

5.0 SAMPLING PROCEDURES

5.1 INTRODUCTION

This section discusses practices and procedures for use by DEC Bureau of Spill Prevention and Response (DEC BSPR) personnel or contractor during field operations to ensure the collection of representative samples. All sampling activities conducted by DEC BSPR personnel or contractor are conducted with the expectation that they will be used for enforcement purposes, unless specifically stated to the contrary in advance of the field investigation. Therefore, the use of proper sampling procedures cannot be over emphasized. The collection of representative samples depends upon:

- ensuring that the sample taken is truly representative of the material or medium being sampled;
- using proper sampling, sample handling, preservation, and quality control techniques;
- properly identifying the collected samples and documenting their collection in permanent field records;
- maintaining sample chain-of-custody; and
- protecting the collected samples by properly packing and transporting (shipping) them to a laboratory for analysis.

The objectives of this section are to present:

- general considerations that must be incorporated in all sampling operations conducted by the DEC BSPR personnel or contractor;
- specific sampling site selection and collection procedures for individual media; and
- specific sampling quality assurance procedures as well as equipment calibration and maintenance requirements for sampling equipment.

5.2 GENERAL CONSIDERATIONS

The following factors and procedures shall be considered and implemented in planning and conducting all sampling operations with the specific objectives and scope of individual site investigation. Representative sampling sites are dependent on the type of investigation being undertaken and are discussed under the sample procedures for each medium later in this section. The type of sampling equipment to be used is also discussed in Section 3.0 and the sampling procedure in this section. Section 4.0 describes the equipment cleaning procedures.

5.2.1 Sampling Equipment Construction Material

The material that sampling equipment is constructed of can affect sample analytical results. Materials must not contaminate the sample being collected and must be readily cleaned so that samples are not cross-contaminated. The standard materials for sampling equipment used for trace organic compounds or metals analyses are, in order of decreasing desirability; Teflon, glass, stainless steel and steel.

5.2.2 Selection of Parameters to be Measured

Parameters to be measured are usually dictated by the purpose of an investigation and should be based on

required monitoring conditions (NPDES or RCRA permits for example) or on the field investigator's or requester's knowledge of the problem being investigated.

5.2.3 Dissolved and Particulate Sample Fractions

A water sample is generally composed of dissolved and particulate fraction. When it is necessary to analyze samples for each fraction, instead of the total sample, it may be necessary to filter the sample in the field. Field filtration should be conducted only for conventional analytical parameters or metals; samples that will be analyzed for organic compounds shall not be filtered in the field.

5.2.4 Required Sample Volumes

The volume of samples obtained should be sufficient to perform all required analyses with an additional amount collected to provide for quality control needs, split samples, or repeat examinations. Individual aliquots of a composite sample should be at least 100 milliliters in order to minimize sample solids bias when using a peristaltic pump.

Although the volume of sample required by contract laboratories depends on the analyses to be performed, the amount of sample required for a complete water or wastewater analysis can be up to two gallons (7.6 liters) for each laboratory receiving a sample. However, the laboratory receiving the sample should be consulted for any specific volume requirements.

The volumes of samples collected from waste sources at hazardous waste sites or samples from sources which are known to be toxic should be kept to an absolute minimum.

The sample volume required for each analysis is the volume of the standard container less ullage (empty space) required for sample mixing by laboratory personnel and safe shipment of samples to the laboratory. Sampling personnel shall allow a minimum of ten percent ullage in every sample container for this purpose. The only exceptions are samples collected for purgeable organic analysis (VOA) or dissolved gases such as sulfides for which sample containers must be completely filled.

5.2.5 Selection and Proper Preparation of Sample Containers

The type of sample container is dictated by the analyses required. Standard sample containers to be used by sampling personnel are presented in Appendix 5X.1. Special sample container preparation requirements are in Section 4.0.

5.2.6 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservations required for routine analyses of samples are given in Appendix 5X.1. All samples should be preserved immediately upon collection in the field. The only samples that should not be immediately preserved in the field are:

- Samples collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials. Barrel, drum, closed container, spillage, or other source samples from hazardous waste sites are not to be preserved with any chemical. These samples may be preserved with ice, if necessary.
- Samples that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved using the procedures given in Appendix 5X.1.
- Well or ground water samples that contain visible sediment, that are not filtered in the field, shall

not be preserved with nitric acid in the field. These samples shall be preserved with ice and taken to the laboratory for additional sample preparation.

- Samples for metals analysis which are to be shipped as soon as possible. They shall not be preserved with nitric acid in excess of the amount specified in Appendix 5X.3.
- Samples for purgeable organic compounds (VOA) analyses which are shipped by air shall not be preserved with hydrochloric acid in excess of the amount specified in Appendix 5X.3.

All samples preserved with chemicals shall be clearly identified by indicating on the sample tag that the sample is preserved. If the samples are not preserved, field records shall indicate why.

5.2.7 Sample Holding Times

The elapsed time between sample collection and initiation of laboratory analyses must be within a prescribed time frame for each individual analysis. Sample holding times for all routine samples are shown in Appendix 5X.1.

5.2.8 Sample Handling and Mixing

Once a sample has been collected, it may have to be split into separate containers for different analyses. The best way to split liquid samples is to continually stir the sample contents with a clean pipette or pre-cleaned Teflon rod and allow the contents to be alternately siphoned into respective sample containers using Teflon or PVC (Tygon type) tubing. Teflon must be used when analyses for organic compounds or trace metals are to be conducted. Any device used for stirring, or tubing used for siphoning, must be cleaned in the same manner as other equipment (Section 4.0).

A true split of soil, sediment, or sludge samples is almost impossible to accomplish under field conditions. The more moisture samples contain, the more difficult it is to split them. Procedures such as the one outlined in Section 5.7.4.5 should be used to obtain a homogenous sample. Even when such procedures are followed, the sample should be considered a duplicate and not a split sample.

After collection, all samples should be handled as few times as possible. Sample handling personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that melted ice cannot cause sample containers to become submerged, as this may result in sample cross-contamination. Plastic bags, such as zip-lock bags, should be used to prevent cross-contamination when small sample containers (e.g., VOA's or bacterial samples) are placed in ice chests.

5.2.9 Special Precautions for Trace Contaminant Sampling

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care must be taken to prevent cross-contamination of these samples. The following precautions shall be taken when trace contaminants are of concern:

- A clean pair of new, disposable gloves will be worn each time a different point or location is sampled;
- Sample containers for source samples or samples suspected of containing high concentrations of contaminants shall be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.;
- If possible, ambient samples and source samples should be collected by different field teams. If separate collection is not possible, all ambient samples shall be collected first and placed in separate

ice chests or shipping containers. Samples of waste or highly contaminated samples shall never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected of containing high concentrations of contaminants shall be lined with new, clean, plastic bags.

- If possible, one member of the field team should take all the notes, fill out tags, etc., while the other member does all of the sampling.
- When sampling surface waters, the water sample should always be collected before the sediment sample is collected.
- Sample collection activities should proceed progressively from the least contaminated area to the most contaminated area (if this fact is known).
- Sampling personnel should use equipment constructed of Teflon, stainless steel, or glass that has been properly precleaned (Section 4.0) for collecting samples for trace metals or organic compounds analyses. Teflon or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC shall not be used to collect samples for trace organic compounds analyses.

5.2.10 Sample Identification

All samples will be fully documented, as outlined in Appendix 5X.2, in the field records, on the field sample chain-of-custody record, and on the sample tags.

5.2.11 Procedures for Identifying Potentially Hazardous Samples

Any sample either known or thought to be hazardous should be so identified on both the sample tag and the field sample chain-of-custody sheet. Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed.

5.2.12 Collection of Auxiliary Data

All auxiliary data such as flow measurements, photographs of sampling sites, meteorological conditions, and other observations shall be entered into field records when the auxiliary data are collected. Auxiliary data relative to a particular sampling location should be collected as close to the sample collection time as possible. Specific types of auxiliary data to collect for each medium sampled are discussed later in this section.

5.2.13 Time Records

All records of time shall be kept using local time in the 2400 hour time format and shall be recorded to the nearest five minutes.

5.2.14 Transporting and Shipping of Samples

Samples may be hand delivered to the laboratory using government owned (or private) vehicles or they may be shipped by common carrier. All sample handling personnel must be aware that certain samples are hazardous materials and, as such, are regulated by the U.S. Department of Transportation under the Transportation Safety Act of 1974. These regulations are contained in Title 49, CFR, Parts 110-119. All DEC BSPR employees shall be aware of and meet the regulations that have been set up by the Transportation Safety Act of 1974 when shipping samples by common carrier.

5.2.15 Sample Chain-of-Custody

DEC BSPR employees or all sample handling personnel shall maintain chain-of-custody during all field investigations for all samples collected. The recommended sample chain-of-custody procedures used by DEC BSPR employees or contractor are given in Appendix 5X.2.

5.3 DEFINITIONS

5.3.1 Grab Sample

A grab sample is an individual sample collected over a period of time generally not exceeding 15 minutes. A grab sample is normally associated with water or polluted water sampling. However, soil, sediment, liquid hazardous samples, etc., may also be considered grab samples. No particular time limit would apply for the collection of such samples.

1. Grab samples are:
 - Used to characterize the medium at a particular instant in time; and
 - Always associated with instantaneous water or polluted water flow data (where appropriate).
2. Grab sampling is conducted when:
 - The water or polluted water stream is not continuous (e.g., batch-discharges or intermittent flow);
 - The characteristic of the water or polluted stream are known to be constant or nearly so;
 - The sample is to be analyzed for parameters whose characteristics are likely to change significantly with time, i.e., dissolved gases, bacteria, etc.;
 - The sample is to be collected for analysis of a parameter such as oil and grease where the compositing process could significantly affect the actual concentration; and
 - Data on maximum/minimum concentrations are desired for a continuous water or polluted water stream.
3. Analyses for which samples of water should always be collected by the DEC BSPR personnel or contractor using the grab sample method or for which measurements shall be made in-situ include:

pH	phenol
temperature	oil and grease
dissolved oxygen	bacteria
sulfide	volatile organic compounds
chlorine residual	specific conductance
other dissolved gases	dissolved constituents in field filtered samples (total-P, ortho-P, metals, etc.)

5.3.2 Composite Sample

5.3.2.1 Timed Composite - A sample containing some discrete samples in equal volume collected at equal time intervals over the compositing period. (A timed composite may be collected continuously). Timed composites may be collected where water or polluted water flows vary widely and are not dampened by polluted water treatment units.

5.3.2.2 Flow Proportional Composites - A sample containing some discrete samples collected proportionally to the flow rate over the compositing period. Flow proportional samples may be collected where water or polluted water flows vary widely and are not dampened by polluted water treatment units.

5.3.2.3 Timed and Flow Proportional Composite Samples - The following guidance is given concerning the collection of composite samples:

1. Composite samples are:
 - collected when average concentrations are of interest; and are
 - always associated with average flow data (where appropriate).
2. Composite sampling is used when:
 - the water or polluted water stream is continuous;
 - it is necessary to calculate mass/unit time loadings; or when
 - analytical capabilities are limited.
3. A timed composite shall be collected as follows:
 - continuously; or
 - with constant sample volume at a constant time interval between samples.
4. A flow proportional composite shall be collected as follows:
 - continuously, proportional to stream flow;
 - with constant sample volume and with the time between samples proportional to stream flow; or
 - at a constant time interval between samples and with the sample volume proportional to flow at time of sampling.

5.3.2.4 Areal Composite - A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites shall be made up of equal volumes of grab samples. Each grab sample shall be collected in an identical manner. Examples include sediment composites made up of quarter-point grab samples from a stream, soil samples from grid points on a grid system, water samples collected at various depths at the same point or from quarter points in a stream, etc.

5.3.3 Split Sample

A split sample is a sample which has been portioned into two or more containers from a single sample container. Portioning assumes adequate mixing to assure the "split samples" are, for all practical purposes, identical.

5.3.4 Duplicate Samples

Duplicate samples are samples collected simultaneously from the same source under identical conditions into separate containers.

5.3.5 Reference or Control Sample

A sample collected upstream or upgradient from a source or site to isolate the effects of the source or site

on the particular ambient medium being sampled.

5.3.6 **Background Sample**

A sample collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

5.3.7 **Sample Aliquot**

A portion of a sample that is representative of the entire sample.

5.4 SPECIFIC SAMPLE COLLECTION QUALITY CONTROL PROCEDURES

5.4.1 General

This subsection provides guidelines for establishing quality control procedures for DEC BSPR sampling activities. Specific guidelines for sample site selection, selection of sampling equipment, types of samples to be collected, standard sample collection procedures, specific maintenance and calibration procedures for sampling equipment, and other considerations are presented for each medium later in this Section. Specific recommendations for all sampling activities are presented in Section 5.2. Adherence to the standard operating procedures outlined in this section form the basis for the DEC BSPR sampling quality assurance program.

5.4.2 Experience Requirements

There is no substitute for field experience. Therefore, all employees shall have field experience before they are permitted to select sampling sites. This field experience shall be gained by on-the-job training using the "buddy" system. Each new employee shall accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of qualified technical staff.

5.4.3 Traceability Requirements

All sample collection activities shall be traceable through field records to the person collecting the sample and to the specific piece of sampling equipment (where appropriate) used to collect that sample. All maintenance and calibration records for sampling equipment (where appropriate) shall be kept so that they are similarly traceable.

5.4.4 Measurement of Relative Sampling Precision

The following duplicate sampling procedures shall be used during the collection of samples as a relative measure of the precision of the sample collection process. Duplicate grab and composite samples shall be collected during all major investigations and studies conducted by the DEC BSPR. No more than ten percent of all samples shall be collected in duplicate. These duplicates shall be collected at the same time, using the same procedures, the same equipment, and in the same types of containers as the required samples. They shall also be preserved in the same manner and submitted for the same analyses as the required samples. The collection of duplicate composite samples shall require the installation of duplicate automatic sampler setups if automatic samplers are used for sample collection. Duplicate sample data shall be reported to the quality assurance officer or his designee.

These data will be periodically examined to determine if any problems are evident with specific types of media samples or with the procedures used by specific sample handling personnel. The Quality Assurance Officer will advise the appropriate Section Chiefs of any problems encountered so that corrective action can be taken.

5.4.5 Measurement of Sample Handling Effectiveness

The effectiveness of sampling handling personnel's sample handling techniques will be monitored by utilizing preserved and unpreserved field blank samples. These blank samples will be prepared by DEC BSPR personnel or proper suppliers. These blank samples will be handled as follows:

- Water Sample Organic Trip Blank - Two sealed preserved (or unpreserved if appropriate) VOA vials and one sealed container each for other organic compounds will be transported to the field. These samples will be handled and treated by sampling personnel in the same manner as the other samples collected for organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-of-Custody Records as trip blanks. These water sample trip

blanks will also be used to monitor the effectiveness of sample handling techniques where samples other than water, i.e., sludge, soil, sediment, etc. are collected. At least one set of trip blanks will be used on every study where samples for organic compounds analyses will be conducted.

- Metals and General Inorganic Preservation Blanks - Metals and general inorganic sample containers filled with blank water will be transported to the field and treated in the same manner as other like samples. These blank samples will be preserved and submitted for the same analyses as the other samples collected. These samples will be clearly identified as preservative blanks on sample tags and in the Chain-of-Custody Record(s). At least one preservative blank for each type of sample should be collected on non-routine field investigations. A minimum of one preservative blank should be prepared at the beginning and at the end of all major field investigations (20 samples or more). At least one preservative blank for each type of sample should be prepared once per quarter for routine inspections.
- Automatic Sampler Blanks - The procedure for collecting automatic sampler blanks is given separately at its own category of automatic sampling. In general, cleaning procedures outlined in Section 4.0 should be adequate to insure sample integrity. However, it is the standard practice of the DEC BSPR to submit automatic sampler blanks for analysis when automatic samplers are used to collect samples for organic compounds analyses. Automatic sampler blanks for other standard analyses shall be submitted at least once per quarter.

The DEC BSPR will advise the Quality Assurance Officer when trip blanks or preservative blanks are unacceptably contaminated. The Quality Assurance Officer will immediately initiate an investigation to determine the cause of the problem. The results of this investigation will be promptly reported to the appropriate Section Chiefs so that corrective action can be initiated.

5.4.6 Measurement of Sample Container, Sample Equipment, and Cleaning Procedure Integrity

Specific quality control procedures are outlined in Section 4.0.

5.4.7 Special Quality Control Procedures for Water Samples for Extractable, Pesticide, or Herbicide Organic Compounds Analyses

Sampling personnel shall submit duplicate water samples for extractable organic, pesticide, and/or herbicide organic compounds analyses from one sampling location per project. This sample should be collected from a location expected to be relatively free from contamination, since this sample will be used for laboratory quality control purposes. The duplicate sample should be clearly identified as "Duplicate Sample for Matrix Spike" on the sample tag, in the Chain-of-Custody Record, in the field logbook. This procedure shall be followed for all DEC BSPR projects where water samples are collected for the indicated organic compounds analyses, whether the samples are submitted to the contracted lab or other certified laboratory.

5.5 GROUNDWATER SAMPLING

5.5.1 General

Groundwater sampling may be required for a variety of reasons, such as examining potable or industrial water supplies, checking for and/or tracking contaminant plume movement in the vicinity of a land disposal or spill site, RCRA compliance monitoring, or examining a site where historical information is minimal or non-existent, but where it is thought groundwater contamination could have occurred.

Groundwater is usually sampled from a monitoring well, either temporarily or permanently installed. However, it can also be sampled anywhere groundwater is present, as in a pit or a dug or drilled hole.

Occasionally, a well will not be in the ideal location to obtain the sample needed. For example, a well may not be ideally located for tracking a contaminant plume. In that case, a well will have to be installed, and it may be either temporary or permanent. An experienced and knowledgeable person, preferably a hydrogeologist, will be needed to locate a place for the well and to supervise its installation so that the samples ultimately collected will be representative of the groundwater.

5.5.2 Site Selection

The relationship of the following factors to potential pollution sources shall be considered and evaluated when selecting groundwater sampling sites; the direction of groundwater flow; depth of groundwater; thickness of the aquifer (if applicable); type of stratigraphy; presence of perched water tables; types of soils; depth of bedrock; type of vegetation; surface drainage patterns; type of topography; and general land use. Surface features such as rock outcrops, seeps, springs, streams, rivers, and wet areas should also be considered (14). The area of interest should be located on an aerial photograph, a USGS 7.5 minute quadrangle map, a USDA soils map, and/or any other appropriate map that shows topography and general relationships between surface features. Aerial photographs can usually be obtained at the local Agricultural Stabilization Conservation Service Office or the local county tax office. USGS 7.5 minute quadrangle maps can be acquired from the State Department of Transportation or from the USGS, and soils maps from the USDA-SCS. A visual inspection of the area may be sufficient to evaluate and determine the surface conditions and their relationship to the subsurface conditions (14). In some cases, surface conditions and subsurface conditions cannot be correlated by site inspection or reconnaissance. When this occurs, a more detailed study, possibly involving test drilling, will have to be conducted.

It is extremely important to sample the unconfined or surficial aquifer downgradient of potential pollution sources or spills to determine if this aquifer has been affected. Generally the direction of groundwater flow can be estimated by two vectors - one in the direction of surface water flow (i.e., downstream) and another toward the surface water stream or river, if present. The relative magnitude of these vectors will vary according to site conditions and in some instances, both direction and magnitude may be changed by construction activities. If both shallow and deep aquifers are involved in the zone of interest, a screening study will reveal whether or not the deep aquifer needs to be sampled and/or if a more detailed study is required. To adequately assess subsurface conditions, a minimum of three wells are required, one in the upgradient portion of the area of interest, one in the middle portion, and one in the downgradient portion. In some cases, a more complex system of wells may be needed to define the subsurface conditions. Site conditions and the scope of the project will determine the total number of wells required. Existing wells should be used whenever possible. Where well installation is necessary, the wells should be installed according to the following procedures.

5.5.3 Monitoring Well Installation

Wells shall be dug, driven, drilled, or bored depending on the scope of work. Hand equipment such as augers, post hole diggers, picks, and shovels can be used to dig shallow wells in soft soils. Wells can be installed

by driving a piece of casing connected to a well point into the ground with an axe, sledge hammer, or mechanical or power device. Power equipment such as drilling rigs can be used to drill or auger wells in all types of soils and to any depth.

Where feasible the following procedures shall be used for well installation:

5.5.3.1 Temporary Monitoring Wells - For very shallow wells (15 feet) in soft material, hand augers or small portable power augers shall be used for boring. A temporary well casing with a well screen attached shall be installed as soon as the hole is augured.

For deeper temporary wells, larger well drilling equipment should be used with a hollow stem auger wherever possible. The use of a hollow stem auger prevents cave-in of the hole during drilling, allows for the collection of soil samples at various depths during the drilling operation, and may serve as a temporary casing for the well. In some cases the bottom of the auger will become plugged and not allow water to flow into the casing. When this happens, the auger should be pulled and the hole allowed to fill with water. A well casing can be installed if caving occurs.

When the water table is 25 to 30 feet deep, permanent well installation procedures should be considered.

5.5.3.2 Permanent Monitoring Well - Permanent monitoring wells shall be installed using hollow stem augers or continuous flight augers in soil and by standard rock coring procedures when drilling in rock. Where site conditions do not warrant the use of augers, alternate drilling methods such as straight rotary by using mud as a drilling fluid, or air rotary can be used. When muds are used in the drilling, a sample of the mud shall be collected for appropriate analyses. Casings with well screens shall be installed in unconsolidated and semi-consolidated soils to prevent soil and other foreign material from entering the well during pumping. Screens may or may not be required for wells in rock. The space surrounding the screen should be back filled with sand and filter pack and the remainder of the space surrounding the casing above the screen should be backfilled with natural clay, bentonite and/or cement bentonite grout, depending on the conditions at the site. Stainless steel well casings are preferred if samples are to be collected for organic compounds analyses. PVC casings are not acceptable where organic contaminants are a concern or under extremely corrosive conditions (18).

5.5.3.3 Monitoring Well Security - Wells should be capped and locked at the conclusion of installation or sampling for future use, or if the well will not be used in the future, it should be properly abandoned by backfilling with suitable permanent fill materials such as concrete, bentonite, compatible soil, or a combination of these materials (15).

5.5.4 Monitoring Well Development

All permanently installed wells shall be adequately developed prior to initial sampling efforts (14). Adequate development should eliminate all fine material from the area of the well screen and allow for the collection of a sample which is free of suspended materials. Wells installed by "wet drilling" where drilling muds are used shall be developed so that residual drilling muds will not settle around the well screens or in the surrounding soil and contaminate future sampling.

Various methods may be used to develop wells. These methods consist of suction lift pumping (pitcher, centrifugal, roller, piston, peristaltic pumps), pressure ejection pumping, submersible pumping, surge blocks, bailing, and air lift pumping (15, 16, 17).

5.5.5 Purging Equipment and Techniques

5.5.5.1 General - Wells shall be purged before taking samples in order to clear the well of stagnant water which has been standing in the well casing and may not be representative of aquifer conditions. One method of

purging is to pump the well until three to five times the volume of standing water in the well has been removed. A second method is to pump the well until the specific conductance, temperature and pH of the groundwater stabilizes. Normally, a combination of the two methods is employed (i.e., specific conductance, temperature, and pH are measured at intervals and a record of the volume purged is monitored). If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery (15, 17).

5.5.5.2 Equipment Available - Monitoring well purging is accomplished by using in-place plumbing/pumps or when in-place pumps are not available, by using a proper equipment, such as peristaltic, turbine, bladder, centrifugal, or other appropriate pump, depending on well depth. A Teflon, closed top bailer may be used for purging; however, bailing stirs up sediment in the well and tends to increase turbidity. Thus pumping is preferred.

Other monitoring equipment used during purging includes water level indicators, pH meters, thermometers, and conductivity bridges.

5.5.5.3 Purging Techniques (Wells Without Plumbing or in Place Pumps)

5.5.5.3.1 General - For permanently installed wells, the depth of water shall be determined (if possible) before purging. This can be accomplished by attaching a weight on the end of tape and lowering it into the well until it touches the water, or by use of a mechanical or electrical water level indicator. Operating personnel shall exercise extreme caution during this procedure to prevent contamination of the well. This is a critical concern when samples for trace organic compounds or metals analyses are collected.

5.5.5.3.2 Using Pumps to Purge - When suction lift or centrifugal pumps are used, only the intake line is placed into the water column. To minimize contamination, the line placed into the water is either standard cleaned Teflon (see Section 4.0), in the case of the suction lift pumps, or standard cleaned stainless steel pipe attached to a hose, when centrifugal pumps are used.

When submersible pumps (bladder, turbine, displacement, etc.) are used, the pump itself is lowered into the water column.

5.5.5.3.3 Using Bailers to Purge - Standard cleaned (Section 4.0) closed-top bailers with nylon rope are lowered into the top of the water column, allowed to fill and be removed, and the water is discarded.

Cleaning and decontamination materials are specifically mentioned as having to be collected and disposed of properly in Section 4.1.6. Purge water should be held in drums, labeled for each well, then disposed of properly in accordance with water quality laboratory results.

5.5.5.3.4 Field Care of Purging Equipment - Regardless of which method is used for purging, new aluminum foil or plastic sheeting shall be placed on the ground surface around the well casing. This is done to prevent contamination of the pumps, hoses, ropes, etc. in the event they need to be placed on the ground during the purging or accidentally come into contact with the ground surface.

It is preferable that hoses used in purging that come into contact with the groundwater be kept on a spool, both during transporting and during field use, to further minimize contamination from the transportation vehicle or ground surface.

5.5.5.3.5 Purging Entire Water Column - The pump/hose assembly or bailer used in purging should be lowered into the top of the standing water column and not deep into the column. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump was placed deep into the water column, the water above the pump may not be removed, and the subsequent samples collected may not be representative of the groundwater.

To minimize cross contamination between wells, no more than three to five feet of hose should be lowered into the water column. If the recovery of the well is at least as fast as the pump rate, the pump may be left hanging at the initial level until an adequate volume has been purged. If the pump rate exceeds the recovery rate of the well, the pump will have to be lowered, as needed, to accommodate the draw-down.

After the pump is removed from the well, all wetted portions of the hose and the pump shall be cleaned as outlined in Section 4.0.

Careful consideration shall be given to using pumps to purge wells which are excessively contaminated with oily compounds, because it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells such as this are encountered, alternative purging methods, such as bailers, should be considered.

5.5.5.4 Purging Techniques - Wells With in-Place Plumbing

5.5.5.4.1 General - In-place plumbing is found at water treatment plants, industrial water supply wells, private residences, etc. The objective of purging is the same as with monitoring wells without in-place pumps -- to ultimately collect a sample representative of the groundwater.

The volume to be purged depends on several factors: whether the pumps are running continuously or intermittently, how close to the source the sample can be collected, and the presence of any storage/pressure tanks between the sampling point and the pump. If storage/pressure tanks are present, an adequate volume must be purged to totally exchange the volume of water in the tank.

5.5.5.4.2 Continuously Running Pumps - If the pump runs continuously, and the sample can be collected prior to a storage/pressure tank, no purge, other than opening a valve and allowing it to flush for a few minutes, is necessary.

If the pump runs continuously, and a storage/pressure tank is located ahead of the sample location, the purge must include the entire storage volume to be sure that a sample representative of the groundwater will be collected.

5.5.5.4.3 Intermittently Running Pumps - If the pump runs intermittently, it is necessary to determine the volume to be purged, including storage/pressure tanks that are located ahead of the sampling location.

The pump should then be run continuously until the required volume has been purged.

5.5.6 Sampling Equipment and Techniques

5.5.6.1 Equipment Available - Sampling equipment which can be used includes closed-top bailers and the peristaltic pump/vacuum jug assembly.

Other monitoring equipment used during sampling includes water level indicators, pH meters, thermometers, and conductivity bridges.

5.5.6.2 Sampling Techniques - Wells With in Place Plumbing - Following purging, samples should be collected from a valve or cold water tap as near to the well as possible. Samples should be collected directly into the appropriate containers (see recommended Sample Containers, Appendix 5X.1). Also, refer to the Potable Water Supply discussion in Section 5.6.

5.5.6.3 Sampling Techniques - Wells Without Plumbing - Following purging, samples should be collected using a peristaltic pump - vacuum jug procedure, if possible, or with a closed top bailer. The pump used for purging

generally should not be used for sampling. When the peristaltic pump is used, samples for purgeable organic compounds should be collected using a bailer or by allowing the Teflon tube to fill and then allowing the water to drain into the sample vials. All equipment shall be cleaned using the procedures described in Section 4.0. Also, refer to the Potable Water Supply discussion, Section 5.6, for additional information.

When bailing, new foil or plastic sheeting should be placed on the ground around each well to prevent contamination of sampling equipment in the event any equipment is dropped or otherwise comes in contact with the ground. Braided nylon cord may be used to haul the bailer if the nylon cord is used only one time and then discarded. Teflon coated wire, single strand stainless steel wire, or monofilament are acceptable for hauling bailers and may be decontaminated for reuse.

5.5.7 Special Sample Collection Procedures

5.5.7.1 Trace Organic Compounds and Metals - Special sample handling procedures shall be instituted when trace organic compounds and metals samples are being collected. All sampling equipment, including pumps, bailers, drilling equipment, water level measurement equipment, etc., which come into contact with the water in the well must be cleaned in accordance with the cleaning procedures described in Section 4.0. Synthetic drilling mud (i.e., Revert) should not be used when constructing wells which will be used for trace organic compounds and metals sampling. Pumps shall not be used for sampling, unless the interior and exterior portions of the pump and discharge hoses can be thoroughly cleaned. Blanks should be collected to determine the adequacy of cleaning prior to collection of any sample using a pump. Peristaltic pumps using Teflon tubing and a Teflon insert can be used to collect samples without the sample coming into contact with the pump. This is accomplished by placing the Teflon insert into the opening at a standard cleaned gallon glass container (Section 4.0). The Teflon tubing connects the container to the pump and sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without coming into contact with the pump tubing. Samples for purgeable organic compounds analyses shall be collected with well bailers or by allowing the Teflon tube to fill and then allowing the water to drain into sample collection vials. The procedures given in the General Considerations, Special Precautions for Trace Contaminant Sampling (Section 5.2) shall be followed.

5.5.7.2 Filtering - As a standard DEC BSPR policy, groundwater samples should not be filtered in the field. However, if required by RCRA regulations, then both filtered and non-filtered samples will be submitted for analyses. Proper well installation and development as well as proper well purging technique should minimize the turbidity of samples. Whenever extremely high concentrations of sediment are present in a well sample, the aliquot of the sample for metals analyses should not be preserved without first being filtered. Whenever samples for dissolved metals analyses are collected, an additional sample, unfiltered and acid preserved will also be collected for total metals analyses. Samples for analyses of organic compounds shall never be filtered. When samples are being filtered in the field, the filter apparatus must be cleaned (Section 4.0) and samples must be filtered properly.

5.5.7.3 Bacterial Sampling - Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in Sampling for Organic Chemicals and Microorganisms in the Subsurface (19) as well as References 4 and 5.

5.5.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect groundwater samples shall be cleaned as outlined in Section 4.0 and repaired, if necessary, before being stored at the conclusion of field studies.

All equipment shall be tested before being issued for field studies.

Cleaning procedures conducted in the field (Section 4.0) or field repairs shall be thoroughly documented in field records.

5.5.9 Auxiliary Data Collection

Water table measurements from the top of the well casings (referenced to National Geodetic Vertical Datum) in permanent wells, and ground surface elevation in temporary wells should be made to determine the general direction of groundwater flow and gradient. Traced dyes and thermal detection methods can be used to determine direction and velocities of flow (14). Also, a study of the general topography and drainage patterns will generally indicate direction of groundwater flow.

Water table measurements shall not be taken until the water table has stabilized, preferably 24 hours after well installation for permanent wells (20). The ground surface elevation at the wells should be determined by standard engineering survey practices.

In addition to water level measurements, the pumping rate used to purge a well, the volume of water in wells, and drillers logs are examples of auxiliary data that should be collected during groundwater sampling activities. This information should be documented in field records. Methodology for obtaining these data are given in the following sections.

Temperature, specific conductance, and pH shall be measured each time a well is sampled. This information is generally obtained during the purging process to evaluate the adequacy of the purging procedure. In this situation, the final measurements for these parameters prior to sampling shall be considered the measurement of record for the well. If these parameters were not evaluated during purging, they shall be obtained prior to sampling.

5.5.9.1 Well Pumping Rate - Bucket/Stop Watch Method - The pumping rate of a pump can be determined by collecting the flow of water from the pump in a bucket of known volume and timing how long it takes to fill the bucket. The results should be flow rate in gallons per minute. This method shall be used only with pumps with a constant pump rate, such as gasoline powered or electric submersible pumps. It should not be used with battery powered pumps. As the batteries lose their charge, the pump rate decreases so that pumping time calculations using initial, high pump rates are erroneously short.

5.5.9.2 Volume of Water in Wells - In order to purge wells, the volume of water in the well should be known. To determine the volume, the following method should be used; measure the distance from the bottom of the well to the static water level, then measure the inside diameter of the well or casing. Obtain the volume of the well by the formula:

$$V = 0.041 d^2 h \quad \text{Where:} \quad \begin{array}{l} h = \text{depth of water in feet} \\ d = \text{diameter of well in inches} \\ V = \text{volume of water in gallons} \end{array}$$

If preferred, a quick reference nomograph or table may be used.

5.5.9.3 Driller's Log - It is imperative that drilling logs be concise, complete, and described in a manner that is easily understood to all who read them. The following items shall be included in the logging data:

- hole number and location;
- description of soils and subsurface conditions (if applicable);
- type of drilling equipment, driller, and drilling company (if applicable);
- method of drilling;
- type and size of casing;
- type and size of well screen;
- depth to well screen;
- type of pump and pumping rate;
- drilling and sampling times;

- depth to water table, and date and time measured;
- types of samples taken and depths at which the sample was taken;
- volume of water purged;
- type of well (permanent or temporary);
- type of sampling equipment and/or cleaning procedure; and
- depth of sampling and description (if applicable).

Additional groundwater related data can be obtained from most local, state, and federal agencies dealing with water resources. Some sites require well drillers to be licensed, and all work performed on wells must be reported to the state on prescribed forms. These forms are available to the public, so a study of wells installed in the area of interest will provide background information as to the subsurface conditions. State geological surveys, as well as the USGS, have various types of water related papers and reports on all phases of groundwater studies in each state. City and county governments usually have departments that deal with water related projects that may provide data for the local area. Federal agencies such as the U.S. Army Corps of Engineers, the Bureau of Reclamation, U.S. Forest Service, Science and Education Administration, and the U.S. Public Health Service have water programs and may provide data. Other sources include the Bureau of Mines, colleges, universities, and technical societies such as the American Association of Petroleum Geologists, American Institute of Mining and Metallurgical Engineers, National Water Well Association, Association of Engineering Geologists, and Geological Society of America (14,21).

5.6 SAMPLING OF POTABLE WATER SUPPLIES

5.6.1 General

When sampling potable water supplies, utmost care must be taken to insure that samples are representative of the water supply being sampled. This is important not only from a technical and public health perspective, but also from a public relations standpoint. Poor sampling techniques may result in incorrect results (either not detecting a compound which is present or by contaminating the sample and falsely indicating a compound which is not present). If incorrect results are disclosed to the public, it may be very difficult to change public opinion when correct results are reported.

5.6.2 Sampling Site Selection/Sampling Techniques

Even though the same care and techniques used in groundwater, etc., sampling (including thorough documentation of location, date, time, etc.) are used by sampling personnel in potable water supply sampling, there are certain additional special procedures which shall be used.

When water samples are collected from wells, either by mechanical or hand pumping, the wells must be purged before the sample is collected (see Section 5.5 for groundwater sampling methods). This procedure insures that water in the well field is sampled, not the standing water in the pump or holding tank. As a rule of thumb, at least one volume of water in the well casing and storage tank should be evacuated (see Section 5.5.5.4 for more details). This also insures that any contaminants that might have entered the area of the tap from external sources are flushed away (19).

Potable water samples shall be representative of the water quality within a given amount of the distribution network. Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest and should not be separated from the segment of interest by a storage tank. The sampling tap must be protected from exterior contamination associated with being too close to the sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collecting procedure since it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. Leaking taps that allow water to flow out from around the stem of the valve 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. Aerator, strainer, and hose attachments on the tap must be removed before sampling. These devices can harbor a bacterial population if they are not cleaned routinely or replaced when worn or cracked. Whenever a steady stream of water cannot be obtained from taps, after such devices are removed, a more suitable tap shall be sought. Taps where the water flow is not steady should be avoided because temporary fluctuation in line pressure may cause sheets of microbial growth that are lodged in some pipe section or faucet connection to break loose and contaminate the sample. The cold water tap should be opened for two or three minutes or for sufficient time to permit clearing of the service line. A smooth-flowing water stream at moderate pressure without splashing should be obtained. Then, without changing the water flow which could dislodge some particles in the faucet, the samples can be collected (19).

Regardless of the type of sample bottle being used, the bottle cap should not be placed on the ground or in a pocket. Instead, hold the bottle in one hand and the cap in the other, keeping the bottle cap right side up (threads down) and using care not to touch the inside of the cap. Exercise care not to lose the Teflon liner in certain bottle caps. Avoid contaminating the sample bottle with fingers or permitting the faucet to touch the inside of the bottle. When sampling for bacterial content, the bottle should not be rinsed before use. This may not only contaminate the bottle, but also remove the thiosulfate dechlorinating agent (if used). When filling any container, care should be taken so splashed drops of water from the ground or sink do not enter into either the bottle or cap. In order to avoid dislodging particles in the pipe or valve, do not adjust the stream flow while sampling.

When sampling at a water treatment plant, samples should be collected both from the raw water supply and after chlorination.

Duplicate samples will always be collected for VOA and bacterial analyses. Single samples may be collected for extractable organic compounds, metals, phenol, cyanide, and conventional parameter analyses. The procedures given in Section 5.2.9 (Special Precautions for Trace Contamination Sampling) and in the Section 5.6.2.1 below (Purgeable Organic Compounds Sample Collection) shall always be followed when potable water supplies are sampled.

DEC BSPR or contractor shall always obtain the name(s) of the resident or water supply owner/operator and the resident's exact mailing address, as well as the resident's home and work telephone numbers. The information is required so that the residents or water supply owner/operators can be informed of the results of the sampling program.

5.6.2.1 Purgeable Organic Compounds Analyses (VOA) - Samples to be analyzed for purgeable organic compounds should be stored in 40 ml septum vials with screw caps that have a Teflon lined silicone disk in the cap to prevent contamination and loss of the sample through the cap. The disks should be placed in the caps (Teflon in contact with the sample) in the laboratory prior to the beginning of the sampling program.

When sampling for purgeable organic compounds, duplicate samples should always be collected from each location. The investigator should determine if the water to be sampled contains chlorine. If the water contains no chlorine, two 40-ml vials containing four drops of concentrated HCl should be filled with the sample and labeled PA (preserved acid). If the sample contains no chlorine and only if it will be analyzed within 24 hours, the HCl preservation is not necessary. If the water contains chlorine, the following sampling and preservation procedure should be followed:

- Fill a 4-ounce (120 ml) soil VOA sampling container containing 0.008 percent sodium thiosulfate with the water sample. Cap and mix thoroughly but gently by swirling to eliminate residual chlorine. Transfer the sample to two 40-ml VOA vials containing four drops of concentrated HCl*. Label 40-ml vials - PTA (preserved/sodium thiosulfate/acid).

* The sodium thiosulfate preservatives must be added in this order and in two separate steps because HCl reacts with sodium thiosulfate.

The purgeable organics vials (40-ml) should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus". The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles; if any are present, repeat the procedure.

Sampling and preservation containers should be pre-labeled (i.e., PA, PT, or PTA) prior to any field activities. This will reduce the chances of confusion during sampling activities by the investigation team.

5.6.3 Sampling Equipment/Specific Sampling Equipment Quality Assurance Techniques

Sampling equipment and specific equipment quality assurance techniques are contained in Section 5.5 (Groundwater Sampling).

5.7 SOIL SAMPLING

5.7.1 General

The objective of this section is to give general guidance for the collection of soil samples during field investigations. Guidance for preparing soil sampling protocols, including statistical sampling methodology are included in the EPA publication "Preparation of Soil Sampling Protocol, Techniques and Strategies" (22).

5.7.2 Sampling Location/Site Selection

Any site selected for soil sampling shall be strategically located in order to collect a representative fraction of the soils with the minimum number of samples and amount of effort. A surface inspection of the subject area shall be made to locate pertinent features (e.g., rock outcrops, drainage patterns, surface runoff, ponds, lakes, wet areas, seeps, springs, permanent structures, fill areas, erosional areas, depositional areas, etc.) and to evaluate the relationship between these features and potential sources of pollution. The location of sediment depositional areas are good indicators of surface runoff direction. If the direction of surface runoff or drainage is difficult to detect, observation of new depositions or sediment movement following a rain may prove helpful in establishing this direction. The spreading or fanning of the sediment body will indicate direction of flow.

In most instances, the first investigation of a site will be a reconnaissance type survey. Soil sampling in these instances will generally be confined to surface or near-surface soils and/or sediments with hand equipment. For screening purposes, sampling of this type should be conducted in depositional areas on the periphery of the study area, primarily at the downstream or downgradient portion(s) of the area of interest; however, an upgradient location should also be selected for obtaining background and/or control samples. Investigators should be aware that sampling in depositional areas tends to bias the sampling toward elevated concentrations, which is useful as a screening tool, but should not be construed as representative of the area conditions.

More in-depth investigations are usually conducted after a preliminary study or reconnaissance survey has been completed. Review of previous investigations will aid in selection of suitable sampling locations, and these studies should be examined when the study plan for the more detailed study is prepared. The number of samples and the number of test pits and/or borings and the specific depth that samples are collected will vary according to the site conditions and the scope of the investigation. A determination of soil sample number and location based on statistical concept is discussed in Section 6B.1.

5.7.3 Equipment Available

The following equipment is available for field use in soil sampling: stainless steel spoons; stainless steel hand augers; stainless steel shovels; Shelby tubes; portable power augers (Little Beaver); stainless steel scoops; glass pans; and drill rigs and associated equipment (i.e., split spoon samplers), which may, on occasion, be rented or borrowed for special projects.

5.7.4 Sampling Techniques

5.7.4.1 General - Sampling is often conducted in areas where a vegetative turf has been established. In these cases a clean stainless steel shovel should be used to carefully remove the turf so that it may be replaced at the conclusion of sampling. When the soil sample is obtained, it should be deposited into a glass pan for mixing (or compositing, if applicable) prior to filling in the sample containers. Mixing of the soil sample for non-volatile or non-semi volatile chemical analysis should be performed in accordance with the procedures outlined in Section 5.7.4.5. If an undisturbed sample is needed, the Shelby tube sampler may be used as described in Section 5.7.4.2.

If practical, and at the project leader's discretion, all trenches or holes that were excavated for sampling

should be filled in and the turf replaced.

5.7.4.2 Surface Soil Sampling - Prior to sampling, leaves, grass, and surface debris should be removed from the area to be sampled using a clean stainless steel spoon or shovel. Surface soil samples shall then be collected using a precleaned, stainless steel scoop or spoon.

5.7.4.3 Shallow Subsurface Soil Sampling - Shallow subsurface soil samples may be collected by digging a hole or trench with a stainless steel shovel, then removing all of the loose soil and collecting a sample at the desired depth using a stainless steel spoon, a stainless steel hand auger, or a Shelby tube.

The stainless steel hand auger consists of three basic parts: (1) the bucket, (2) extension, and (3) handle. At the bottom end of the bucket are two cutting edges. The extensions are three feet long. When sampling deeper subsurface soil (Section 5.7.4.4), a number of extensions may be joined end to end to increase the depth from which soil may be sampled.

The Shelby tube is a stainless steel tube approximately 12 inches long and 2 inches in diameter. One end of the tube has the edges beveled into a cutting edge. The other end can be mounted on an adapter which allows attachment to the end of the hand auger. The Shelby tube is pushed into the soil to be sampled and then removed. The tube can then be removed from the adapter and the soil pushed out using a decontaminated piece of equipment such as the handle of a stainless steel spoon. If an undisturbed sample is required, the Shelby tube with its sample intact may be shipped directly to the laboratory for analyses.

5.7.4.4 Deeper Subsurface Soil Sampling - For deeper sampling using hand equipment, a stainless steel auger (see Section 5.7.4.3) is used to bore a sampling hole until the desired depth is reached. Another clean auger bucket or a Shelby tube is then used to collect the sample which is placed in a glass pan as described in Section 5.7.4.1. Surface debris should be removed from the location of the sampling hole using a clean, stainless steel shovel or spoon before auguring operations are initiated.

Often the depth which can be reached using a hand auger is limited due to the soil having low cohesion which leads to the hole collapsing or to the soil being very tightly packed, which can make turning and removing the auger difficult. In cases such as these, a portable power auger Little Beaver may be used to reach the desired depth. The sample can then be collected as described in the previous paragraph. The portable power auger consists of a powered drive unit (hand-held) used by sampling personnel to drive crew-like auger flights. The auger flights should be cleaned using the same procedures as for the other soil sampling equipment (Section 4.0). For safety reasons, the Little Beaver portable power auger should never be used with less than two sampling personnel present.

The split spoon sampler may be used for sampling at greater depths. Because of its weight, the split spoon sampler is generally used with power equipment, i.e., drilling rig. A hollow stem auger is used to advance the hole to the desired depth. The split spoon is added to the correct length of drill rod and forced into the undisturbed soil by means of a 140-pound weight or hammer. The split spoon is retrieved from the hole and opened to reveal the sample. The top two or three inches of the sample normally will be disturbed and should be discarded. The undisturbed portion should be placed in a glass pan by means of a clean, stainless steel spoon or spatula. The procedure is repeated until the desired amount of sample is collected. The sample should then be thoroughly mixed.

5.7.4.5 Mixing - Regardless of the method of collection, soil samples collected for non-volatile chemical analyses should be thoroughly mixed before being placed in the appropriate sample containers. The soil should be removed from the sampling device (dredge, core tube, scoop, etc.) and placed in a glass or Teflon coated stainless steel pan. The soil in the pan should be scraped from the sides, corners, and bottom of the pan, rolled to the middle of the pan, and initially mixed. The sample should then be quartered and moved to the four corners of the container. Each quarter of the sample should be mixed individually. Each quarter is then rolled to the center of the container and the entire sample is mixed again.

This procedure should be continued to ensure that all parts of the sample are mixed and that the sample is as homogenous as possible before being placed in the sample containers.

5.7.5 Special Precautions for Trace Contaminant Soil Sampling

The procedures outlined in Section 5.2.11 shall be followed. All soil sampling equipment used for sampling of trace contaminants should be constructed of stainless steel whenever possible. In no case will chromium, cadmium, or galvanized plated or coated equipment be used for soil sampling operations. Similarly, no painted equipment shall be used. All paint and primer must be removed from soil sampling equipment by sandblasting or other means before such equipment can be used for collecting soil samples.

5.7.6 Soil Samples Collected for Purgeable Organic Compounds Analyses (VOA)

When soil samples collected for purgeable organic compounds analyses need to be mixed in field, they should be done properly and placed in containers as soon as possible after sampling. The sample should be placed in the sample container so that no head space is left in the container after the container is closed.

5.7.7 Specific Sampling Equipment Quality Assurance Techniques

Drilling rigs and other major equipment used to collect soil samples shall be identified so that they can be traced through field records. A log book shall be established for this equipment so that all cleaning, maintenance and repair procedures can be traced to the person performing these procedures and to the specific repairs made. Sampling spoons, hand augers, Shelby tubes, and other minor disposable type equipment are exempted from this equipment identification requirement.

All equipment used to collect soil samples shall be cleaned as outlined in Section 4.0 and repaired, if necessary, before being stored at the conclusion of field studies.

Any cleaning conducted in the field (Section 4.0) or field repairs should be thoroughly documented in field records.

5.7.8 Auxiliary Data Collection

In addition to information pertaining to an area of specific site/location that may be available in DEC BSPR files from previous investigations (i.e., site screenings, water quality, well monitoring studies, etc.) information and data may be obtained from various city, county, federal, and other state agencies.

A system of logging all pertinent data collected during drilling and sampling operations should be maintained. The test hole locations should be recorded and referenced to the site map and/or datum base so that each location can be permanently established. Samples should be accurately tagged and labeled with all pertinent site information at the time of sampling.

APPENDIX 5X.1
RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, MAXIMUM SAMPLE HOLDING TIMES, AND
PERMISSIBLE SAMPLE TYPES

<u>Parameter</u>	<u>Container</u>	<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
<u>Concentrated Waste Samples</u>					
Organic Compounds	8-oz. widemouth glass with Teflon liner	None	7 days	G or C	A
Metals and Other Inorganic Compounds	8-oz. widemouth glass with Teflon liner	None	7 days	G or C	A
EP Toxicity	8-oz. widemouth glass with Teflon liner	None	7 days	G or C	B
Flash Point and/or Heat Content	8-oz. widemouth glass with Teflon liner	None	ASAP - NS	G	B
<u>Fish Samples</u>					
Organic Compounds	Wrap in aluminum foil	Freeze	ASAP	G or C	A
Metals and Other Inorganic Compounds	Place in plastic ziplock bag	Freeze	ASAP	G or C	A
<u>Liquid - Low to Medium Concentration Samples</u>					
Alkalinity	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	12-14 days	G or C	C
Acidity	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	12-14 days	G or C	C
Bacteriological Tests (coliform)	250-ml glass with glass closure or plastic capable of being autoclaved	Cool, 4°C	6 hrs.	G	C

Parameter Container

Liquid - Low to Medium Concentration Samples (Continued)

		<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
Static Bioassay	1-gal. amber glass (not solvent rinsed)	Cool, 4° C	48 hrs.	G or C	D
Biochemical Oxygen Demand (BOD)	1/2-gal. polyethylene ¹ with polyethylene closure	Cool, 4° C	48 hrs.	G or C	C
Chloride	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	None	28 days	G or C	C
Chlorine Residual	In-situ, beaker or bucket	None	Analyze Immediately	G	C
Color	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4° C	48 hrs.	G or C	C
Conductivity	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4° C	28 days (determine on site if possible)	G or C	C
Chromium, Hexavalent	1-liter polyethylene with polyethylene closure	Cool, 4° C	24 hrs.	G	C
Cyanide	1-liter or 1/2-gallon polyethylene with polyethylene or polyethylene lined closure	Ascorbic Acid ^{2,3} Sodium Hydroxide, pH > 12, Cool, 4° C.	14 days	G	C
Dissolved Oxygen (Probe)	In-situ, beaker or bucket	None	Determine On Site	G	C
Dissolved Oxygen (Winkler)	300-ml glass, BOD bottle	Fix on site, store in dark	8 hrs. (determine on site if possible)	G	C

Parameter Container

Liquid - Low to Medium Concentration Samples (Continued)

<u>Parameter</u>	<u>Container</u>	<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
EP Toxicity	1-gal. glass (amber) with Teflon liner	Cool, 4°C	7 days till extraction	G or C	B
Fluoride	1-liter polyethylene or 1/2-gal. polyethylene with polyethylene or polyethylene lined closure	None	28 days	G or C	C
Hardness	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	50% Nitric ² Acid, pH <2	6 months	G or C	C
LAS	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	C
Metals (Total)	1-liter polyethylene with polyethylene lined closure	50% Nitric ² Acid, pH <2	6 months (except Hg, Cr ⁺⁺⁶)	G or C	C
Metals, Dissolved	1-liter polyethylene with polyethylene lined closure	Filter-on-site ² 50% Nitric Acid, pH <2	6 months (except Hg, Cr ⁺⁺⁶)	G	C
Nutrients ⁴ (NH ₃ , COD, TOC, nitrate-nitrite, etc.)	1-liter polyethylene or 1/2-gal. polyethylene with polyethylene or polyethylene lined closure	50% Sulfuric ² Acid, pH <2 Cool, 4°C	28 days	G or C	C
Oil and grease	1-liter widemouth glass with Teflon liner	50% Sulfuric ² Acid, pH <2 Cool, 4°C	28 days	G	C

Parameter
Liquid - Low to Medium Concentration Samples (Continued)
Container

	<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
Organic Compounds -- Extractable and Pesticide Scan				C
No Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon liner Cool, 4° C	5-7 days ³	G or C	C
Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon liner Add 3 ml 10% sodium thiosulfate per gallon Cool, 4° C	5-7 days ³	G or C	C
Organic Compounds - Purgeable (VOA)				
No Residual Chlorine Present	2 40-ml vials with Teflon lined septum caps 4 drops conc. hydrochloric acid, Cool, 4° C	14 days	G	C
No Residual Chlorine Present	2 40-ml vials with Teflon lined septum caps Cool, 4° C	7 days	G	C
Residual Chlorine Present	2 40-ml vials with Teflon lined septum caps Footnote 6	14 days	G	C
Organic Compounds - Specified and Pesticides (Non-Priority Pollutants such as Herbicides)	1-gal. galss (amber) or 2 1/2-gal. glass (amber) with Teflon lined closure Footnote 7	7 days ⁷	G or C	C
Organic Halides - Total (TOX)	250-ml amber glass with Teflon lined septum closure Cool, 4° C	7 days	G	E
pH	In-situ, beaker or bucket None	Analyze Immediately	G	C
Phenols	1-liter amber glass with Teflon lined closure 50% Sulfuric Acid, pH <2 Cool, 4° C	28 days	G	C

<u>Parameter</u>	<u>Container</u>	<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
<u>Liquid - Low to Medium Concentration Samples (Continued)</u>					
Phosphate-Ortho	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site Cool, 4°C	48 hrs.	G	C
Phosphorus, Total Dissolved	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site 50% Sulfuric Acid, pH < 2, Cool, 4°C	28 days	G	C
Solids, Settleable	1/2-gal. polyethylene with polyethylene closure	Cool, 4°C	48 hrs.	G or C	C
Solids (Total and Suspended, etc.)	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	7 days	G or C	C
Sulfates	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	28 days	G or C	C
Sulfides	500-ml or 1-liter poly- ² ethylene with polyethylene or polyethylene lined closure	2 ml Zinc Acetate ² Conc. Sodium Hydroxide to pH > 9, Cool, 4°C	7 days	G	C
Temperature	In-situ, beaker or bucket	None	Determine On Site	C	C
Turbidity	500-ml or 1-liter poly- ¹ ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	C

<u>Parameter</u>	<u>Container</u>	<u>Preservative</u>	<u>Maximum Holding Time</u>	<u>Permissible Sample Type</u>	<u>Reference</u>
<u>Soil, Sediment or Sludge Samples - Low to Medium Concentration</u>					
E.P. Toxicity	8-oz. widemouth glass with Teflon lined closure	Cool, 4°C	7 days till extraction	G or C	B
Metals	8-oz. widemouth glass with Teflon lined closure	Cool, 4°C	6 months	G or C	A
Nutrients Including: Nitrogen, Phosphorus, Chemical Oxygen Demand	500-ml polyethylene with polyethylene closure or 8 oz. widemouth glass with Teflon lined closure	Cool, 4°C	ASAP	G or C	A
Organics - Extractable	8-oz. widemouth glass with Teflon liner	Cool, 4°C	ASAP	G or C	A
Organics - Purgeable (VOA)	4-oz. (120 ml) widemouth glass with Teflon liner	Cool, 4°C	7 days	G or C	A
Other Inorganic Compounds - Including Cyanide	500-ml polyethylene with polyethylene closure or 8-Oz. widemouth glass with Teflon lined closure	Cool, 4°C	7 days	G or C	A
<u>Radiological Tests</u>					
Alpha, beta, gamma	Polyethylene container with polyethylene closure	HNO ₃ to pH < 2	6 months	G or C	

Abbreviation: G = Grab
 C = Composite
 ASAP = As Soon As Possible
 NS = Not Specified

Footnotes:

1. Use indicated container for single parameter requests, 1/2-gallon polyethylene container for multiple parameter requests except those including BOD, or 1-gallon polyethylene container for multiple parameter request which include BOD.
2. Must be preserved in the field at time of collection. For dissolved metals, the sample must be filtered through a 0.45 membrane filter immediately after collection and prior to preservation in the field.
3. Use ascorbic acid only if the sample contains residual chlorine. Test a drop of sample with potassium iodide-starch test paper; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.
4. May include nitrogen series (ammonia, total Kjeldahl nitrogen, nitrate-nitrite), total phosphorus, chemical oxygen demand and total organic carbon.
5. Samples must be extracted within seven days and extract must be analyzed within 40 days.
6. Collect the sample in a 4 oz. soil VOA container which has been pre-preserved with four drops of 10 percent sodium thiosulfate solution. Gently mix the sample and transfer to a 40 ml VOA vial that has been pre-preserved with four drops concentrated HCl, cool to 4°C.
7. See Organic Compounds - Extractable (the 4th page of this appendix). The Analytical Laboratory should be consulted for any special organic compound analyses in order to check on special preservation requirements and or extra sample volume.

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- D. US-EPA, Region IV, Environmental Services Division, "Ecological Support Branch, Standard Operating Procedures Manual," latest version.
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APPENDIX 5X.2 -- CHAIN-OF-CUSTODY, DOCUMENT CONTROL, AND STANDARD OPERATING PROCEDURES

The objectives of this section are to present the laboratory proper operating procedures for sample identification, sample control, chain-of-custody, maintenance of records, and document control.

The laboratory should not deviate from the procedures described herein without the written approval of the Bureau of Spill Prevention and Response, or without a DEC approved equivalent.

1. Sample Chain-of-Custody

A sample is physical evidence collected from a facility or from the environment. An essential part of hazardous chemical or material investigations is that samples and data may be used as evidence in NYSDEC enforcement proceedings. To satisfy enforcement uses of the data, the following chain-of-custody procedures have been established.

1.1 Sample Identification

To ensure traceability of samples while in possession of the laboratory, a method for sample identification shall be developed and documented in laboratory Standard Operating Procedures (SOPs) (see Section 3). Each sample or sample preparation container shall be labeled with a unique number identifier (or the NYSDEC Sample Number). This identifier shall be cross-referenced to the NYSDEC Sample Number. There shall be a written description of the method of assigning this identifier and attaching it to the sample container included in the laboratory SOPs.

Samples, other than those collected for in-situ field measurements or analyses, are identified by using a sample tag which is attached to the sample container. In some cases, particularly with biological samples, the sample tag may have to be included with or wrapped around the sample. The sample tags are sequentially numbered and are accountable documents after they are completed and attached to a sample or other physical evidence. The following information shall be included on the sample tag:

- DEC spill, project number and name;
- field identification or sample point number;
- date and time of sample collection;
- designation of the sample as a grab or composite;
- type of sample (groundwater, wastewater, leachate, soil, sediment, etc.) and a very brief description of the sampling location;
- the signature(s) of the sampler(s);
- whether the sample is preserved or unpreserved;
- the general types of analyses to be conducted (VOA, PAH, EPA 624, etc.)
- any relevant comments (such as readily detectable or identifiable odor, color, or known toxic properties).

1.2.1 A Sample is under custody if:

- It is in your actual possession,
- It is in your view after being in your physical possession,
- It was in your possession and then you locked or sealed it up to prevent tampering, or
- It is in a secure area.

1.2.2 - Upon receipt of the samples in custody, the laboratory shall inspect the shipping

**FIGURE 5X.2-1 (Example)
Sample Chain-of-Custody Record***

Must be completed for samples that might be used for enforcement proceedings or litigation.					
Sample ID (Lab Use Only)	Field Reference No.	Date/Time Collected	Sample Collection Point	Type/Number: Water, Air, Soil, Etc.	Remark
Specify Method of Preservation _____ NaOH _____ Cool, 4° C _____ Acidification (specify) _____ Other (specify)			Transporting Samples During transport of the sample from sampling site to laboratory, the chain-of-custody must be unbroken. Generally, this will require that the sample be delivered by the sample collector or a designated representative, who will sign for the receipt, integrity, and transfer of the sample during shipment. <u>If integrity of sample is questioned, describe problem on reverse side of this form.</u>		

		CUSTODY OF SAMPLES		
Time	Name	Affiliation	Date	
1. Sample Container:				
Prepared by:	_____	_____	_____	_____
2. Received by:	_____	_____	_____	_____
3. Received by:	_____	_____	_____	_____
4. Sample Collected by:	_____	_____	_____	_____
5. Sample Received by:	_____	_____	_____	_____
6. Sample Received by:	_____	_____	_____	_____
7. Sample Received by:	_____	_____	_____	_____
8. Sample Received by:	_____	_____	_____	_____
9. Sample Received by:	_____	_____	_____	_____
10. Sample Rec'd for Lab by:	_____	_____	_____	_____
11. Sample Accessed by:	_____	_____	_____	_____

* This form was developed based on the chain-of-custody report used by Center for Laboratories and Research, New York State Department of Health.

FIGURE 5X2-2 (Example)

PROJECT NO.:	PROJECT NAME:							Name of Facility/Site:								
SAMPLERS (Signature)								Facility/Site Location:								
Split Samples Offered <input type="checkbox"/> Accepted <input type="checkbox"/> Declined																
STATION NO.	DATE	TIME	COMP	GRAB	SPLIT SAMPLES	TAG NUMBERS	SITE/STATION DESCRIPTION	NO. OF CONTAINERS	REMARKS							
Transferred by: (Signature)								Received by: (Signature)					Telephone			
Date:	Time:			Date:			Title:			Time:						

container and sample bottles and shall document receiving information as specified in Section 3.2. The sample custodian or a designated representative shall sign and date appropriate receiving documents at the time of receipt (i.e. chain-of-custody forms (Figure 5X.2-1), Contract Lab Receipt of Samples Information Sheets (Figure 5X.2-2), airbills, etc.). The laboratory shall contact the Bureau of Spill Prevention and Response if documents are absent, if information on receiving documents does not agree, if custody seals are not intact, or if the sample is not in good condition. The laboratory shall document resolution of any discrepancies, and this documentation shall become a part of the permanent case file.

1.2.3 - Once samples have been accepted by the laboratory, checked, and logged in, they must be maintained in accordance with custody and security requirements specified in 3.3.

2. Document Control Procedures

The goal of the laboratory document control program is to ensure that all documents for a specified case will be accounted for when the project is completed. Accountable documents used by Contractor laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to ensure that all laboratory records are assembled and stored for delivery to NYSDEC upon request from NYSDEC.

2.1 Pre-printed Data Sheet and Logbooks

Pre-printed data sheets shall contain the name of the laboratory and be dated and signed by the analyst or individual performing the work. All documents produced by the laboratory which are directly related to the preparation and analysis of NYSDEC samples shall become the property of the NYSDEC and shall be placed in the case file. For that reason, all observations and results recorded by the laboratory, but not on pre-printed data sheets, shall be entered into permanent laboratory logbooks. The person responsible for the work shall sign and date each entry and/or page in the logbook. When all data from a case is compiled, copies of all NYSDEC case-related logbook entries shall be included in the documentation package. Analysts' logbook entries must be in chronological order and shall include only one Case per page. Instrument run logs shall be maintained so as to enable a reconstruction of the run sequences of individual instruments.

Because the laboratory must provide copies of the instrument run logs to NYSDEC, the laboratory may exercise the option of using only laboratory or NYSDEC sample identification numbers in the logs for sample ID rather than government agency or commercial client names.

Using laboratory or NYSDEC Sample Number ID's only in the run sequences will assist the laboratory in preserving the confidentiality of commercial clients.

2.2 Error Correction Procedure

All documentation in logbooks and other documents shall be in ink. If an error is made, corrections shall be made by crossing a line through the error and entering the correct information. Changes shall be dated and initialed. No information shall be obliterated or rendered unreadable.

2.3 Consistency of Documentation

Before releasing analytical results, the laboratory shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or case is consistent throughout the case file.

2.4 Document Numbering and Inventory Procedure

In order to provide document accountability of the completed analysis records, each item in a case

shall be inventoried and assigned a serialized number and identifier associating it to the case and region.

Case # - Region - Serialized Number (For Example: 75-2-0240)

The number of pages of each item must be accounted for if each page is not individually numbered. All documents relevant to each case, including logbook pages, bench sheets, mass spectra; chromatographs, custody records, library search results, etc., shall be inventoried. The laboratory shall be responsible for ensuring that all documents generated are placed in the file for inventory and are delivered to NYSDEC in the Case File Purge package. Figure 1 is an example of a document inventory.

FIGURE 1 (Example) DOCUMENT INVENTORY		
<u>Document Control #*</u>	<u>Document Type</u>	<u># Pages</u>
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	Contract Lab Sample Information Sheets	10
232-2-0006	Inorganics Analysis Data Summary Sheets	10
232-2-0007	Analysts' Notebook Pages	14
232-2-0008	ICP and AA Instrument Logbook Pages	12
232-2-0009	GC/MS Spectra for Sample R-388-061-01	
232-2-0010	GC/MS Spectra for Sample R-388-0610-2	
etc.	etc.	etc.

* This number is to be recorded on each set of documents.

2.5 Shipping Data Packages and Case Files

The laboratory shall have written procedures to document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that it cannot be opened without damaging or breaking the seal. The laboratory shall also document what was sent, to whom, the date, and the method (carrier) used.

3. Standard Operating Procedures

The laboratory must have written standard operating procedures (SOPs) for: (1) receipt of samples, (2) maintenance of custody, (3) sample storage, (4) tracking the analysis of samples, and (5) assembly of completed data.

An SOP is defined as a written narrative step-by-step description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this protocol are acceptable for use in NYSDEC enforcement case preparation and litigation. The laboratory's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by NYSDEC as the basis for laboratory evidence audits.

3.1 The laboratory shall have a designated sample custodian responsible for receipt of samples

and have written SOPs describing his/her duties and responsibilities.

3.2 The laboratory shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to, documenting the following information:

- Presence or absence of appropriate chain-of-custody forms
- Presence or absence of airbills
- Presence or absence of appropriate Contract Lab Receipt of Samples Information Sheet
- Presence or absence of custody seals on shipping and/or sample containers and their condition
- Presence or absence of sample tags
- Sample tag ID numbers if not recorded on the chain-of-custody record(s) or packing list(s)
- Condition of the shipping container
- Condition of the sample bottles
- Verification of agreement or non-agreement of information on receiving documents
- Resolution of problems or discrepancies with the Bureau of Spill Prevention and Response.

3.3 The laboratory shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage and laboratory areas. The SOPs shall specifically include descriptions of all storage areas for NYSDEC samples in the laboratory, and steps taken to prevent sample contamination. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.4 The laboratory shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.4.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.4.2 A description of the documentation used to record instrument calibration and other QA/QC activities.

3.4.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.5 The laboratory shall have written SOPs for organization and assembly of all documents relating to each NYSDEC Case, including technical and managerial review. Documents shall be filed on a Case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the Case are compiled in one location for submission to NYSDEC. The system must include a document numbering and inventory procedures.

3.6 The laboratory shall have written SOPs for laboratory safety.

3.7 The laboratory shall have written SOPs for cleaning of glassware used in preparing and analyzing samples under this protocol.

3.8 The laboratory shall have SOPs for traceability of standards used in sample analysis QA/QC.

4. Handling of Confidential Information

Laboratory conducting work under this protocol may receive NYSDEC designated confidential information from the Agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

4.1 All confidential documents shall be under the supervision of a designated Document Control Officer (DCO).

4.2 Confidential Information

Any samples or information received with a request of confidentiality shall be handled as "confidential". A separate locked file shall be maintained to store this information and shall be segregated from other non-confidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the Bureau of Spill Prevention and Response. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the Bureau of Spill Prevention and Response. The DCO shall remove and retain the cover page of any confidential information disposed of for one year, and shall keep a record of the disposition in the Confidential Inventory Log.

APPENDIX 5X.3

**CHEMICALS LISTED IN THE HAZARDOUS MATERIALS TABLE (49 CFR 172.101)
USED FOR PRESERVING SAMPLES**

PRESERVATIVE	SAMPLE TYPE/ PARAMETER	pH RECOMMENDATION	QUANTITY OF PRESERVATIVE ADDED PER LITER	WT. % OF PRESERVATIVE
HCl	Volatile Organic Analysis	<2 - ≥1	4 drops conc. HCL/40 ml	0.22% (2)
HgCl ₂	Nitrogen Species	N.A.	40 mg.	0.004% (1)
HNO ₃	Metals, Hardness	<2 - ≥1	5 ml of conc. (70%)	0.35% (1)
H ₂ SO ₄	Nitrogen Species COD, Oil & Grease, P (hydrolyzable) Organic Carbon, Phenols	<2 - ≥1	2 ml of 36N	0.35% (1)
NaOH	Cyanides, Sulfides	>12 - ≤13	2 ml of 10N	0.080% (1)
Freezing* 0°C (Dry Ice)	Biological - Fish & Shellfish Tissue	N.A.	N.A.	N.A.

* - Dry ice is classified as a ORM-A hazard by DOT. There is no labeling requirement for samples preserved with dry ice, but each package must be plainly and durably marked on at least one side or edge with the designation "ORM-A". The package should also be marked "Dry Ice" or "Carbon Dioxide, Solid" and "Frozen Diagnostic Specimens". Samples must be packaged in accordance with the requirements of 49 CFR 173.615 and advance arrangements must be made between the shipper and each carrier.

N.A. - Not applicable.

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