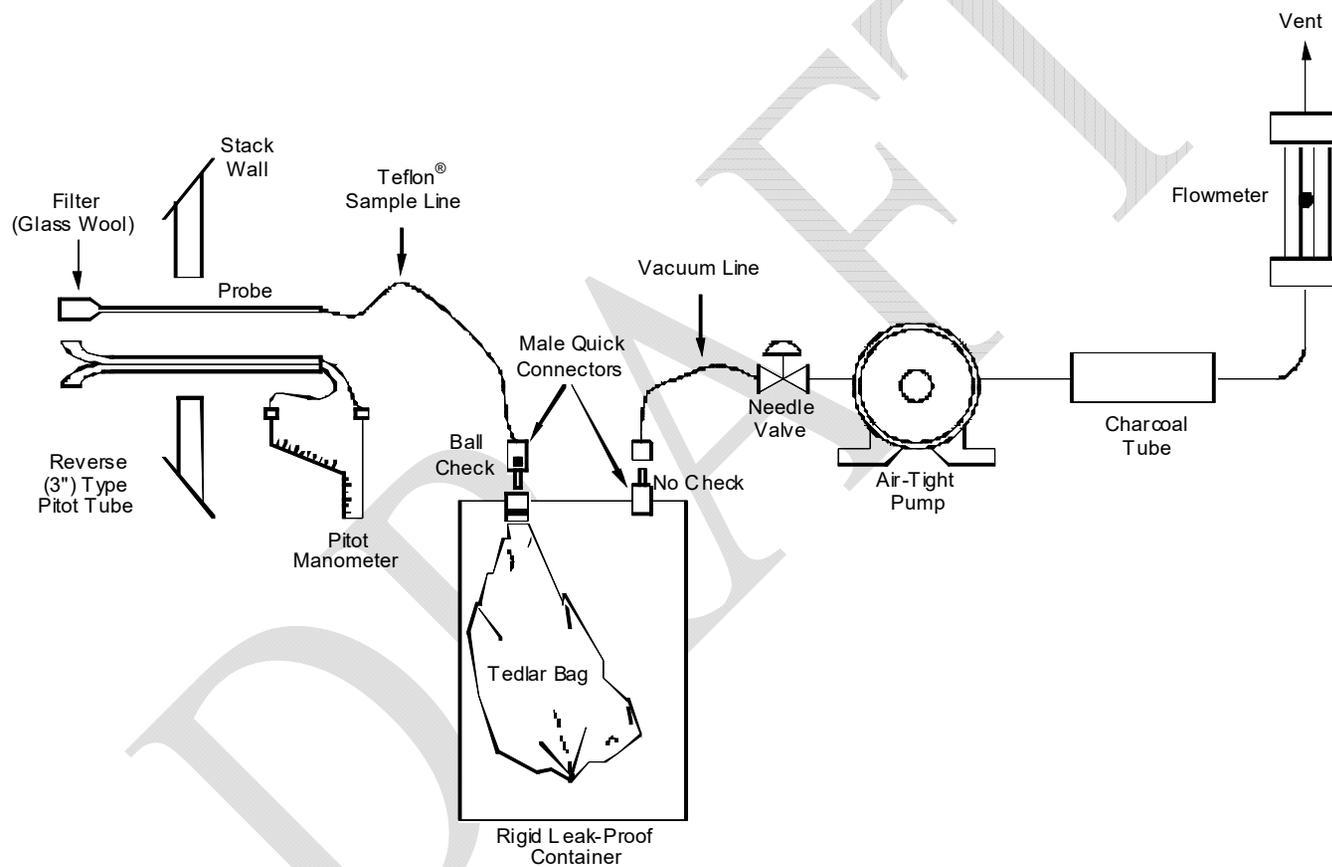


Figure 5 - EPA Method 15 Summa Canister

https://www.epa.gov/sites/production/files/2019-12/documents/to-15a_vocs.pdf

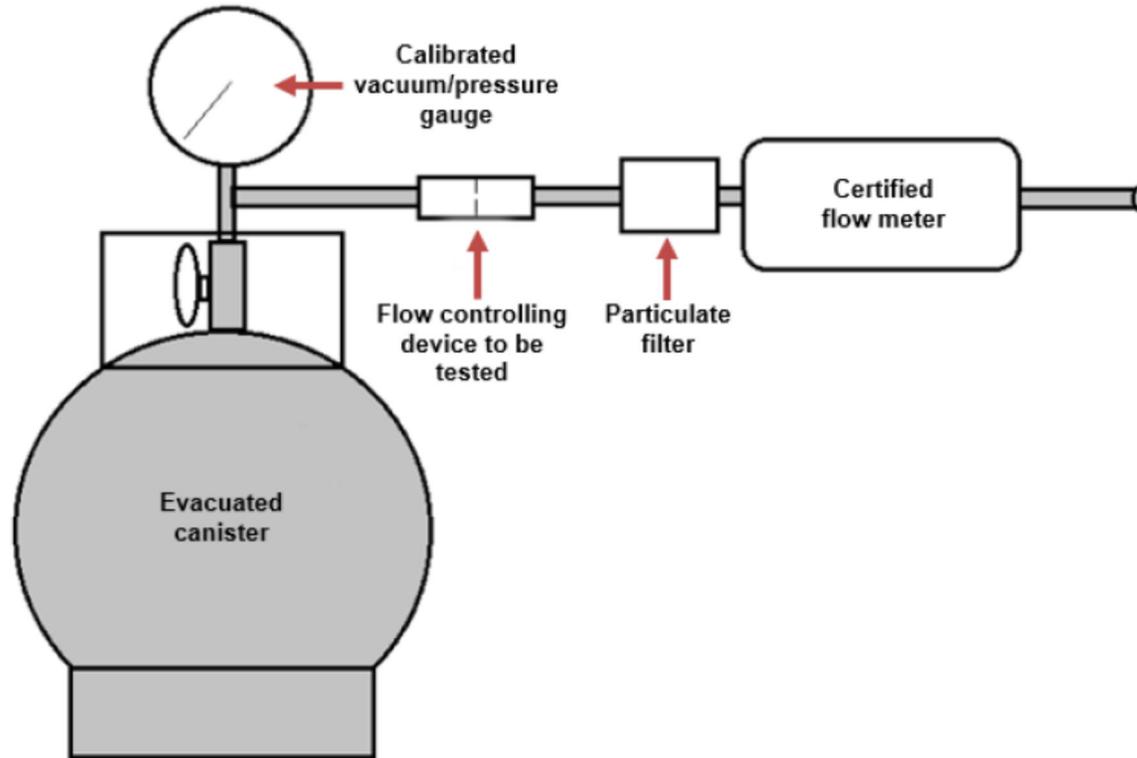


Figure 9-1: Flow controlling device characterization apparatus.