

List of Appendices

Appendix A - Selecting a Laboratory	A - 1
Appendix B - Analytical Reporting Requirements	B - 1
Appendix C - Decontamination of Sampling Equipment	C - 1
Appendix D - Calibration and Maintenance of Field Equipment	D - 1
Appendix E - Collecting Soil Samples	E - 1
Appendix F - Collecting Groundwater Samples	F - 1
Appendix G - Collecting Samples from Wells with Plumbing in Place	G - 1
Appendix H - Collecting Surface Water Samples	H - 1
Appendix I – Collecting Air Samples	I - 1

Appendix A Selecting a Laboratory

The NCDENR DWQ 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 NCDENR DWQ Chemistry Laboratory at 4405 Reedy Creek Road, Raleigh, NC 27607 or the DWQ Laboratory Certification Program web page at <http://portal.ncdenr.org/web/wq/lab/cert> or by calling (919) 733-3908. Required analytical reporting elements are outlined in Appendix B of this document and in Attachment A in permits issued by the NCDENR UST Section. 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:

1. 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 NCDENR's DWM UST Section.
2. Think in terms of quality, not just dollars: A laboratory that produces data that is not acceptable to the regulatory agency usually means that the laboratory will need to repeat the work. It is more cost effective to select a laboratory that will meet the quality needs of the project even if that laboratory is not the low bidder.
3. Continue evaluation: In order to ensure that the laboratory provides data of a consistent quality, do not rely on just the initial evaluation of a laboratory. Other quality control measures will provide the ability to continuously evaluate the laboratory data quality.
4. Evaluate the reported data: Review the final laboratory reports against the original expectations and acceptable quality control measures.
5. Ask questions: The consumer has the right to question laboratory results and receive a logical, clear response. An informed client increases the probability of quality data and data acceptability.

A. Identifying Laboratory Needs

The consumer should be able to identify these critical needs before considering any laboratory:

1. The purpose for which the data are needed.
 - a) The consumer must determine expectations 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 in your company 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. DENR DWQ 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 you sign 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

Appendix A
Selecting A Laboratory

- 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 (See Appendix B for routine lower reporting concentrations). 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. DENR DWM UST Section and DWQ 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 Appendix B.
 - A. Copies of all raw data and associated records, or
 - B. 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. Multiple dilutions will not be reimbursed as a separate analysis by the State Trust Fund.*

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 are 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.

Appendix B Analytical Reporting Requirements

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. DWQ-certified Laboratory selected to perform analyses on samples have complied with the requirements relevant to sampling and analysis, respectively, in this appendix.

A. 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. DENR/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,.

B. 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 NCDENR upon request:

1. The NCDENR DWQ 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.

C. 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.

D. Required Target Analytes for Approved Methods

Methods EPA 8260B and EPA 8270D are approved by the UST Section for petroleum-contaminated soil, and SM 6200B, 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 this appendix (App. B. Tables 1-6). 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.

E. 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 (MRL), 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 MRL 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 1-6 in this appendix.

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.

F. Required EPH and VPH reporting forms

These forms are provided with instructions for completion.

**Method EPA 8260B NC UST Section Required Target Analytes for Petroleum
Contaminated Soil**

8260B Target Analytes	CASRN	Soil-to-Groundwater MSCC mg/kg	EPA 8260B Minimum Reporting Limit, mg/kg
Acetone	67-64-1	24	0.05
tert-Amyl alcohol (TAA)	75-85-4	0.1	0.4
tert-Amyl methyl ether (TAME)	994-05-8	0.52	0.1
Benzene	71-43-2	0.0056	0.005
Bromobenzene	108-86-1		0.005
Bromochloromethane	74-97-5		0.005
Bromodichloromethane	75-27-4		0.005
Bromoform	75-25-2	0.026	0.005
Bromomethane (methylbromide)	74-83-9	0.4	0.005
2-Butanone (MEK)	78-93-3	16	0.05
tert-Butyl alcohol (TBA)	75-65-0	0.04	0.2
n-Butylbenzene	104-51-8	4.3	0.005
sec-Butylbenzene	135-98-8	3.3	0.005
tert-Butylbenzene	98-06-6	3.4	0.005
tert-Butyl formate (TBF)	762-75-4	0.1	0.4
Carbon tetrachloride	56-23-5		0.005
Chlorobenzene	108-90-7	0.44	0.005
Chlorodibromomethane	124-48-1	0.0021	0.005
Chloroethane	75-00-3		0.005
Chloroform	67-66-3	0.37	0.005
Chloromethane	74-87-3	0.02	0.005
2-Chlorotoluene	95-49-8		0.005
4-Chlorotoluene	106-43-4	0.1	0.005
1,2- Dibromoethane (EDB)	106-93-4	0.000098	0.005
1,2-Dichlorobenzene	95-50-1	0.23	0.005
1,3-Dichlorobenzene	541-73-1	7.6	0.005
1,4-Dichlorobenzene	106-46-7	0.099	0.005
Dichlorodifluoromethane	75-71-8	210	0.005
1,1-Dichloroethane	75-35-3	0.032	0.005
1,2-Dichloroethane (1,2-DCA)	107-06-2	0.0019	0.005
1,1-Dichloroethene	75-35-4	0.045	0.005
cis-1,2-Dichloroethene	156-59-2	0.35	0.005
trans-1,2-Dichloroethene	156-60-5	0.54	0.005
1,2-Dichloropropane	78-87-5	0.0030	0.005
1,3-Dichloropropane	142-28-9		0.005

Appendix B
Analytical Reporting Requirements

8260B Target Analytes	CASRN	Soil-to-Groundwater MSCC mg/kg	EPA 8260B Minimum Reporting Limit, mg/kg
2,2-Dichloropropane	590-20-7		0.005
1,1-Dichloropropene	563-58-6		0.005
cis-1,3-Dichloropropene	10061-01-5	0.001 (cis & trans)	0.005
trans-1,3-Dichloropropene	10061-02-6	0.001 (cis & trans)	0.005
Ethanol	64-17-5	16	0.25
Ethylbenzene	100-41-4	4.9	0.005
Ethyl tert-butyl ether (ETBE)	63-79-23	0.2	0.1
2-Hexanone	591-78-6	0.1	0.010
Isopropyl benzene	98-82-8	1.7	0.005
Isopropyl ether	108-20-3	0.37	0.005
4-Isopropyl toluene	99-87-6	0.12	0.005
Methylene chloride	75-09-2	0.02	0.005
Methyl isobutyl ketone (MIBK)	108-10-1	0.4	0.05
Methyl-tert-butyl ether (MTBE)	1634-04-4	0.091	0.005
Naphthalene	91-20-3	0.16	0.005
n-Propylbenzene	103-65-1	1.7	0.005
Styrene	100-42-5	1.5	0.005
1,1,1,2-Tetrachloroethane	630-20-6	0.004	0.005
1,1,2,2-Tetrachloroethane	79-34-5	0.001	0.005
Tetrachloroethene (PCE)	127-18-4	0.0074	0.005
Toluene	108-88-3	4.3	0.005
1,2,3-Trichlorobenzene	87-61-6		0.005
1,2,4-Trichlorobenzene	120-82-1	2.6	0.005
1,1,1-Trichloroethane	71-55-6	1.6	0.005
1,1,2-Trichloroethane	79-00-5	0.002	0.005
Trichloroethene (TCE)	79-01-6	0.019	0.005
Trichlorofluoromethane	75-69-4	29	0.005
1,2,3-Trichloropropane	96-18-4		0.005
1,2,4-Trimethylbenzene	95-63-6	8.5	0.005
1,3,5-Trimethylbenzene	108-67-8	8.3	0.005
Vinyl acetate	108-05-4	0.36	0.010
Vinyl chloride	75-01-4	0.00018	0.005
o-Xylene	95-47-6		0.005
m-Xylene	108-38-3		0.005
p-Xylene	106-42-3		0.005
(Xylenes, Total)	1330-20-7	4.6	0.015

¹ Please note that the Minimum Reporting Limits (MRLs) listed in this table should be routinely achievable but are not corrected for dilution factors (i.e. due to high levels of contamination and/or soil moisture). Dilution factors must be applied, which will result in the elevation of these routinely achievable lower reporting concentrations.

Appendix B Analytical Reporting Requirements

Soil moisture content, which is sample specific, will result in sample-specific adjustments in method detection limit and reporting limit concentrations.

- ² The MRL is set at the level of 0.005 mg/kg for most of the target analytes listed; however, the MRLs for some target analytes is set at levels higher than 0.005 mg/kg due to the lower purging efficiency of some instruments.
- ³ If no value is entered in the MSCC column, no standard limit has been established. Detection is a violation.
- ⁴ Shading indicates that the MRL for an analyte is greater than its standard limit. If decisions hinge on the concentration of this analyte, then it may be necessary to repeat the analysis in order to reduce the MRL.

**Method EPA 8270D NC UST Section Required Target Analytes for Petroleum
Contaminated Soil**

EPA 8270D Target Analytes	CASRN	Soil-to-Groundwater MSCC (Residential MSCC) mg/kg	EPA 8270D Minimum Reporting Limit, mg/kg
Acenaphthene	83-32-9	8.2	0.167
Acenaphthylene	208-96-8	11	0.167
Anthracene	120-12-7	940	0.167
Benzoic Acid	65-85-0	120	1.67
Benz(a)anthracene	56-55-3	0.35	0.167
Benzo(b)fluoranthene	205-99-2	1.2 (0.88)	0.167
Benzo(k)fluoranthene	207-08-9	12 (9)	0.167
Benzo(g,h,i)perylene	191-24-2	6400 (469)	0.167
Benzo(a)pyrene	50-32-8	0.096 (0.088)	0.167
Benzyl alcohol	100-51-6	2	0.333
Bis(2-chloroethoxy)methane	111-91-1		0.167
Bis(2-chloroethyl)ether	111-44-4	0.00016	0.167
Bis(2-chloroisopropyl)ether	108-60-1		0.167
Bis(2-ethylhexyl)phthalate	117-81-7	6.6	0.167
4-Bromophenyl phenyl ether	101-55-3		0.167
Butyl benzyl phthalate	85-68-7		0.167
4-Chloroaniline	106-47-8		0.167
4-Chloro-3-methylphenol	59-50-7		0.333
2-Chloronaphthalene	91-58-7		0.167
2-Chlorophenol	95-57-8		0.167
4-Chlorophenyl phenyl ether	7005-72-3		0.167
Chrysene	218-01-9	39	0.167
Dibenz(a,h)anthracene	53-70-3	0.17 (0.088)	0.167
Dibenzofuran	132-64-9	4.7	0.167
Di-n-butyl phthalate	84-74-2		0.167
1,2-Dichlorobenzene	95-50-1	0.23	0.167
1,3-Dichlorobenzene	541-73-1	7.6	0.167
1,4-Dichlorobenzene	106-46-7	0.099	0.167
3,3'-Dichlorobenzidine	91-94-1		1.67
2,4-Dichlorophenol	120-83-2	0.0034	0.167
Diethyl phthalate	84-66-2		0.167
2,4-Dimethylphenol (2,4-xyleneol)	105-67-9	0.64	0.167
Dimethyl phthalate	131-11-3		0.167
4,6-Dinitro-2-methylphenol	534-52-1		0.667
2,4-Dinitrophenol	51-28-5		1.67
Di-n-octyl phthalate	117-84-0		0.167

Appendix B
Analytical Reporting Requirements

EPA 8270D Target Analytes	CASRN	Soil-to-Groundwater MSCC (Residential MSCC) mg/kg	EPA 8270D Minimum Reporting Limit, mg/kg
1,2-Diphenylhydrazine (as Azobenzene)	122-66-7		0.333
Fluoranthene	206-44-0	290	0.167
Fluorene	86-73-7	47	0.167
Hexachlorobenzene	118-74-1		0.167
Hexachlorobutadiene	87-68-3	0.23	0.167
Hexachlorocyclopentadiene	77-47-4		0.167
Hexachloroethane	67-72-1		0.167
Indeno(1,2,3-c,d)pyrene	193-39-5	3.4 (0.88)	0.167
Isophorone	78-59-1		0.167
1-Methylnaphthalene	90-12-0	0.004	0.167
2-Methylnaphthalene	91-57-6	3.6	0.167
2-Methylphenol	95-48-7		0.167
4-Methylphenol	106-44-5		0.167
Naphthalene	91-20-3	0.16	0.167
Nitrobenzene	98-95-3		0.167
2-Nitrophenol	88-75-5		0.167
4-Nitrophenol	100-02-7		0.667
N-Nitrosodiphenylamine	86-30-6		0.167
N-Nitroso-di-n-propylamine	621-64-7		0.167
Pentachlorophenol	87-86-5	0.0065	0.667
Phenanthrene	85-01-8	56	0.167
Phenol	108-95-2	0.17	0.167
Pyrene	129-00-0	270	0.167
1,2,4-Trichlorobenzene	120-82-1	2.6	0.167
2,4,6-Trichlorophenol	88-06-2	0.01	0.167

¹ Please note that the Minimum Reporting Limits (MRLs) listed in this table should be routinely achievable but are not corrected for dilution factors (i.e. due to high levels of contamination and/or soil moisture). Dilution factors must be applied, which will result in the elevation of these routinely achievable lower reporting concentrations. Soil moisture content, which is sample specific, will result in sample-specific adjustments in method detection limit and reporting limit concentrations.

² The MRL is set at the level of 0.167 mg/kg for most of the target analytes listed; however, the MRLs for some target analytes are set at levels higher than 0.167 mg/kg due to the lower response of some instruments.

³ If no value is entered in the MSCC column, no standard limit has been established. Detection is a violation.

⁴ Shading indicates that the MRL for an analyte is greater than its standard limit. If decisions hinge on the concentration of this analyte, then it may be necessary to repeat the analysis in order to reduce the MRL.

**Method SM 6200B NC UST Section Required Target Analytes for Petroleum
Contaminated Water**

SM 6200B Target Analytes	CASRN	2L Groundwater Standard	SM 6200B Minimum Reporting Limit, µg/L
		µg/L	µg/L
Acetone	67-64-1	6000	5
tert-Amyl alcohol (TAA)	75-85-4	40	40
tert-Amyl methyl ether (TAME)	994-05-8	128	10
Benzene	71-43-2	1	0.5
Bromobenzene	108-86-1		0.5
Bromochloromethane	74-97-5		0.5
Bromodichloromethane	75-27-4	0.6	0.5
Bromoform	75-25-2	4	0.5
Bromomethane (methylbromide)	74-83-9	100	0.5
2-Butanone (MEK)	78-93-3	4000	5
tert-Butyl alcohol (TBA)	75-65-0	10	20
tert-Butyl formate (TBF)	762-75-4	40	40
n-Butylbenzene	104-51-8	70	0.5
sec-Butylbenzene	135-98-8	70	0.5
tert-Butylbenzene	98-06-6	70	0.5
Carbon tetrachloride	56-23-5	0.3	0.5
Chlorobenzene	108-90-7	50	0.5
Chlorodibromomethane	124-48-1	0.4	0.5
Chloroethane	75-00-3	3000	0.5
Chloroform	67-66-3	70	0.5
Chloromethane	74-87-3	3	0.5
2-Chlorotoluene	95-49-8	100	0.5
4-Chlorotoluene	106-43-4	24	0.5
1,2- Dibromoethane (EDB)	106-93-4	0.02	0.5
1,2-Dichlorobenzene	95-50-1	20	0.5
1,3-Dichlorobenzene	541-73-1	200	0.5
1,4-Dichlorobenzene	106-46-7	6	0.5
Dichlorodifluoromethane	75-71-8	1000	0.5
1,1-Dichloroethane	75-35-3	6	0.5
1,2-Dichloroethane	107-06-2	0.4	0.5
1,1-Dichloroethene	75-35-4	7	0.5
cis-1,2-Dichloroethene	156-59-2	70	0.5
trans-1,2-Dichloroethene	156-60-5	100	0.5
1,2-Dichloropropane	78-87-5	0.6	0.5
1,3-Dichloropropane	142-28-9		0.5
2,2-Dichloropropane	590-20-7		0.5
1,1-Dichloropropene	563-58-6		0.5

Appendix B
Analytical Reporting Requirements

SM 6200B Target Analytes	CASRN	2L Groundwater Standard µg/L	SM 6200B Minimum Reporting Limit, µg/L
cis-1,3-Dichloropropene	10061-01-5	0.4 (cis & trans)	0.5
trans-1,3-Dichloropropene	10061-02-6	0.4 (cis & trans)	0.5
Ethanol	64-17-5	4000	50
Ethylbenzene	100-41-4	600	0.5
Ethyl tert-butyl ether (ETBE)	63-79-23	47	10
2-Hexanone	591-78-6	40	1
Isopropyl benzene	98-82-8	70	0.5
Isopropyl ether	108-20-3	70	0.5
4-Isopropyl toluene	99-87-6	25	0.5
Methylene chloride	75-09-2	5	0.5
Methyl isobutyl ketone (MIBK)	108-10-1	100	0.5
Methyl-tert-butyl ether (MTBE)	1634-04-4	20	0.5
Naphthalene	91-20-3	6	0.5
n-Propylbenzene	103-65-1	70	0.5
Styrene	100-42-5	70	0.5
1,1,1,2-Tetrachloroethane	630-20-6	1	0.5
1,1,2,2-Tetrachloroethane	79-34-5	0.2	0.5
Tetrachloroethene (PCE)	127-18-4	0.7	0.5
Toluene	108-88-3	600	0.5
1,2,3-Trichlorobenzene	87-61-6		0.5
1,2,4-Trichlorobenzene	120-82-1	70	0.5
1,1,1-Trichloroethane	71-55-6	200	0.5
1,1,2-Trichloroethane	79-00-5	0.6	0.5
Trichloroethene (TCE)	79-01-6	3	0.5
Trichlorofluoromethane	75-69-4	2000	0.5
1,2,3-Trichloropropane	96-18-4	0.005	0.5
1,2,4-Trimethylbenzene	95-63-6	400	0.5
1,3,5-Trimethylbenzene	108-67-8	400	0.5
Vinyl acetate	108-05-4	88	1
Vinyl chloride	75-01-4	0.03	0.5
o-Xylene	95-47-6		0.5
m-Xylene	108-38-3		0.5
p-Xylene	106-42-3		0.5
(Xylenes, Total)	1330-20-7	500	1.5

¹ Please note that the Minimum Reporting Limits (MRLs) listed in this table should be routinely achievable but are not corrected for dilution factors (i.e. due to high levels of contamination and/or soil moisture). Dilution factors must be applied, which will result in the elevation of these routinely achievable lower reporting concentrations. Soil moisture content, which is sample specific, will result in sample-specific adjustments in method detection limit and reporting limit concentrations.

Appendix B Analytical Reporting Requirements

- ². The MRL is set at the level of 0.5 µg/L for most of the target analytes listed; however, the MRLs for some target analytes are set at levels higher than 0.5 µg/L due to the lower purging efficiency of some instruments.
- ³ If no value is entered in the standard column, no standard limit has been established. Detection is a violation.
- ⁴ Shading indicates that the MRL for an analyte is greater than its standard limit. If decisions hinge on the concentration of this analyte, then it may be necessary to repeat the analysis in order to reduce the MRL.

Method EPA 625 NC UST Section Required Target Analytes for Petroleum Contaminated Water

EPA 625 Base/Neutrals Target Analytes	CASRN	2L Groundwater Standard µg/L	EPA 625 Minimum Reporting Limit, µg/L
Acenaphthene	83-32-9	80	5
Acenaphthylene	208-96-8	200	5
Anthracene	120-12-7	2000	5
Benz(a)anthracene	56-55-3	0.05	5
Benzo(b)fluoranthene	205-99-2	0.05	5
Benzo(k)fluoranthene	207-08-9	0.5	5
Benzo(g,h,i)perylene	191-24-2	200	5
Benzo(a)pyrene	50-32-8	0.005	5
Benzyl alcohol	100-51-6	700	10
Bis(2-chloroethoxy)methane	111-91-1		5
Bis(2-chloroethyl)ether	111-44-4	0.03	5
Bis(2-chloroisopropyl)ether	108-60-1		5
Bis(2-ethylhexyl)phthalate	117-81-7	3	5
4-Bromophenyl phenyl ether	101-55-3		5
Butyl benzyl phthalate	85-68-7	1000	5
4-Chloroaniline	106-47-8		5
2-Chloronaphthalene	91-58-7		5
4-Chlorophenyl phenyl ether	7005-72-3		5
Chrysene	218-01-9	5	5
Dibenz(a,h)anthracene	53-70-3	0.005	5
Dibenzofuran	132-64-9	28	5
Di-n-butyl phthalate	84-74-2	700	5
1,2-Dichlorobenzene	95-50-1	20	5
1,3-Dichlorobenzene	541-73-1	200	5
1,4-Dichlorobenzene	106-46-7	6	5
3,3'-Dichlorobenzidine	91-94-1		50
Diethyl phthalate	84-66-2	6000	5
Dimethyl phthalate	131-11-3		5
Di-n-octyl phthalate	117-84-0	100	5
1,2-Diphenylhydrazine (as Azobenzene)	122-66-7		10
Fluoranthene	206-44-0	300	5
Fluorene	86-73-7	300	5
Hexachlorobenzene	118-74-1	0.02	5
Hexachlorobutadiene	87-68-3	0.4	5
Hexachlorocyclopentadiene	77-47-4		5

Appendix B
Analytical Reporting Requirements

EPA 625 Base/Neutrals Target Analytes	CASRN	2L Groundwater Standard	EPA 625 Minimum Reporting Limit, $\mu\text{g/L}$
		$\mu\text{g/L}$	$\mu\text{g/L}$
Hexachloroethane	67-72-1		5
Indeno(1,2,3-c,d)pyrene	193-39-5	0.05	5
Isophorone	78-59-1	40	5
1-Methylnaphthalene	90-12-0	1	5
2-Methylnaphthalene	91-57-6	30	5
Naphthalene	91-20-3	6	5
Nitrobenzene	98-95-3		5
N-Nitrosodiphenylamine	86-30-6		5
N-Nitroso-di-n-propylamine	621-64-7		5
Phenanthrene	85-01-8	200	5
Pyrene	129-00-0	200	5
1,2,4-Trichlorobenzene	120-82-1	70	5

EPA 625 Acids Target Analytes			
Benzoic Acid	65-85-0	30000	50
4-Chloro-3-methylphenol	59-50-7		10
2-Chlorophenol	95-57-8	0.4	5
2,4-Dichlorophenol	120-83-2	0.98	5
2,4-Dimethylphenol	105-67-9	100	5
4,6-Dinitro-2-methylphenol	534-52-1		20
2,4-Dinitrophenol	51-28-5		50
2-Methylphenol	95-48-7	400	5
4-Methylphenol	106-44-5	40	5
2-Nitrophenol	88-75-5		5
4-Nitrophenol	100-02-7		20
Pentachlorophenol	87-86-5	0.3	20
Phenol	108-95-2	30	5
2,4,6-Trichlorophenol	88-06-2	4	5

Please note that the Minimum Reporting Limits (MRLs) listed in this table should be routinely achievable but are not corrected for dilution factors (i.e. due to high levels of contamination and/or soil moisture). Dilution factors must be applied, which will result in the elevation of these routinely achievable lower reporting concentrations. Soil moisture content, which is sample specific, will result in sample-specific adjustments in method detection limit and reporting limit concentrations.

² The MRL is set at the level of 5 $\mu\text{g/L}$ for most of the target analytes listed; however, the MRLs for some target analytes are set at levels higher than 5 $\mu\text{g/L}$ due to the lower response of some instruments.

³ If no value is entered in the standard column, no standard limit has been established. Detection is a violation.

⁴ Shading indicates that the MRL for an analyte is greater than its standard limit. If decisions hinge on the concentration of this analyte, then it may be necessary to repeat the analysis in order to reduce the MRL.

Method EPA 602 NC UST Section Required Target Analytes for Petroleum Contaminated Water

EPA 602 Analytes	CASRN	2L Groundwater Standard μg/L	EPA 602 Minimum Reporting Limit, μg/L
Benzene	71-43-2	1	1
Chlorobenzene	108-90-7	50	1
1,2-Dichlorobenzene	95-50-1	20	1
1,3-Dichlorobenzene	541-73-1	200	1
1,4-Dichlorobenzene	106-46-7	6	1
Ethylbenzene	100-41-4	600	1
Toluene	108-88-3	600	1
o-Xylene	95-47-6		1
m-Xylene	108-38-3		1
p-Xylene	106-42-3		1
(Xylenes, Total)	1330-20-7	500	3

¹ If no value is entered in the standard column, no standard limit has been established. Detection is a violation.

Instructions for Completing the VPH Laboratory Reporting Form

- 1) Client Name: Enter the consultant's or contractor's company name.
- 2) Project Name: It could be the Incident Number, facility name, or a residence.
- 3) Site Location: The address
- 4) Laboratory Name: Enter the laboratory's name which the laboratory analyzed the sample. The laboratory should have been certified by the Certification Section of North Carolina.
- 5) NC Certification # (Lab): Enter the certification number issued by the Certification Section of North Carolina.
- 6) Lab ID: The ID number was assigned by the laboratory to track the sample.
- 7) Sample Description: Enter the field ID. It could be the well number or the depth of soil.
- 8) Sample Matrix: Indicate the sample as soil or aqueous
- 9) Dry Weight %: Enter the moisture % of the sample.
- 10) Date Collected: Enter the day that the sample was collected.
- 11) Date Received: Enter the day that sample was received by the laboratory.
- 12) Date Extracted (if Applicable): This entry is for samples that were not preserved before or after they were collected on site, for example, a sample collected with a EnCore sampling device.
- 13) Date Analyzed: Enter the date that the sample was analyzed.
- 14) Diluting Factor:
 - a) Aqueous sample - If the sample was not analyzed straight, enter the dilution factor.
 - b) Soil sample - Based on a 1:1 ratio of methanol: soil and analysis of a 100uL aliquot of the methanol extract in 5mL water. There is a 50 times dilution factor when the lab deposits 100uL extract to 5mL water for the initial purge-and-trap. However, the Report Limit (RL) will not be multiplied on this initial 50 times dilution. The RL will be multiplied only if the analysis needs to be further diluted. (Refer the SOP for VPH calculation in 9.6.2 and 12.0.)
- 15) Report Limit (RL):
 - a) The RLs for target VPH analytes shall be based on the concentration of the lowest calibration standard for the analyte of interest.
 - b) The RLs for the hydrocarbon ranges will be set at 100x the concentration of the lowest calibration standard for the associated analyte. Therefore the RL for aqueous is 100 ug/L, and soil/sediment sample is 5mg/kg. (100ug/Lx5000uL/100uL=5000ug/kg=5mg/kg)
- 16) Unit: Distinguish carefully between ug/l and mg/L; or ug/kg and mg/kg.
- 17 -19): Unadjusted C5-C8 and C9-C12 Aliphatics, and unadjusted C9-C10 Aromatics
The result before the known target compounds within the range are subtracted. An unadjusted value should exclude the concentration of any surrogate(s), internal standards, and/or concentrations of other ranges that elute within the specified range. (The unadjusted concentration of C9-C12 is defined as the value remaining after the concentration of the unadjusted C9-C10 is subtracted from the raw concentration of C9-C12.)
- 20 - 26): Enter the results of individual target compounds. These results should match/confirm each other between the FID and PID detectors if both results are available (WSC-CAM-IVA, Section 2.1, p.17), but it is optional for the lab to report the individual target compounds or not.
- 27 - 29): Adjusted C5-C8 and C9-C12 Aliphatics, and adjusted C9-C10 Aromatics
The result after the known target compounds within the range from the unadjusted C5-C8 and C9-C12 Aliphatics, and unadjusted C9-C10 Aromatics are subtracted.
- 30 - 31): Enter the PID and FID Surrogate % Recovery. Use the one that will be eluted out after Naphthalene, then there is no concern about the overlap.
- 32) Comments: Report the result and qualify any QA/QC issues in a narrative summary.

EPH (Aliphatics/Aromatics) Laboratory Reporting Form

¹ Client Name _____ ⁴ Laboratory Name _____
² Project Name _____ ⁵ NC Certification # (Lab) _____
³ Site Location _____

Sample Information and Analytical Results					
⁶ Lab ID					
⁷ Sample Description					
⁸ Sample Matrix					
⁹ Dry Weight %					
¹⁰ Date Collected					
¹¹ Date Received					
¹² Date Extracted					
¹³ Date Analyzed					
¹⁴ Diluting Factor					
	¹⁵ Report Limit				
¹⁶ Units					
¹⁷ Unadjusted C ₁₁ -C ₂₂ Aromatics*					
¹⁸ Naphthalene					
¹⁹ 2-Methylnaphthalene					
²⁰ Acenaphthylene					
²¹ Acenaphthene					
²² Fluorene					
²³ Phenanthrene					
²⁴ Anthracene					
²⁵ Fluoranthene					
²⁶ Pyrene					
²⁷ Benz(a)anthracene					
²⁸ Chrysene					
²⁹ Benzo(b)fluoranthene					
³⁰ Benzo(k)fluoranthene					
³¹ Benzo(a)pyrene					
³² Indeno(1,2,3-c,d)pyrene					
³³ Dibenz(a,h)anthracene					
³⁴ Benzo(g,h,i)perylene					
³⁵ Unadjusted C ₉ -C ₁₈ Aliphatics*					
³⁶ Unadjusted C ₁₉ -C ₃₆ Aliphatics*					
³⁷ Adjusted C ₁₁ -C ₂₂ Aromatics					
³⁸ Ortho-terphenyl Surr. % Rec.					
³⁹ 1-Chloro-octadecane Surr. % Rec.					
⁴⁰ 2-Bromonaphthalene Fractionation Surr. % Rec.					
⁴¹ 2-Fluorobiphenyl Fractionation Surr. % Rec.					
⁴² % LCS/LCSD 2-Methyl/Naphthalene Breakthrough (≤ 5%)					
⁴³ Comments:					

1/7/2008

Instructions for Completing the EPH Laboratory Reporting Form

- 1) Client Name: Enter the consultant's or contractor's company name.
- 2) Project Name: It could be the Incident Number, facility name, or a residence.
- 3) Site Location: The address
- 4) Laboratory Name: Enter the laboratory's name which the laboratory analyzed the sample. The laboratory should have been certified by the Certification Section of North Carolina.
- 5) NC Certification # (Lab): Enter the certification number issued by the Certification Section of North Carolina.
- 6) Lab ID: The ID number was assigned by the laboratory to track the sample.
- 7) Sample Description: Enter the field ID. It could be the well number or the depth of soil.
- 8) Sample Matrix: Indicate the sample as soil or aqueous
- 9) Dry Weight %: Enter the moisture % of the sample.
- 10) Date Collected: Enter the day that the sample was collected.
- 11) Date Received: Enter the day that sample was received by the laboratory.
- 12) Date Extracted: Enter the date that sample was extracted.
- 13) Date Analyzed: Enter the date that sample was analyzed.
- 14) Diluting Factor: Based on 1-liter aqueous sample or 10 grams of the solid sample. Adjust the final extract volume to 1 ml as undiluted sample. Analytical conditions that require sample dilution include:
 - a) Any target concentration exceeds the concentration of their respective highest calibration standard;
 - b) Any non-target peak exceed twice the peak height of the highest range-specific calibration standard;
 - c) Anytime a saturated chromatographic peak, flap-topped peak, is encountered;
 - d) For 1 ml extract with 5 grams silica gel/cartridges must not be overloaded, no more than 25,000 µg/ml.
 - e) The target post-dilution concentration must be at least 50% of its highest calibration standard.
- 15) Report Limit (RL):
 - a) The RLs for target EPH analytes shall be based on the concentration of the lowest calibration standard for the analyte of interest.
 - b) The RLs for the hydrocarbon ranges will be set at 100x the concentration of the lowest calibration standard for the associated analyte.
- 16) Unit: Distinguish carefully between ug/l and mg/L; or ug/kg and mg/kg.
- 17) Unadjusted C11-C22 Aromatics:

The result before the known Polycyclic Aromatic Hydrocarbon (PAH) target compounds within the range are subtracted. An unadjusted value should exclude the concentration of any surrogate(s), internal standards, and/or concentrations of other ranges that elute within the specified range.
- 18 - 34) Enter the results of individual target compounds. These results should be confirmed by *Gas Chromatography/Mass Spectrometry* at the first time of that particular site, but it is optional for the lab to report the individual target compounds or not.
- 35 - 36) Unadjusted C9-C18 and C19-C36 Aliphatics:

By definition, it is not necessary to identify or quantify individual aliphatic compounds within this range. Therefore, there is no any target compound need to be subtracted. An unadjusted value should exclude the concentration of any surrogate(s), internal standards, and/or concentrations of other ranges that elute within the specified range.
- 37) Adjusted C11-C22 Aromatics:

The result after the known Polycyclic Aromatic Hydrocarbon (PAH) target compounds within the range from the unadjusted C11-C22 Aromatics are subtracted.
- 38 - 41) Enter the Surrogate % Recovery.
- 42) LCS/LCSD naphthalene or 2-methylnaphthalene breakthrough must ≤5% for either constituent in EPH aliphatic fraction. Sample must be re-fractionated if concentration of either compound >5% in aliphatic fraction.
- 43) Comments: Report the result and qualify any QA/QC issues in a narrative summary.

Appendix C - Decontamination of Field Equipment

Decontamination of personnel, sampling equipment, and containers - before and after sampling - must be used to ensure collection of representative samples and to prevent the potential spread of contamination. Decontamination of personnel 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.

A. 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 Liqui-Nox (or a non-phosphate equivalent) or Alconox (or equivalent). Liqui-Nox (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 ultratrace 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.

B. Reagent Storage Containers

The contents of all containers must be clearly marked.

1. Detergents: Store in the original container or in a high density polyethylene (HDPE) or PP container.
2. Solvents
 - a) 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 container.
 - b) Use dispensing containers constructed of glass, Teflon or stainless steel. Note: if stainless steel sprayers are used, any gaskets that contact the solvents must be constructed of inert materials.
3. Analyte-Free Water:
 - a) 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.
 - b) Use glass or Teflon to transport organic-free sources of water on-site. Polypropylene or HDPE may be used, but are not recommended.

- c) Dispense water from containers made of glass, Teflon, high density polyethylene (HDPE) or polypropylene.
- d) Do not store water in transport containers for more than three days before beginning a sampling event.
- e) If working on a project that has oversight from EPA Region 4, use glass containers for the transport and storage of all water.
- f) Store and dispense acids using containers made of glass, Teflon or plastic.

C. General Requirements

1. Before using any equipment, clean/decontaminate all sampling equipment (pumps, tubing, lanyards, 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 you use certified pre-cleaned equipment 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 one of 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.

D. Cleaning Sample Collection Equipment

1. On-Site/In-Field Cleaning

- a) Cleaning equipment on-site is not recommended because:
 1. Environmental conditions cannot be controlled.
 2. Wastes (solvents and acids) must be containerized for proper disposal.
- b) If you must clean equipment on-site or in 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.
 - 1. Scrub, rather than soak, all equipment with sudsy water.
 - 2. 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

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.
 1. Rinse equipment with hot tap water.
 2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent).
 3. If necessary, use a brush to remove particulate matter or surface film.
 4. Rinse thoroughly with hot tap water.
 5. 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.
 6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water.
 7. 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.
 8. Allow to air dry. Wrap and seal as soon as the equipment is air-dried.
 9. 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.
 10. Wrap clean sampling equipment according to the procedure described above.
- b) General Cleaning Procedure for Plastic Sampling Equipment
 1. Rinse equipment with hot tap water.
 2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent).
 3. If necessary, use a brush to remove particulate matter or surface film.
 4. Rinse thoroughly with hot tap water.
 5. Thoroughly rinse (wet all surfaces) with the appropriate acid solution.
 6. Check manufacturer's instructions for cleaning restrictions and/or recommendations.
 7. 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.
 8. Wrap clean sampling equipment according to the procedure described above.

Appendix D – 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:
 - Identity (unique identifier code) and description (including software if used),
 - Manufacturer's name, model number and serial number (if applicable),
 - Calibration checks or other tasks that demonstrate that the equipment performs as expected,
 - Manufacturer's operating and maintenance instructions,
 - Written preventive maintenance schedule that includes the activity, and the frequency of each activity,
 - Date(s) of any preventive maintenance, repairs, malfunctions, etc., and
 - Name of person(s) performing the task(s).

A. 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.

Appendix D
Calibration and Maintenance of Field Equipment

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.
- B. 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.

Appendix E - 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. Each of these major activities is described in detail in its own guideline document. This document should be used in conjunction with these activity-specific guidelines, which provide requirements for analytical methods, as well as TPH screening action limits or risk based clean-up levels, to use in decision-making processes.

A. 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. 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 observed 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, 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 ziplock 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, 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 ziplock 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 Appendix D (Calibration and Maintenance of Field Equipment).
- h) All field-screening results must be documented in the field record or log book.

B. 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 with appropriate power.

In some situations, non-statistical approaches may be considered more appropriate to compare site contaminant levels to background constituent levels when selecting Chemicals of Potential Concern (COPCs). This policy-based guidance includes the non-statistical twice background criterion currently used by EPA Region 4. 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 currently established MSCC may be adjusted.

The UST Section acknowledges that the twice background criterion is policy-based. Both non-statistical approaches are easily used and easily reviewed methods for background screening. The UST Section believes its use of the twice background criterion is health-protective and yields a reasonable decision without recourse to statistics. 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 COPCs is at the discretion of the UST Section regional office supervisor.

1. Data Evaluation and Collection for Background-Based Adjustment of Maximum Soil Contaminant 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. A soil scientist can usually identify fill areas because of the disturbed nature of the soil profile. 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 Central Office 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 using the non-statistical twice background approach 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 near-by, off-site 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 (unit) under investigation. **Specific guidelines cannot be outlined for every site; 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:
 1. 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
 2. 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:
 1. 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;
 2. roads, roadsides, parking lots, areas surrounding parking lots or other paved areas, railroad tracks, railway areas or other areas affected by their runoff;
 3. storm drains or ditches presently or historically receiving industrial or urban runoff;
 4. spill areas, material handling areas, such as truck or rail car loading areas, or near pipelines, fill areas and other areas as determined by NCHWS.
- 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 Chemicals of Potential Concern

COPCs are chemicals that are carried through the risk assessment process. EPA Region 4 Office of Technical Services (OTS) has designed a screening process to identify COPCs, 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 ground water must be considered for high or intermediate risk sites as required by title 15A NCAC 2L .0408. 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. However, the Department may reclassify the risk posed by a release, if warranted by further information. Such information would concern the potential exposure receptors to the discharge or release or upon receipt of new information concerning changed conditions at the site.

3. COPC Selection Process - The process of selecting COPCs includes a screen 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 COPC
5. Eliminate chemicals as COPCs based on comparison to blanks. See Appendix B for an explanation of blank evaluation criteria.
6. Compare maximum detected concentrations in surface soils to the residential or industrial/commercial maximum soil contaminant concentration, whichever is applicable.
 - a) Eliminate the chemical as a COPC for human exposures if the concentration is less than the screening level.
 - b) Industrial screening values should be used for comparison to the subsurface soils data only for construction work scenarios.
7. For naturally occurring inorganics, compare the on-site maximum detected concentration to two times the average site-specific background concentration. Eliminate the chemical as a COPC if it is less than two times the background level. It should be noted that one background sample, if elevated, is usually not acceptable 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 twice background criterion, the maximum detected concentration on site is compared to twice the average background concentration. If the maximum detected concentration is greater than twice the average background concentration, the chemical is included as a COPC.

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.*

C. 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 1, 2, 3, 4, 5 or 6. Approved sample containers and preservation requirements for the specified methods sampling activities are included in Tables 7 or 8. Be sure to consult with your selected laboratory for specific needs and requirements prior to sampling.

1. 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 the collection and shipment of samples. If you use these services, carefully follow the instructions provided and do not discard any preservative that may have been added to the containers. If you do not choose to use a customized kit provided by your laboratory, use only new containers of the appropriate type for the contaminants you are sampling. 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 your 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 as determined in the site's work plan or by the project manager, select an upgradient, undisturbed location to obtain the background samples. See Section B above for additional guidance.
4. Do not collect samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, unless you are characterizing 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 15 for selection of appropriate equipment.

- b) For information on sampling equipment cleaning requirements, see Appendix C.
- c) For information on preservation and holding time requirements, see Tables 7 or 8.

D. 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.

1. 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 is to occur 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 Appendix C.
 - 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) Containers must immediately be preserved according to procedures in this Section. Unless specified otherwise, at a minimum, the samples must be immediately cooled to $4 \pm 2^{\circ}\text{C}$ and this temperature must be maintained throughout delivery to the laboratory.
2. Surface Soil Sampling - Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.
 - a) Remove leaves, grass and surface debris from the area to be sampled.
 - b) Collect samples for volatile organic analyses as described below in Section E.
 - c) Select an appropriate, pre-cleaned sampling device and collect the sample.
 - d) Transfer the sample to the appropriate sample container.
 - e) Clean the outside of the sample container to remove excess soil.
 - f) Label the sample container, place on wet ice to preserve to 4°C , and complete the field notes.
3. Subsurface Soil Sampling - The interval begins at approximately 12 inches below ground surface.

- a) Collect samples for volatile organic analyses as described below in Section E.
 - b) For other analyses, select an appropriate, pre-cleaned sampling device and collect the sample.
 - c) Transfer the sample to the appropriate sample container.
 - d) Clean the outside of the sample container to remove excess soil.
 - e) Label the sample container, place on wet ice to preserve to 4°C, and complete the field notes.
4. 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. In these situations, obtain a sufficient volume of soil so the samples can be 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 to a depth when using the implement becomes impractical.
 1. Dig a hole or trench to the required depth.
 2. Follow the general sample collection procedures outlined above in Section D
 - b) Backhoe - Used for soils from approximately 12 inches to a depth when using the implement becomes impractical.
 1. Dig a trench to the appropriate depth.
 2. 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.
 3. Use a second pre-cleaned utensil to actually collect the sample from the trench.
 4. Follow the general sample collection procedures outlined above in Section D.
 - c) Hand/ Bucket Augers and Hollow Corers - Suitable to reach soils from approximately 12 inches to a depth when using the implement becomes impractical.
 1. Push and rotate the auger into the soil until the bucket is filled.
 2. 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. In this case, it is the sleeve which fills with soil.
 3. Remove the sleeve from the auger and cap.
 4. 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.
 5. 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.
 6. Remove any portion of the sample that has been disturbed and discard.
 7. Follow the general sample collection procedures outlined above in Section D.
- 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. 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.

1. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.
 2. Insert a catcher device in the head ring to prevent the loss of unconsolidated sample material during recovery.
 3. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.
 4. 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.
 5. For volatile organic compounds, collect the sample immediately from the center portion of the split spoon using the procedures described below in Section E.
 6. For other analyses, slice the sample from the center portion of the split spoon. Use a clean, decontaminated utensil.
 7. Select an appropriate, pre-cleaned sampling device and collect the sample.
 8. Transfer the sample to the appropriate sample container.
 9. Clean the outside of the sample container to remove excess soil.
 10. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.
- e) Direct Push Rigs – These may be used for depths greater than 10 feet below ground surface. 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.
1. Place the liner inside the metal probe rod.
 2. Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod.
 3. Advance the rod a full rod length.
 4. Retrieve the rod.
 5. Remove the point holder.
 6. Remove the liner.
 7. Slice the liner to expose the soil.
 8. 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).
 9. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.
 10. Select an appropriate, pre-cleaned sampling device and collect the sample.
 11. Transfer the sample to the appropriate sample container.
 12. Clean the outside of the sample container to remove excess soil.

13. Label the sample container, place on wet ice to preserve to 4°C, 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.
1. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or push manually with a sledge hammer.
 2. Remove the tube from the sampler head.
 3. Extrude the sample from the Shelby tube.
 4. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.
 5. Collect samples for volatile organics immediately from the center portion of the Shelby tube using the procedures described below in Section E.
 6. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.
 7. Transfer the sample to the appropriate sample container.
 8. Clean the outside of the sample container to remove excess soil.
 9. Label the sample container, place on wet ice to preserve to 4°C, 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.
1. Retrieve the sample core by unscrewing the head ring. Slide the sample into a pre-cleaned container.
 2. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.
 3. Remove the sample from the sampler (corer) with a pre-cleaned tool.
 4. Transfer the sample to the appropriate sample container.
 5. Clean the outside of the sample container to remove excess soil.
 6. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.
5. 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.
6. Composite Soil Samples - Composite sampling will be used for soil samples collected for soil remediation permitting. Samples will be composited as a means to reduce sample handling and analytical costs. Each composite sample for soil remediation will be composed of six primary samples. The six primary samples that comprise each composite sample shall be mixed together by the laboratory, with a representative portion of this mixture to be analyzed.

Composite samples are prepared differently depending on the analysis required and the relative tendency of the contaminant analyzed to vaporize.

- 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 8260B). 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
- b) The six primary samples collected for metals analysis may be added to a single sample container, clearly marked as composite, for the laboratory to dry, sieve, mix and sub-sample for analysis of a representative portion.
- c) The six primary samples collected for semi-volatiles analysis (i.e. TPH DRO or SW-846 8270D) may be added to a single sample container, clearly marked as composite, for the laboratory to mix and sub-sample for analysis of a representative portion.

7. General Composite Sample Collection Procedures

- a) Soil samples taken to 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.
- b) 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.
- c) 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.
- d) Immediately upon removal from the ground, each primary soil sample must be placed in the appropriate container (See Table 7). 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
- e) Collect six representative portions of soil at the same time the six primary samples are collected. These are to be added to a single, unpreserved container for determination of soil moisture content and dry weight correction factors.

8. Initial Characterization Sampling - 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. Soil samples for laboratory analysis must be collected from each stockpile.

- 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 Section 5.1 B of the “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 (See Section 5.1 B).
 - b) 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 7.
 - c) 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 DWQ-certified laboratory.
9. 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
 1. 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. 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. See Section 5.1 B of the “Guidelines for Ex Situ Petroleum Contaminated Soil Remediation” for soil analytical methods.
 2. 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 7.
 3. 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 DWQ-certified laboratory.
 - b. Soil Sampling for Containment and Treatment Technologies
 1. 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. See

Section 5.1 B of the “Guidelines for Ex Situ Petroleum Contaminated Soil Remediation” for soil analytical methods.

2. 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 7.
3. 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 DWQ-certified laboratory.

E. 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. DENR 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:
 1. A sample field preserved with sodium bisulfate has a 14-day holding time.
 2. An unpreserved sample (no acid) must be analyzed within 48 hours.
 3. An unpreserved sample in a vial with premeasured analyte-free water must be analyzed within 48 hours.
 4. 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.
 5. 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.
 6. 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. DENR 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:

1. A sample field preserved with methanol has a 14-day holding time.
2. An unpreserved sample (no methanol) must be analyzed within 48 hours.
3. 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.
4. 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.
5. If transported to the laboratory in a sealed coring device, the samples must be analyzed within 48 hours.
6. 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.

NOTE: *While all collection and preservation options for the high level method will be allowed, STF reimbursement will not be made for Sealed-Tube Sampling/Storage Devices used to collect soil remediation permitting composite samples. There are two other less expensive options for collection and preservation listed below. Reimbursement under task code 4.010 and 408 will only be allowed for closure and assessment and corrective action sampling activities conducted through 2003.*

2. Basic Options for Collection and Preservation - Acceptable options to collect and preserve volatiles soil samples to be analyzed and reported to the N.C. DENR 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 7 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 DWQ 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.

1. 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.
2. 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.
3. 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.
4. 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.

1. 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).
2. 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.
3. 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

Although the use of these devices will continue to be allowed, State Trust Fund reimbursement under task code 4.010 and 408 will only be allowed for closure and assessment and corrective action sampling activities conducted through 2003.

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.

1. Obtain pre-cleaned and/or disposable samplers/containers that allow the collection and air-tight storage of at least five grams of soil.
2. 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.
3. 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, +/-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 Appendix C).
- 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:
 1. Do not lose any of the liquid either through evaporation or spillage.
 2. Do not use a vial if some of the liquid has spilled, or if it appears that some has leaked during transport.
 3. Use the laboratory-supplied container label for identification information.
 4. Do not apply any additional labels or chain of custody seals to the pre-weighed container.
 5. Do not interchange vial caps or septa.
 6. 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.
 - 1. 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.
 - 2. 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.
 - 3. 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.

F. Shipping Sample Containers/Collection Devices on Ice

All samples must be stored and transported carefully to prevent samples from breaking and to maintain a temperature of approximately 4°C. 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 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.

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.

G. Laboratory Preparation and Shelf-life of Methanol Preserved Vials

1. Preparation

- 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. Shelf-life of 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.*

H. Shipping Methanol Preserved Samples

NOTE: *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.*

1. 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 your shipping company for complete details.
2. 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."
- 3. 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
- 4. Labeling & Placarding - Labeling and placarding is not required for valid small quantity exemptions (per 49 CFR Section 173.118).

Appendix F Collecting 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 may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques.

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. They are generally found at wellfields, industrial facilities and private residences. See Appendix G for procedures to sample wells with plumbing in place.

Air Strippers or Remedial Systems: These types of systems are installed as remediation devices. Sample wells associated with these systems as outlined in Appendix G.

A. 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 3 or 5. Table 8 lists approved sample containers, preservation and hold times for the specified methods. Be sure to consult your 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 11, 12, 13, 14 and 15 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 sampling, and sampling low permeability aquifers or formations.
 - B. 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.
 - C. Install a check valve at the output side of the pump to prevent backflow.
 - D. If purging **and** sampling for organics:
 - The entire length of the delivery tube must be Teflon, polyethylene or polypropylene (PP) tubing.
 - The electrical cord must be sealed in Teflon, polyethylene or PP and any cabling must be sealed in Teflon, polyethylene or PP, or be constructed of stainless steel.
 - 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, PP 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:
 - The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, polyethylene or PP.
 - The entire length of the delivery tube must be Teflon, polyethylene or PP.
 - Any cabling must be sealed in Teflon, polyethylene or PP, 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 below in Section D 4.
 - b) Sampling: Bailers must be used with caution following the procedures outlined below in Section D 4.
 - 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 PP bailers may be used to sample all analytes.
 - 2. Use disposable bailers when sampling grossly contaminated sample sources.
 - 3. NCDENR 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 lanyard to minimize contact with the bailer surface.
3. Lanyards
- a) Lanyards 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 PP.
 - b) Discard cotton twine, nylon, and non-stainless steel braided lanyards after sampling each monitoring well.
 - c) Decontaminate stainless steel, coated Teflon, polyethylene and PP lanyards between monitoring wells (see Appendix C). They do not need to be decontaminated between purging and sampling operations.

B. 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 because improper bailing:
 1. Introduces atmospheric oxygen, which may precipitate metals (i.e., iron) or cause other changes in the chemistry of the water in the sample (i.e., pH).
 2. Agitates groundwater, which may bias volatile and semi-volatile organic analyses due to volatilization.
 3. Agitates the water in the aquifer and resuspends fine particulate matter
 4. Surges the well, loosening particulate matter in the annular space around the well screen.
 5. May introduce dirt into the water column if the sides of the casing wall are scraped.

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.
 - 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 you place a protective covering 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 or chalked tape 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. Record the measurement.
- a) Electronic Probe
 1. Decontaminate all equipment before use.
 2. Follow the manufacturer's instructions for use.
 3. Record the measurement.
 - b) Chalked Line Method
 1. Decontaminate all equipment before use.

2. Lower chalked tape into the well until the lower end is in the water. This is usually determined by the sound of the weight hitting the water.
 3. Record the length of the tape relative to the reference point (see Section 3.2 above).
 4. Remove the tape and note the length of the wetted portion.
 5. Record the length.
 6. Determine the depth to water by subtracting the length of the wetted portion from the total length. Record the result.
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.

C. 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. Therefore, you must measure the volume during the purging operation.

- 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.

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

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

2. 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

3. Make new measurements each time the pumping rate is changed.

- c) Use a totalizing flow meter.

1. Record the reading on the totalizer prior to purging.
2. Record the reading on the totalizer at the end of purging.
3. 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..
4. 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:

- 1. Purging rate.
 - 2. Drawdown in the well, if any.
 - 3. A description of the process and the data used to design the well.
 - 4. The equipment and procedure used to install the well.
 - 5. The well development procedure.
 - 6. 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:
- 1. Temperature: $\pm 0.2^{\circ} \text{C}$
 - 2. pH: ± 0.2 Standard Units
 - 3. Specific Conductance: $\pm 5.0\%$ of reading
 - 4. Dissolved Oxygen: ± 0.2 mg/L or 10%, whichever is greater
 - 5. 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:
- 1. Purging rate.
 - 2. Drawdown in the well, if any.
 - 3. 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.

4. 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.
5. A description of the process and the data used to design the well.
6. The equipment and procedure used to install the well.
7. The well development procedure.
8. 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.

1. Purging rate.
2. Drawdown in the well, if any.
3. 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.
4. 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.
5. A description of the process and the data used to design the well.
6. The equipment and procedure used to install the well.
7. The well development procedure.
8. 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:

1. Place the pump or tubing intake within the well screened interval.

2. Use very small diameter Teflon, polyethylene or PP tubing and the smallest possible pump chamber volume. This will minimize the total volume of water pumped from the well and reduce drawdown.
 3. Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.
 4. Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.
 5. Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).
 6. 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”). See section C.3.d. above.
5. Lanyards
- a) Securely fasten lanyards, if used, to any downhole equipment (bailers, pumps, etc.).
 - b) See Section A above for acceptable lanyard types and use.
 - c) Use bailer lanyards in such a way that they do not touch the ground surface.

D. Wells Without Plumbing

1. Tubing/Pump Placement

- a) If you are 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 you intend to collect samples 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.

2. Non-dedicated (portable) pumps

a) Variable Speed Peristaltic Pump

1. Wear sampling gloves to position the decontaminated pump and tubing.
2. Attach a short section of tubing to the discharge side of the pump and into a graduated container.
3. Attach one end of a length of new or pre-cleaned tubing to the pump head flexible hose.
4. Place the tubing as described in one of the options listed above.
5. Change gloves before beginning to purge.

6. Measure the depth to groundwater at frequent intervals.
 7. Record these measurements.
 8. Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
 9. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
 10. 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.
 11. Record the purging rate each time the rate changes.
 12. Measure the purge volume.
 13. Record this measurement.
 14. Decontaminate the pump and tubing between wells (see Appendix C) or if precleaned tubing is used for each well, only the pump.
- b) Variable Speed Centrifugal Pump
1. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
 2. Wear sampling gloves to position the decontaminated pump and tubing.
 3. Place the decontaminated suction hose so that water is always pumped from the top of the water column.
 4. Change gloves before beginning to purge.
 5. Equip the suction hose with a foot valve to prevent purge water from re-entering the well.
 6. Measure the depth to groundwater at frequent intervals.
 7. Record these measurements.
 8. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
 9. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
 10. 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.
 11. Record the purging rate each time the rate changes.
 12. Measure the purge volume.
 13. Record this measurement.
 14. Decontaminate the pump and tubing between wells (see Appendix C) or if precleaned tubing is used for each well, only the pump.
- c) Variable Speed Electric Submersible Pump
1. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
 2. Wear sampling gloves to position the decontaminated pump and tubing.
 3. Carefully position the decontaminated pump as described in one of the options in Section D 1 above.
 4. Change gloves before beginning to purge.
 5. Measure the depth to groundwater at frequent intervals.
 6. Record these measurements.

7. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
 8. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
 9. 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.
 10. Record the purging rate each time the rate changes.
 11. Measure the purge volume.
 12. Record this measurement.
 13. Decontaminate the pump and tubing between wells (see Appendix C) or only the pump if pre-cleaned tubing is used for each well.
- d) Variable Speed Bladder Pump
1. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
 2. Wear sampling gloves to position the decontaminated pump and tubing.
 3. Attach the tubing and carefully position the pump.
 4. Change gloves before beginning purging.
 5. Measure the depth to groundwater at frequent intervals.
 6. Record these measurements.
 7. To minimize drawdown, adjust the purging rate so that it is equivalent to the well recovery rate.
 8. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
 9. 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.
 10. Record the purging rate each time the rate changes.
 11. Measure the purge volume.
 12. Record this measurement.
 13. Decontaminate the pump and tubing between wells (see Appendix C) or if pre-cleaned tubing is used for each well, only the pump.
3. Dedicated Portable Pumps - Place dedicated pumps as described in one of the options in Section D 1 above.
- a) Variable Speed Electric Submersible Pump
1. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
 2. Wear sampling gloves.
 3. Measure the depth to groundwater at frequent intervals.
 4. Record these measurements.
 5. Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
 6. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdraw with the recharge rate.
 7. Record the purging rate each time the rate changes.
 8. Measure the purge volume.

9. Record this measurement.
- b) Variable Speed Bladder Pump
 1. Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
 2. Wear sampling gloves.
 3. Measure the depth to groundwater at frequent intervals.
 4. Record these measurements.
 5. Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
 6. If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdraw with the recharge rate.
 7. Record the purging rate each time the rate changes.
 8. Measure the purge volume.
 9. Record this measurement.
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.
 1. Minimize handling the bailer as much as possible.
 2. Wear sampling gloves.
 3. Remove the bailer from its protective wrapping just before use.
 4. Attach a lanyard of appropriate material (see Section A).
 5. Use the lanyard to move and position the bailer.
 6. Lower and retrieve the bailer slowly and smoothly.
 7. Lower the bailer carefully into the well to a depth approximately a foot above the water column.
 8. 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.
 9. 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.
 10. Allow time for the bailer to fill with aquifer water as it descends into the water column.
 11. 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.
 12. Measure the purge volume.
 13. Record the volume of the bailer.
 14. 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.
 15. 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.

E. Groundwater Sampling Techniques

1. Purge wells using the techniques outlined in Section B.
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.*

- 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:
 1. Handle the sampling equipment as little as possible.
 2. Minimize the equipment that is exposed to the sample.
- d) Dedicated Sampling Equipment
 1. Whenever possible, use dedicated equipment. It significantly reduces the chance of cross-contamination.
 2. Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).
 3. All material construction and restrictions from Tables 13 and 14 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.
 4. Cleaning/Decontamination
 - A. 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.
 - B. Clean or make sure any permanently mounted tubing is clean before installation.
 - C. Change or clean tubing when the pump is withdrawn for servicing.
 - D. Clean any replaceable or temporary parts as specified in Appendix C.
 - E. Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.
 - F. Clean or make sure dedicated bailers are clean before placing them into the well.
 - G. Collect an equipment blank on dedicated bailers before introducing them into the water column.
 - H. Suspend dedicated bailers above the water column if they are stored in the well.

F. Sampling Wells Without Plumbing

1. Sampling with Pumps – The following may be used to sample for organics: Variable speed stainless steel and Teflon submersible pumps; and stainless steel, Teflon 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, polyethylene or PP. Extractable organics may be collected through a peristaltic pump if flexible interior-wall Teflon, polyethylene or PP 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 Section A when using pumps to collect samples.

a) Peristaltic Pump

1. Volatile Organics:

- A. Remove the drop tubing from the inlet side of the pump.
- B. Submerge the drop tubing into the water column.
- C. Prevent the water in the tubing from flowing back into the well.
- D. Remove the drop tubing from the well.
- E. Carefully allow the groundwater to gravity drain into the sample vials. Avoid turbulence. Do not aerate the sample.
- F. Repeat steps B through E until enough vials are filled.

Alternatively

- G. Use the pump to fill the drop tubing.
- H. Quickly remove the tubing from the pump.
- I. Prevent the water in the tubing from flowing back into the well.
- J. Remove the drop tubing from the well.
- K. Carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample.
- L. Repeat steps G through K. until enough vials are filled.

Or:

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

2. Extractable Organics

- A. If the tubing in the pump head is polyethylene or PP, or is Teflon lined, the samples may be collected through the pump.
- B. If the tubing in the pump head is not polyethylene or PP, or is not Teflon lined, use the pump and vacuum trap method.
 1. Assemble the components of the pump and trap according to Figure 4.
 2. The sample container should be the trap.
 3. All equipment that contacts the groundwater before the sample container must be constructed of Teflon, polyethylene, PP, 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.
 4. Connect the outflow tubing from the container to the influent side of the peristaltic pump.
 5. Turn the pump on and reduce the flow rate to a smooth and even flow.
 6. Discard a small portion of the sample to allow an air space.
 7. Preserve (if required), label and complete the field notes.

3. 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.
- b) Variable Speed Bladder Pump
1. If sampling for organics, the pump body must be constructed of stainless steel and the valves and bladder must be Teflon. All tubing must be Teflon, polyethylene, or PP and any cabling must be sealed in Teflon, polyethylene or PP, or made of stainless steel.
 2. After purging to a smooth even flow, reduce the flow rate.
 3. When sampling for volatile organic compounds, reduce the flow rate to 100-200 mL/minute, if possible.
- c) Variable Speed Submersible Pump
1. The housing must be stainless steel.
 2. If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon, polyethylene or PP. The delivery tubing must be Teflon, polyethylene or PP; the electrical cord must be sealed in Teflon; and any cabling must be sealed in Teflon or constructed of stainless steel.
 3. After purging to a smooth even flow, reduce the flow rate.
 4. When sampling for volatile organic compounds, reduce the flow rate to 100-200 mL/minute, if possible.
2. Sampling with Bailers - A high degree of skill and coordination are necessary to collect representative samples with a bailer.
- a) General Considerations
1. Minimize handling the bailer as much as possible.
 - A. Wear sampling gloves.
 - B. Remove the bailer from its protective wrapping just before use.
 - C. Attach a lanyard of appropriate material (see Section A above).
 - E. Use the lanyard to move and position the bailers.
 2. Do not allow the bailer or lanyard to touch the ground.
 3. Rinsing
 - 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. Use the technique described in 2 b, Bailing Technique, below.
 - D. Discard the water appropriately.
 - E. Do not rinse the bailer if Oil and Grease samples are to be collected.
- b) Bailing Technique
1. Collect all samples that are required to be collected with a pump before collecting samples with the bailer.
 2. Raise and lower the bailer gently to minimize stirring up particulate matter in the well and the water column, which can increase sample turbidity.
 3. Lower the bailer carefully into the well to a depth approximately a foot above the water column. 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.
 4. Do not lower the top of the bailer more than one foot below the top of the water table, so that water is removed from the top of the water column.

5. Allow time for the bailer to fill with aquifer water as it descends into the water column.
 6. Do not allow the bailer to touch the bottom of the well or particulate matter will be incorporated into the sample. 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.
 7. Lower the bailer to approximately the same depth each time.
 8. Collect the sample.
 - A. Install a device to control the flow from the bottom of the bailer and discard the first few inches of water.
 - B. Fill the appropriate sample containers by allowing the sample to slowly flow down the side of the container.
 - C. Discard the last few inches of water in the bailer.
 9. Repeat steps 1 through 9 for additional samples.
 10. As a final step measure the DO, pH, temperature, turbidity and specific conductance after the final sample has been collected. Record all measurements and note the time that sampling was completed.
3. Sampling Wells with Floating Non-Aqueous Phase Liquid: NCDENR 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. NCDENR 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.
- a) Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.
 1. Non-aqueous phase liquid is normally sampled for two reasons:
 - A. Documentation for its existence and thickness; and
 - B. 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.
 2. Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable polyethylene and PP bailers are also acceptable. Other wide mouth vessels may be used for sampling non-aqueous phase liquid in an excavation.
 3. Monitoring Well
 - A. 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.
 - B. Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.
 - C. Pour a portion of the product into a glass sample container.

- D. 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.
- 4. Equipment that is dedicated to sampling non-aqueous phase liquid does not need to be cleaned according to the standard, full decontamination protocols. Acrylic or PVC bailers that are never used for trace contaminant sampling may be cleaned as listed below. It is recommended that all cleaning be done in the lab, office or base of operations and not in the field.
 - A. Disassemble bailers and intermediate vessels and soak in hot, sudsy tap water using a brush to clean away all particulates and greasy films.
 - B. Rinse with hot tap water.
 - C. Thoroughly rinse with analyte free water.
 - D. An optional acid rinse may be used to strip the equipment of any hard to clean residues.
 - E. The solvent rinse is not mandatory since this equipment is not used for contaminant sampling, other than the product itself. It is not recommended on clear acrylic.
- b) Sampling Below Product
 - 1. This type of depth-specific sampling is an attempt to sample the dissolved constituents in the water column below the product layer. This sampling is performed only at the request of the UST Section.
 - 2. These data provide information that helps define adequate groundwater treatment. Without these data, incorrect and sometimes unnecessarily expensive remediation techniques may be designed for a situation where they are not required.
 - 3. There are some substantial logistical problems involved with sending a sampler through non-aqueous phase liquid to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of “self-engineered” equipment or coverings may be the best option.
 - 4. These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.
 - 5. Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.
 - 6. Although not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.
 - A. Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.
 - B. Introduce the wrapped hose slowly so as not to introduce any more product into the dissolved layer located below.
 - C. Perform this procedure with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet. Be aware that actual sampling may occur several feet below the product layer.

G. 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.

Appendix G Collecting Samples from Wells with Plumbing in Place

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. See 15A NCAC 2N .0304 for monitoring requirements associated with enhanced leak detection. Water supply wells may also be sampled if there is suspicion of a release from a UST facility.
 - a) 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. You must choose 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.

b) Collecting Volatile Organic Samples

1. Equipment Needed

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

2. Sampling Procedure

- A. Run the water from your well for at least 15 minutes. If you have a deep well you will need to run the water for longer (purging three well volumes is best). If your tap or spigot is located directly before a holding tank, it is a good idea to open a tap after the holding tank to prevent any backflow into the tap where you will take your sample. This will ensure that the water you collect is water “fresh” from the well and not from the holding tank.
- B. After running the water for at least 15 minutes, reduce the flow of water. The flow should be reduced to a trickle but not so slow that it begins to drip. A smooth flow of water will make collecting the samples easier and more accurate.
- C. 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.
- D. 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 (see Figure 1 below). 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.
- E. 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. (see Figure 2 below) It is a good idea not to overfill the vial, especially if an acid preservative is present in the vial.



Figure 1



Figure 2

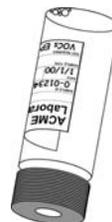


Figure 3

Appendix G
Collecting Samples from Wells with Plumbing in Place

- F. Carefully replace and screw the cap onto the vial. Some water may overflow as the cap is put on.
 - G. 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 (see E above). Repeat as necessary.
 - H. After two samples 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.
- c) Collecting Extractable Organic and/or Metals Samples
- 1. Equipment Needed
 - A. 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]
 - B. Disposable gloves and protective goggles
 - C. Ice Chest/Cooler
 - D. Ice
 - E. Packing materials (sealable plastic bags, bubble wrap, etc.)
 - F. Lab forms
 - 2. Sampling Procedure
 - A. Run the water from your well for at least 15 minutes. If you have a deep well you will need to run the water for longer (purging three well volumes is best). If your 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 you will take your sample. This will ensure that the water you collect is water “fresh” from the well and not from the holding tank.
 - B. After running the water for at least 15 minutes, reduce the flow of water. A low flow of water will make collecting the samples easier and more accurate.
 - C. Remove the cap of a SVOC or metals bottle and hold it under the stream of water to fill it.
 - D. The bottle does not have to be completely filled (i.e., you can leave an inch or so of headspace in the bottle).
 - E. 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.

Appendix H 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. General Cautions - 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 11 and 12 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 8. For information on sampling equipment cleaning requirements, see Appendix C. Sampling events will most frequently employ the suction lift sample gathering system.

A. 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.).

1. Grab directly into sample container
2. Slowly submerge the container, opening neck first, into the water.
3. 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.
4. Return the filled container quickly to the surface.

5. 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.
 6. Add preservatives, securely cap container, label and complete field notes.
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 prepreserved 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.
1. Rinse the intermediate vessel with ample amounts of site water prior to collecting the first sample.
 2. Collect the sample as outlined above using the intermediate vessel.
 3. 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 11 and 12). 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 PP tubing is used in the pump head or if used with the organic trap setup as shown in Figure 4.
1. Lower appropriately pre-cleaned tubing to a depth of 6 – 12 inches below water surface, where possible.
 2. Pump 3 – 5 tube volumes through the system to acclimate the tubing before collecting the first sample.
 3. Fill individual sample bottles via the discharge tubing. Be careful not to remove the inlet tubing from the water.
 4. 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. You may also use pumps with tubing or double check-valve bailers. Certain construction material details may preclude its use for certain analytes (see Tables 11 and 12). Many Kemmerer samplers are constructed of plastic and rubber that preclude their use for all volatile and extractable organic sampling. Some newer devices are constructed of stainless steel or are all Teflon or Teflon-coated. These are acceptable for all analyte groups without restriction.
1. Measure the water column to determine maximum depth and sampling depth prior to lowering the sampling device.
 2. Mark the line attached to the sampler with depth increments so that the sampling depth can be accurately recorded.

3. Lower the sampler slowly to the appropriate sampling depth, taking care not to disturb the sediments.
 4. At the desired depth, send the messenger weight down to trip the closure mechanism.
 5. Retrieve the sampler slowly.
 6. 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.
 7. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described in section A above.
- 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.
1. 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.
 2. Slowly lower the bailer to the appropriate depth. Upon retrieval, the two check-valves seat, preventing water from escaping or entering the bailer.
 3. Rinse the sampling device with ample amounts of site water prior to collecting the first sample.
 4. 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 12-volt peristaltic pump. Use appropriately pre-cleaned, silastic tubing in the pump head and attach high density polyethylene (HDPE), Tygon, etc. tubing to the pump (see restrictions listed in Tables 11 and 12). 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 PP tubing is used in the pump head, or if used with an organic trap setup.
1. Measure the water column to determine the maximum depth and the sampling depth.
 2. 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 lanyard (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.
 3. Turn the pump on and allow several tubing volumes of water to be discharged before collecting the first sample.
 4. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described above.

Appendix I Collecting Air Samples

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. 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 11 and 12 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 Appendix C. Sampling events will most frequently employ the Tedlar bag sampling system.
3. Air Sampling Techniques - Use the following protocols when collecting air samples. Adhere to all general protocols applicable to air sampling when following the sampling procedures addressed below. 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.

A. Grab Sampling with Tedlar bags

1. 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 (see Figure 6 p. 45).
 - b) Leak-check both the bag and the container.
 - c) Connect the vacuum line from the needle valve to the Teflon 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.
2. 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 (\pm 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.
3. 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 you have used the entire sample tube 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.
4. Summa 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 summa canister 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. When collected in canisters, samples can be screened for high levels prior to low level analysis.