

# ***EM*Services**

---

*Environmental Monitoring Services, LLC*

## **Field Quality Assurance Manual**

106 Hartwood Dr

Suite A

Woodstock, Ga 30189

Phone (770) 516-2081

Fax (678) 445-3276

[inquiry@emservicesonline.com](mailto:inquiry@emservicesonline.com)

*“For all your environmental monitoring needs”*

## **1.0 SAMPLING PROCEDURES**

### **1.1 INTRODUCTION**

This section will discuss the standard practices and procedures utilized by EMServices personnel for site selection and sample collection in various matrices. Also discussed are, field QA/QC procedures, and equipment care and calibration for field sampling activities. Proper collection and handling of samples is of the utmost importance to insure that collected samples are representative of the target matrix. To accomplish this, proper sampling, handling, preservation and quality control techniques for each matrix must be established and strictly followed. Precise identification of the collected samples and field documentation including a complete chain of custody are also vital and will be discussed in detail later in this section.

### **1.2 LIST OF SAMPLING CAPABILITIES**

- **Parameter Groups**

- Extractable Organics
- Volatile Organic Compounds (VOCs)
- Metals
- Inorganic Anions
- Organics
- General Chemistry
- Physical Properties

- **Sample Sources**

- Air
- Surface Water
- Wastewater
- Groundwater
- Storm Water Runoff
- Drinking water
- Sediments
- Soils
- Chemical/Hazardous Wastes
- Domestic Wastewater Sludge
- Hazardous Waste Sludge

## **1.3 GENERAL CONSIDERATIONS**

The following procedures are used in all of EMServices sampling activities. These procedures must be considered in relation to the objectives and scope of each sampling event.

### **1.3.1 Selecting a Representative Sampling Site**

Selecting a representative sampling site is dependent upon the matrix to be sampled and the type of analyses required. These matrix specific procedures are discussed in subsequent sections.

### **1.3.2 Selection and Proper Preparation of Sampling Equipment**

The type of sampling equipment to be used is specific to the sample matrix and the analyses to be conducted. These will be discussed later in this section. Section 1.10 describes the equipment cleaning procedures utilized by EMServices personnel.

### **1.3.3 Sampling Procedures for Industrial Hygiene Samples**

The following factors and procedures shall be considered and implemented in planning and executing an industrial hygiene project. All factors must be considered to develop an effective sampling strategy to achieve the desired objective.

### **1.3.4 Sampling Equipment Construction Materials**

To prevent direct contamination or cross-contamination of the collected sample, great attention must be given to the construction material of the sampling equipment. Materials used must be inert, non-porous and easy to clean. The desired materials are; Teflon®, glass, stainless steel and plastic. Plastics may not be used for collections where organics are to be analyzed. Stainless steel may not be used where metals are to be analyzed.

### **1.3.5 Selection of Parameters to be Measured**

Parameters to be analyzed are usually dictated by, and based upon, regulated monitoring conditions (NPDES or RCRA permits, for example). If these do not apply, proper analyses will be determined by EMServices or the client, based upon federal regulations specific to the matrix being investigated.

### 1.3.6 Order of Sample Collection

Unless field conditions demand otherwise, the order of sample collection is as follows:

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganic (includes Nutrients, demands, and Physical Properties)
7. Radionuclide

### 1.3.7 Special Precautions for Trace Contaminant Sampling

Many contaminants can be detected in the parts per billion and/or parts per trillion range and extreme care must be taken to prevent cross-contamination. Therefore, certain precautions apply where samples are collected for trace contaminants. These precautions are:

- A new pair of disposable latex gloves must be worn at each sampling location.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be sealed in separate plastic bags immediately after collecting and preserving.
- If possible, background samples and source samples should be collected by different field teams. If different field teams cannot be used, all background samples shall be collected first and placed in separate ice chests or shipping containers. Samples of waste or highly contaminated samples shall not be placed in the same container as environmental samples. Ice chests or shipping containers for source samples or Samples suspected to contain high concentrations of contaminants shall be destroyed and discarded after use.
- If possible, one member of the field team should handle all data recording, while the other members collect samples.
- When sampling surface waters, water samples should always be collected before sediment samples are collected.
- Sample collection activities should proceed from the suspected area of least contamination to the suspected area of greatest contamination.

- EMServices personnel should use equipment constructed of Teflon®, stainless steel, or glass that has been properly precleaned (Section 1.10) for collecting samples for trace metals or organic compounds analyses. Teflon®, glass, or plastic 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.
- When fuel powered units are utilized; they will be placed downwind and away from any site activities.
- Monitoring wells with free product shall not be sampled for trace contaminate analysis.

### 1.3.8 Sample Handling and Mixing

Sample handling should be kept to a minimum. EMServices personnel must use extreme care to avoid sample contamination. If samples are placed in an ice chest, personnel should ensure that sample containers do not become submerged or tip over as this may result in cross contamination. Small sample containers (e.g., VOCs or bacterial samples) should be placed in airtight plastic bags to prevent cross-contamination. Once a sample has been collected, it may have to be split into separate containers for different analyses. A liquid sample will be split by shaking the container or stirring the sample contents with a clean pipette or precleaned Teflon® rod. Then the contents are alternately poured into respective sample containers. Items used for stirring must be cleaned in accordance with the guidelines set forth in Section 1.10. Samples for VOCs must be collected as direct grabs. A soil sample may be split but first it must be mixed as thoroughly as possible to ensure a representative sample of the parent material. This is accomplished using the quartering method. The soil is placed in a sample pan and divided into quarters. Each quarter is mixed separately, and then all quarters are mixed together. This is repeated several times until the sample is thoroughly mixed. If a round bowl is used for mixing, mixing is achieved by stirring the material in a circular fashion and occasionally turning the material over. Soil and sediment samples collected for volatile organic compounds shall not be mixed. The appropriate sample container should be filled completely with no headspace remaining. Moisture content inversely affects the accuracy of mixing and splitting a soil sample.

### 1.3.9 Quality Control Samples

Quality control samples should be collected on all sampling projects to demonstrate that the sample materials have not been tainted by sampling equipment, chemical preservatives or procedures relating to the sample collection, transportation and storage. A summary of the recommended frequency for collecting field quality control samples is presented below.

Equipment Blank	Minimum of one; then 5%
Field Blank	Minimum of one; then 5%
Trip Blank	One per cooler containing VOC samples
Duplicate	Minimum of one; then 5%

### 1.3.10 Volatile Organic Compound Sampling

Water samples to be analyzed for volatile organic compounds are collected in pre-labeled 40-ml vials. Prior to leaving the lab, 200 µl of concentrated HCl is added to each vial. Then Teflon®-silicone septum's, Teflon® side to sample, are placed in the caps and the caps screwed into place. The sampler should check the water to be sampled for chlorine. This is done with starch iodide paper. If no chlorine is found, the vials may be filled. If residual chlorine is present, the following sampling and preservation procedure should be followed:

1. Add one crystal of sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) to each vial.
2. Fill the vial to 90% with sample.
3. Add one (1) drop of HCL.
4. Finish filling the vial with sample and cap immediately.

In filling the vial, the sample is poured slowly down the inside wall of the vial until a convex meniscus is formed. Care should be used minimize turbulence. The cap is then applied to the bottle with the Teflon® side of the septum contacting the sample. Some overflow is lost and air space in the bottle should be eliminated. Check for air bubbles by inverting the vial and tapping against the heel of the hand. This will dislodge bubbles hidden in the cap. If any bubbles are present, repeat the procedure. If unsuccessful after three attempts, discard the vial and begin the procedure again with a new vial and septum. At a minimum, duplicate samples should always be collected from each location.

Soil samples for volatile organics analysis should be sampled using traditional core sampling methods. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled. The holding time for this method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon lined screw cap),

containing, 5ml sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4°C. The holding time for this method is 14 days. Unless specifically allowed by the regulatory authority, VOC samples (liquid or solid) should never be mixed or composited.

#### 1.3.11 Oil and Grease Sampling

Aqueous samples collected for oil and grease analyses must be collected as discrete grabs. Sample containers should not be rinsed with sample water prior to sample collection. Oil and grease samples should be collected directly in the sample container. Intermediate vessels should only be used where it is impossible to collect the sample directly into the sample container. Only Teflon® beakers should be used for this purpose. Samples should be taken from well-mixed areas.

#### 1.3.12 Cyanide Sampling

Cyanide is a very reactive and unstable compound and should be analyzed as soon as possible after collection. Samples shall be collected in polyethylene or glass containers and should be preserved with NaOH and cool to 4°C.

#### 1.3.13 Biomonitoring Sampling

Aqueous samples collected for Bioassay can be collected in either glass or HDPE plastic. There is no required preservation for this type of sample and the required volume varies independently with each type of test. The samples can be held for a maximum of 36 hours from the time of collection. Grab and composite sample protocols are utilized for acute and chronic bioassays and are chosen according to the permit requirements. Samples should be collected with minimum aeration during collection and the container should be filled with no headspace.

#### 1.3.14 Procedures for Identifying Potentially Hazardous Samples

Any sample either known, or thought, to be hazardous shall be identified as such on the chain of custody. Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed.

#### 1.3.15 Collection of Auxiliary Data

All auxiliary data shall be entered in the field records. Auxiliary data relative to a particular sampling location should be recorded concurrent with the sample event. Matrix specific auxiliary data are discussed later in this section.

#### 1.3.16 Time Records

All records of time shall be kept using local time in the military (24-hour) time format and shall be recorded to the nearest minute.

#### 1.3.17 References

EMServices maintains copies of the various sampling references. Pertinent pages of these documents may be photocopied and taken to the field during sampling investigations. A bibliography of references used in the development of this section is presented in Section 1.15.

### **1.4 ANCILLARY EQUIPMENT AND SUPPLIES**

The equipment used to collect samples and conduct necessary purging activities is listed in subsequent sections for each type of sample.

### **1.5 WASTEWATER SAMPLING**

#### 1.5.1 Sampling Equipment

Continuous Wastewater Samplers, Peristaltic Pump,  
Tygon, Teflon & silicon rubber tubing;  
Glass or plastic sample containers

#### 1.5.2 General Considerations

The procedures used by EMServices are generally those outlined in the NPDES Compliance Inspection Manual. Additional guidance is given in the EPA Handbook for Monitoring Industrial Wastewater. Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed.
- Samples should not be collected directly from the surface or from the bottom of the waste stream.
  
- In sampling from wide conduits, cross-sectional sampling should be considered.
- If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquot into the composite container.

### 1.5.3 Sampling Site Selection

Wastewater samples should be collected at the location specified in the NPDES or sewer use permit if such exists. If the specified sampling location proves unacceptable, the project manager shall select an appropriate location based upon the site-specific conditions. An attempt should be made to contact the regulating authorities for their approval. The potential for this type of problem highlights the need for a site inspection prior to the time scheduled for a sampling event.

#### 1.5.3.1 Influent

Influent wastewaters should be sampled at points of high turbulence and mixing. These points are: (1) the up flow siphon following a comminutor (in absence of grit chamber); (2) the up flow distribution box following pumping from main plant wet well; (3) aerated grit chamber; (4) flume throat; or (5) pump wet well when the pump is operating. Raw wastewater samples should be collected upstream of side stream returns.

#### 1.5.3.2 Effluent

Effluent samples should be collected at the site specified in the permit or, if no site is specified, at the most representative site downstream from all entering wastewater streams prior to final discharge.

#### 1.5.3.3 Pond and Lagoon Sampling

Composite samples of pond and lagoon effluent are preferred over grabs due to the potential for ponds and lagoons to short-circuit their projected flow paths. However, if dye studies or facility data indicate a homogeneous discharge, grab samples may be taken.

### 1.5.4 Sampling Techniques: General

The choice of a flow-proportional or time-proportional composite sampling program depends upon the variability of flow, equipment availability, sampling point configuration and accessibility. Flow metered sampling is necessary for complete wastewater characterization and should be utilized where possible. Otherwise, a time-proportional composite sample is acceptable. A time-proportional composite sample consists of aliquots collected at constant time intervals and can be collected either manually or with an automatic sampler. A flow-proportional composite sample consists of aliquots collected automatically at constant flow intervals with an automatic sampler and a flow-measuring device. Prior to flow-proportional sampling, the flow measuring system (primary flow

device, totalizer, and recorder) should be examined. The sampler may have to install flow measurement instrumentation if automatic sampling is to be used.

### 1.5.5 Use of Automatic Samplers

#### 1.5.5.1 General

Automatic samplers are used when several points are to be sampled at frequent intervals, with limited personnel, or when a continuous sample is required. Automatic samplers used by EMServices must meet the following requirements:

- Must be properly cleaned to avoid cross-contamination from prior sampling events.
- No plastic or metal parts shall be exposed to the sample when parameters to be analyzed could be impacted by these materials.
- Must be able to provide adequate refrigeration. Commercially available ice is placed in the sampler base and packed around the container approximately half way up the sample container.
- Must be able to collect a large enough sample for all required analyses. Composite sample containers (glass or plastic) hold up to 10 liters.
- A minimum of 100 milliliters should be collected each time the sampler is activated.
- Should provide a lift of at least 20 feet and be adjustable so that sample volume is not a function of pumping head.
- Pumping velocity must be adequate to transport solids without settling.
- The intake line must be purged a minimum of one time before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- Have a power source adequate to operate the sampler for 48 hours at 15-minute sampling intervals.
- Facility electrical outlets may be used if available.
- Facility automatic samplers may be used for conventional parameters if they meet EMServices QA/QC criteria.

Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers presently used by EMServices are included in the respective operating manuals and are not presented

here. All data relative to the actual use of automatic equipment on a specific job is recorded in the EMServices Field Log Book.

#### 1.5.5.2 Equipment Installation

##### 1.5.5.2.1 Conventional Sampling

Automatic samplers may be used to collect time-proportional composite or flow-proportional composite samples. In the flow-proportional mode, the samplers are activated by a compatible flow meter. Flow-proportional samples can also be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow-proportional amounts. Installation procedures include cutting and installing the proper length of tubing, positioning it in the wastewater stream, and sampler programming. All new tubing (Dow® Corning Medical Grade Silastic, or equal, in the pump and Tygon®, or equal, in the sample train) will be used for each sampler installation. For a time-proportional composite, the sampler should be programmed to collect 100 ml samples at 15-minute intervals into a refrigerated 10-liter plastic or glass jug, as appropriate for the particular parameters being analyzed. For a flow-proportional composite, the sampler should be programmed to collect a minimum of 100 mls for each sample interval. The sampling interval should be based on the flow of the waste stream.

##### 1.5.5.3 Automatic Sampler Maintenance, Calibration, and Quality Control

To ensure proper operation of automatic samplers, the procedures outlined in this section shall be used to maintain and calibrate EMServices automatic samplers. Any variance from these procedures will be documented. Proper sampler operation will be checked by EMServices personnel prior to each sampling event. This includes checking operation through three cycles of purge-pump-purge; checking desiccant and replacing if necessary; checking charge date on NiCad batteries to be used; and repairing or replacing any damaged items. Prior to initiating sampling, the purge-pump-purge cycle shall be checked at least once. The sample volume will be calibrated using a graduated cylinder at least twice, and the flow pacer that activates the sampler shall be checked to be sure it operates properly. Upon return from a field trip, the sampler shall be examined for damage. The operation will be checked and any required repairs will be made and documented. The sampler will then be cleaned as outlined in Section 1.10.

### 1.5.6 Manual Sampling

Manual sampling is normally used for collecting grab samples and for immediate in-situ field analyses. Manual sampling may also be used when it is necessary to evaluate unusual waste stream conditions. If possible, manually collected samples should be collected in the actual sample container that will be submitted to the laboratory. This minimizes the possibility of contamination from an intermediate collection container. Manual samples are collected by (1) submerging the container neck first into the water; (2) inverting the bottle so that the neck is upright and pointing into the direction of wastewater flow; (3) quickly returning the sample container to the surface; (4) shake to rinse. Pour the contents out downstream of sample location; (5) collect sample as described in steps 1, 2, and 3; pour out a few mls of sample downstream of sample collection. This allows for addition of preservatives and sample expansion. Exceptions to the above procedure occur when preservatives are present in the sampling container or when oil & grease, microbiological, and/or VOC analyses are needed. In these cases, sample shall be collected directly into the container with no pre-rinsing.

If the water or wastewater stream cannot be physically or safely reached, an intermediate collection container may be used. This container must be properly cleaned (Section 1.10) and made of an acceptable material. A separate collection container should be used at each sampling station to prevent cross-contamination between stations. The sample is collected by lowering a properly cleaned Teflon®, plastic, or glass collection vessel into the waste stream. The intermediate vessel may be lowered by hand, pole or rope.

### 1.5.7 Trace Organic Compounds and Metals

Due to the ability to detect trace organic compounds and metals in extremely low concentrations, care must be taken to avoid contamination of the sample. All containers, composite bottles, tubing, etc., used in sample collection for trace organic compounds and metals analyses should be prepared as described in Section 1.10. Personnel handling the sample should wear a new pair of disposable latex gloves with each set of samples collected to prevent cross-contamination. A more detailed discussion is given in Section 1.3.6 under special precautions for trace contaminant sampling.

### 1.5.8 Bacterial Analysis

Samples for bacterial analysis will always be collected directly into the prepared glass or plastic sample bottle. The sample bottle should be kept closed until actual filling and never rinsed with sample. When the container is opened, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should

be held near the base, plunged neck downward, below the surface and turned until the neck points upward and upstream. The bottle should be filled to within one-inch of the top and capped immediately. Section 1.13 presents preservation procedures and holding times. As holding times are limited to 6 hours for microbiological analyses, special arrangements may be required to ensure that these samples reach a laboratory within this period.

#### 1.5.9 Immiscible Liquids/Oil and Grease

Oil and grease may be present in wastewater as a surface film, emulsion, solution, or a combination of these forms. A representative sample for oil and grease analysis is difficult to collect. The sampler must carefully evaluate the location of the sampling point to find an area of greatest mixing. Quiescent areas should be avoided. Because losses of oil and grease will occur on sampling equipment, collection by composite sampler is impractical. Intermediate sampling vessels should not be used if possible. If intermediate collection vessels are required, they should be made of Teflon® and be rinsed with sample three times before transferring any sample to the sample container. Sample containers, however, should never be rinsed.

#### 1.5.10 Volatile Organic Compounds Analyses

Water samples to be analyzed for volatile organic compounds are collected in 40-ml pre-preserved (200 $\mu$ l of concentrated HCl) vials with screw caps. Teflon®-silicone septums are placed in the caps in the laboratory prior to the sampling event. The Teflon® side must be facing the sample side. Sampling containers with preservatives are pre-labeled prior to any field activities to reduce the chances of confusion during sampling activities. A complete list of sample preservatives, containers, holding times, and volumes is found in Section 1.12. The sampler should check the water to be sampled for chlorine. This is done with starch iodide paper. If no chlorine is found, the vials may be filled. If residual chlorine is present, the sampling and preservation procedures listed in Section 1.3.9 of this manual.

#### 1.5.11 Auxiliary Data Collection

While conducting wastewater sampling, the following information may also be gathered:

- Field measurements -- pH, DO, conductivity, temperature (plus others as required by permit conditions)
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples
- Diagrams and/or written descriptions of the sample locations

- Photographs of pertinent wastewater-associated equipment, such as flow measuring devices, treatment units, etc.
- Completion of applicable forms required during specific investigations.
- All observations, measurements, diagrams, etc., will be entered in field logbooks or attached thereto.

## **1.6 SURFACE WATER AND SEDIMENT SAMPLING**

### 1.6.1 Equipment

#### Surface Water Sampling

Kemmerer Sampler  
Grab Samplers  
Automatic Samplers  
Bailers

#### Sediment Sampling

Hand Augers  
Sediment Core Sampler  
Encore™ Sampling  
Scoops  
Mixing Bowls  
Spoons  
Spatulas

### 1.6.2 General

Selection of surface water sampling locations for water quality studies are determined by the objective of the study and waterway type. Factors that impact and alter water quality and characteristics (dams, bridges, discharges, etc.), must be considered. Accessibility to a sample site is also important.

### 1.6.3 Sample Site Selection

Fresh water environments are commonly divided into two types: (1) rivers, streams, and creeks; and (2) lakes, ponds, and impoundments. Since these waterways differ considerably in general characteristics, site selection must be adapted to each. Prior to conducting a sampling event, an initial survey should be conducted to locate prime sampling points. Bridges and piers provide ready access to sampling points across a body of water. However, they should only be used when at acceptable locations and are found not to be detrimentally impacting stream characteristics. If wading for water samples must be done, caution should

be used to avoid disturbing bottom deposits that could result in increased sediment in the sample. Wadeable areas may be best for sediment sampling.

#### 1.6.3.1 Rivers, Streams, and Creeks

Sampling sites should be located in areas possessing the greatest degree of cross-sectional homogeneity. Such points are easily found directly downstream of a riffle or rapid. These locations are also good for sediment sampling. In the absence of turbulent areas, a site that is clear of immediate point sources, such as tributaries and effluent discharges, may be used. Typical sediment deposition areas are located at the inside of river bends and downstream of islands or other obstructions. Sites immediately upstream or downstream from the confluence of two streams or rivers should be avoided due to inadequate mixing of the combining flows. In addition, backflow can upset normal flow patterns. Great attention should be given to site selection along a stream reach:

- Sites should be spaced at intervals based on time-of-water-travel. Sampling sites may be located about one-half day time-of-water-travel for the first three days downstream of a waste source for the first six sites and then approximately one day for the remaining distance.
- If the study data is to be compared to previous study data, the same sampling sites should be used.
- Sites should be located at marked physical changes in the stream channel.
- Site locations should isolate major discharges as well as major tributaries.

Dams and weirs usually create quiet, deep pools in river reaches that would otherwise be swift and shallow. When times of travel through them are long, sites should be established within them. Some structures, such as dams, permit overflow that may cause significant reaeration of oxygen deficient water. Sites should be located short distances upstream and downstream of these structures to measure the rapid, artificial increase in dissolved oxygen (DO), which is not representative of natural reaeration. A minimum of three sites should be located between any two points of major change in a stream, even if the time-of-travel between the points of change is short. Major changes include, but are not limited to, a waste discharge, a tributary inflow, or a significant change in channel characteristics. Sampling three sites is also important when testing rates of change of unstable constituents. Results from two of three sites will usually support each other and indicate the true pattern of water quality in the sampled zone. If the effects of certain discharges or tributary streams

are of interest, sites should be located both upstream and downstream of these points.

Due to the tendency of the influent from a waste discharge or tributary to mix slowly with the main stream, it is nearly impossible to measure their effect immediately downstream of the source. Thus, samples from quarter points may miss the wastes and only indicate the quality of water above the waste source. Conversely, samples taken directly in the stream portion containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole. Tributaries should be sampled as near the mouth as possible. Often, these may be entered from the main stream for sampling by boat. Care should be taken to avoid collecting water from the main stream that may flow back into the tributary because of density differences created by temperature, salinity, or turbidity differences. Actual sampling locations will vary with the size and amount of turbulence in the stream or river. Generally, with streams less than 20 feet wide, well mixed areas and sampling sites are readily found. In such areas, a single grab sample taken at mid-depth at the center of the channel is adequate. A sediment sample can also be collected at the center of the channel. For slightly larger streams, at least one vertical composite should be taken from mid-stream. It should be composed of at least one sub-surface, mid-depth, and above the bottom sample. DO, pH, temperature, conductivity, etc. should be measured on each aliquot of the vertical composite. Several locations should be sampled across the channel width on the larger rivers. Vertical composites across the channel width should be located proportional to flow, i.e., closer together toward mid-channel where flow is greater and less toward the banks where the flow proportionally lower. The number of vertical composites and depths sampled for each area shall be determined by the field crew. They should base their decisions utilizing two considerations.

1. The larger the number of subsamples, the more nearly the composite sample will represent the water body
2. Taking subsamples is time consuming and expensive, and increases the chance of contamination.

A number of sediment samples should be collected along a cross-section of a river or stream to adequately characterize the bed material. The normal procedure is to sample at quarter points along the cross-section of the site. When the sampling technique or equipment requires that the samples be extruded or transferred at the site, they can be combined into a single composite sample. However, samples of dissimilar composition should not be combined. They should be kept separate for analysis in the

laboratory. To ensure representative samples, coring tubes are employed. The quantity of each subsample that is composited shall be recorded.

#### 1.6.3.2 Lakes, Ponds, and Impoundments

Lakes, ponds, and impoundments have a much greater tendency to stratify than rivers and streams. This lack of mixing requires that more samples be obtained from the different strata. Occasionally, extreme turbidity differences occur vertically where a highly turbid river enters a lake. This stratification is caused by temperature differences where the cooler, heavier river water flows beneath the warmer lake water. A temperature profile of the water column and visual observation of lake samples can detect these layers. Each layer of the stratified water column should be sampled. The number of sampling sites on a lake, pond, or impoundment is determined by the objectives of the investigation dimensions of the basin. In small bodies of water, a single vertical composite at the deepest point may be sufficient. DO, pH, temperature, etc., is to be conducted on each vertical composite aliquot.

In naturally formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam. In lakes and larger impoundments, several vertical subsamples should be composited to form a single sample. These vertical sampling locations should be along a transect or grid. Here too, the number of vertical subsamples and sample depths for each area shall be determined by the field crew. In some cases, separate composites of epilimnetic and hypolimnetic zones may be required. Additional separate composite samples may be needed to adequately represent water quality in a lake possessing an irregular shape or numerous bays and coves. Additional samples should always be taken where discharges, tributaries, agriculture, and other such factors are suspected of influencing water quality.

When collecting sediment samples in lakes, pond, and reservoirs, the sample site should be as near as possible to the center of water mass, especially for impoundments of rivers or streams. Generally, coarser grained sediments are deposited at the headwaters of a reservoir, and the finer sediments are near the center. The shape, inflow pattern, bathymetry, and circulation affect the location of sediment sampling sites in large bodies of water.

#### 1.6.3.3 Control Sites

The collection of samples from control sites is necessary to compile a basis of comparison of water quality. A control site above the point of

interest is as important as the sites below, and must be chosen with equal care. Two or three sites above the waste inflow may be necessary to establish the rate at which any unstable material is changing. The time of travel between the sites should be sufficient to permit accurate measurement of the change in the material under consideration.

#### 1.6.4 Sampling Equipment and Techniques

##### 1.6.4.1 General

Any equipment or sampling techniques used to collect a sample must not alter the integrity of the sample and must be capable of providing a representative sample.

##### 1.6.4.2 Water Sampling Equipment/Techniques

The physical location of the collector will dictate the type of equipment needed to collect samples. Surface water samples may be collected directly into the sample container when possible. Pre-preserved sample containers shall never be used as intermediate collection containers. Samples collected in this manner shall use the methods specified in Section 1.5.6 of this manual. If wading into the stream is required, care should be taken not to disturb bottom deposits that could be collected and bias the sample. In addition, the sample should be collected directly into the sample bottle and **up current** of the wader. If wading is not possible or the sample must be collected from more than one depth, additional sampling equipment may be used. If sampling from a powerboat, samples must be collected upwind and upstream of the motor.

###### 1.6.4.2.1 Sampling Procedure Using a Teflon® or PVC Bailer

If data requirements for surface water sampling do not necessitate sampling from a strictly discrete interval of the water column, Teflon® or PVC constructed bailers can be used for sampling. The type bailer used is dependent on the analytical requirements. A closed top bailer utilizing a bottom check valve will be sufficient for many surface water studies. Water is continually displaced through the bailer as it is lowered down through the water column until the specified depth is attained. At this point, the bailer is retrieved back to the surface. There is the possibility of contamination to the bailer as it is lowered through the upper water layers. Also, this method may not be successful in situations where strong currents are found or where a discrete sample at a specified depth is needed. If depth specific, discrete samples are needed, and

the parameters do not require Teflon® coated sampling equipment; a standard Kemmerer sampler may be used. A plastic bucket can also be used to collect surface samples if parameters to be analyzed do not preclude its use. The bucket shall always be rinsed twice with the sample water prior to collection and the rinse water be disposed of downstream from the sample collection point. All field equipment will be cleaned using standard cleaning procedures.

#### 1.6.4.2.2 Sampling Procedure Using a Kemmerer Sampler

Due to the PVC construction of the Kemmerer sampler, it shall not be used to collect samples for extractable organics, VOCs, and/or oil & grease analysis. The general collection procedure is as follows:

1. Securely attach a suitable line to the Kemmerer bottle.
2. Lock stoppers located at each end of the bottle on the open position. This allows the water to be drawn around the bottom end seal and into the cylinder at the specified depth.
3. The bottle is now in the set position. A separate "messenger" is required to activate the trip mechanism, which releases the stopper and closes the bottle.
4. When the bottle is lowered to the desired depth, the messenger is dropped. This unlocks the trip mechanism and forces the closing of both end seals.
5. Raise the sampler, open one of the end seal, and carefully transfer the sample to the appropriate sample container.

#### 1.6.4.2.3 Sampling Procedures Using Containers

In most cases, sample collection containers are used to collect surface water from easily accessible sampling points. This means that the sample is collected manually, always upstream of the sampling person's position. An extension may be added to the container to make the sampling point more accessible for manual sampling. Extensions can be constructed of aluminum, PVC, steel, or any other suitable material. The sample container is normally attached to the extension using a clamp, vinyl pull ties, or duct tape. Samples collected in this way are done so in the following manner:

1. Place the inverted sample container into the water and lower to the desired depth. Never use a pre-preserved container as an intermediate sample collection device.

2. Reinvert the container with the mouth facing into the direction of flow and at the appropriate depth to collect the desired sample.
3. Carefully raise the container to the surface and transfer to the appropriate container.

#### 1.6.4.3 Sediment Sampling Equipment/Techniques

A variety of methods can be used to collect sediment samples from a streambed. EMServices utilizes corers and scoops. Precautions must be taken to ensure that the sample collected is representative of the streambed. These methods are discussed in the following paragraphs.

##### 1.6.4.3.1 Sediment Core Sampler

Core sampling is used to collect vertical columns of sediment from the stream or lakebed. Many types of coring devices are available for use depending on the depth of water from which the sample is obtained, the type of bottom material, and the length of the core to be collected. Some devices are weight or gravity driven while others are simple hand push tubes. These devices minimize the loss of fine particles and should always be used when collecting sediment samples from flowing waters.

Coring devices are particularly useful in pollutant monitoring because the shock wave created by sampler is minimized and the fines at the sediment water interface are only slightly disturbed. The sample can be withdrawn primarily intact removing only the layers of interest. Core liners manufactured of Teflon® or plastic can be purchased. These liners reduce the possibility of contamination and can be delivered to the laboratory in the tubes in which they were collected. Coring devices sample small surface areas and small sample sizes and often require repetitive sampling to obtain a sufficient amount of sample. This is the primary disadvantage to these devices but they are recommended in the sampling of sediments for trace organic compounds or for metals analyses.

When sampling sediments in wadeable water, the direct use of a core liner is recommended. Stainless steel push tubes are also used because they provide a better cutting edge and higher tensile strength than Teflon® or plastic. One advantage to using the Teflon® or plastic tubes is the elimination of possible metals contamination of the sample from the core barrels or cutting heads. The length of the corer tube should correspond to the desired depth

of the layer being sampled. In general, soft sediments adhere better to the inside of the tube and a larger diameter tube can be used. Coarser sediments require the use of a smaller diameter tube of two inches or less to prevent the sample from falling out of the tube. The inside bottom wall of the tube can be filed down to allow easier entry into the substrate. When samples are obtained by wading, caution should be used to minimize disturbance in the area sampled. Core tubes are pushed directly down into softer substrates until four inches or less of the tube is above the sediment-water interface.

A slight rotation of the tube may be necessary to facilitate ease of entry into harder substrates and reduce compaction of the sample. The tube is then capped and slowly extracted and the bottom of the corer is capped before it is pulled above the water surface.

Subsampling is performed for VOC sample collection using an Encore™ sampling device. This device is used to collect soil/sediment samples, while preventing container headspace. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled to 4°C. The holding time for this method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon lined screw cap) containing, 5ml sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4°C. The holding time for this method is 14 days.

#### 1.6.4.3.2 Scooping Samples

The easiest and quickest way to collect a sediment sample in wadeable water is with a Teflon® coated scoop or stainless steel spoon. This type of sampling should be limited to quiescent (i.e., non-flowing) waters such as lakes or reservoirs.

#### 1.6.4.3.3 Mixing

As specified in Section 1.3.7., sediment samples collected for chemical analysis should be thoroughly mixed (except for volatile organic compounds analysis) before being placed in the sample containers.

### 1.6.5 Special Sample Collection Techniques

#### 1.6.5.1 Trace Organic Compounds and Metals

Samples for trace pollutant analyses in surface water should be collected by dipping the sample containers directly into the water. Sometimes samples are to split for enforcement or quality control purposes. A sufficient volume for all sample containers should be collected in a large glass container and then, while mixing, be alternately dispensed into the appropriate sample bottles. This cannot be done for volatile organic compound samples. Only Teflon® or stainless steel should be used in sediment sampling for trace contaminant analyses. Teflon® coring tubes are the preferred technique.

#### 1.6.5.2 Bacterial Analysis

Samples for bacteriological examination must be collected in sterilized bottles and protected against contamination. The sample is preferably collected directly into the sample bottle. This is done by holding the bottle near the base and plunging, neck downward, below the surface. The container is then turned with the neck pointed slightly upward and the mouth directed toward the current. The bottle is filled to about 1/2 inch from the top and recapped immediately. While the bottle is open, extreme care should be used to protect both the bottle and stopper against contamination. The 1/2 inch air space is left in the bottle to facilitate subsequent shaking in the laboratory. If sampling with an intermediate sampling device (i.e., bailer), the device shall be thoroughly rinsed with sample water prior to collecting the sample. This is the reason microbiological samples are among the final samples collected from a sampling location. Begin pouring sample out of the sampling device before collecting into the sterilized container. Continue pouring sample out of the device, place the container under the flowing stream, and fill the container to 1/2 inch from the top. Flow should remain continuous before and during the filling process. When sampling from a bridge, the sterilized sample bottle can be weighted and lowered to the water on a rope. Collectors must be careful not to dislodge debris from the bridge that could fall into the bottle.

### 1.6.6 Auxiliary Data Collection

A field logbook will be used to record data pertinent to the sampling activities. This data shall describe all sampling locations and techniques, list photographs taken, visual observations, etc. Visual observations of sample site conditions,

weather and overall stream conditions, recorded during the investigation can be valuable in interpreting water quality results.

#### 1.6.7 Split and Duplicate Sample Collection

The splitting of samples is utilized to measure any variability between laboratories and are collected as subsamples of a single sample. This is unlike duplicate samples that are collected to measure any variability in the sampling and analytical methods of an individual lab. Duplicate samples are collected in close succession as sub samples of a single sample.

##### 1.6.7.1 Split Sample Collection

1. Sample must be collected in a properly cleaned container composed of acceptable materials (Sample should be more than twice the volume required for one sample).
2. Add appropriate preservative where required and mix thoroughly.
3. Alternately, decant sample into subsample containers in increments of approximately 10 percent of total subsample volume until full.
4. Seal the sample containers with appropriate, airtight caps.
5. Label each sample container with a field number and complete a chain of custody.

**NOTE:** Volatile organic samples shall not be collected in this manner. Samples for VOCs must be collected as simultaneous, discrete grab samples.

##### 1.6.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession.
2. Preserve where required.
3. Mix thoroughly.
4. Seal the sample containers with appropriate, airtight caps.
5. Label each sample container with a field number and complete a chain of custody.

## 1.7 GROUNDWATER AND DRINKING WATER SAMPLING

### 1.7.1 Groundwater Sampling Equipment

Bailers (disposable and non-disposable)  
Peristaltic Pump  
Tygon Tubing  
Teflon® Tubing

Silastic Rubber Tubing  
Bladder Pump  
Centrifugal Pump

### 1.7.2 General Groundwater Sampling

Groundwater sampling is necessary for a number of purposes. This includes, but is not limited to, evaluating potable or industrial water sources, mapping contaminant plume movement at a land disposal or spill site, RCRA compliance monitoring (landfills), or examining a site where groundwater contamination may have or may be occurring. Normally, groundwater is sampled from a permanent monitoring well. However, this does not exclude collection of samples from a sinkhole, pit, or other drilling or digging site where groundwater is present. Monitoring wells are not always at the optimum location to collect samples for the purpose they are needed. In these situations, additional wells may need to be drilled. Experienced, knowledgeable individuals (hydrologists, geologists) are needed to site the well and oversee its installation so that representative samples of the groundwater can be collected. EMServices utilizes the procedures being reviewed in this section. Further guidance is available in the RCRA Groundwater Monitoring Technical Enforcement Guidance Document (TEGD); EMServices field personnel will at a minimum meet, and when possible exceed, the requirements of this document.

### 1.7.3 Measurement of Well Water Level and Stagnant Water Volume Calculation

The sampling and analysis plan provide for measurement of standing water levels in each well prior to each sampling event. Field measurements will include depth to standing water surface and total depth of the well. This data will then be utilized to calculate the volume of stagnant water in the well and provide a check on the integrity of the well (e.g., siltation problems). The measurement should be taken to 0.01 foot when possible. A battery powered level sensor will be used to measure depth to the surface of the groundwater. Equipment shall be constructed of inert materials and will be cleaned per sample equipment cleaning procedures prior to use at another well. Field data will be recorded on the Monitoring Well Data Sheet

#### 1.7.3.1 Procedure for Water Level Measurement

1. Clear debris from area around well (lay plastic sheathing around well pad as an option).
2. Remove protective casing lid.
3. Open monitoring well lid.
4. Lower the clean water level indicator probe down into the well. A beep will sound upon contact with the water surface. False readings

can be made from the wetted side of the well so it will be necessary to check the level several times until a consistent reading is achieved. Record the distance (to the nearest 0.01ft.) from the top of the well casing to the water surface on the Monitoring Well Data Sheet.

5. Continue to lower the probe until it reaches the well bottom. Record the distance (to the nearest 0.01 ft) from the top of the well casing to the bottom of the well on the Monitoring Well Data Sheet.
6. All water level and well depth measurements shall be made from the top of the well casing unless specified otherwise by the project manager or DER.
7. The wetted depth is obtained by subtracting total well depth from the surface level depth.

#### 1.7.3.2 Calculating Water Volume

Total volume of standing water in a well is calculated by the following formula:

$$V = \pi r^2 h \times 7.48 \text{ gallons/ft}^3$$

Where;

V = volume of standing water in the well (gallons)

r = radius of well (ft)

h = depth of water column in the well (ft)

$\pi = 3.14$

7.48 = conversion factor

#### 1.7.4 Well Evacuation

##### 1.7.4.1 Procedure for Well Evacuation: Wells without In-Place Plumbing

Water standing in a well may not be representative of actual groundwater conditions. The standing water in a well should be removed to allow representative formation water to supplant the stagnant water. The evacuation method depends on the hydraulic characteristics of the well but the following general rules apply. The total amount of water purged must be recorded. Therefore, the volume must be measured during the purging operation. This may be determined by:

1. Collecting the water in a graduated or known volume container (i.e., bucket);
2. Calculate the volume based on the pump rate. pump rate may not be constant and field personnel should be aware of this;

3. Record the time that the actual purging begins in the field record. Purging is considered complete if any one of the following criteria is satisfied:
  - i. Field parameters (pH, temperature, conductivity (Dissolved Oxygen, ORP, Turbidity (Optional))) stabilize within 5% in consecutive readings at least 5 minutes apart. If field parameters have not stabilized after 3 well volumes, the purging is considered complete and sampling can begin.
  - ii. Three well volumes are purged with no monitoring of field parameters.
4. At least one fully dry purge. A second dry purge may be necessary in some situations.

Except for low recovery wells, all wells shall be sampled within 6 hours of purging. Low recovery wells may be sampled as soon as sufficient sample matrix is available or up to 10 hours after purging. Wells that do not recover sufficiently within 10 hours should not be sampled.

Purging equipment includes Teflon® or stainless steel bailers or a peristaltic pump. Any fuel-powered pumping units shall be placed downwind of any sampling site. If purging equipment is reused, it shall be cleaned following standard procedures. Disposable latex gloves shall be worn by sampling personnel and changed prior to starting work at each sampling site.

If the purge water is determined to be hazardous, it should be disposed of in an appropriate manner. The Florida Department of Environmental Regulation requires that during purging of the well, the purging device should be placed just below the surface of the water level and be lowered with the falling water level. For high yield wells, three casing volumes should be evacuated prior to collecting samples. Purging should be conducted at a rate to minimize agitation of the recharge water. Measuring conductivity, pH, and temperature during purging is necessary to monitor variability of the groundwater. **Samples should be collected within 6 hours of purging high yield wells.**

Low-yield wells (incapable of yielding three casing volumes) should be evacuated to dryness at a rate that does not cause turbulence. When the well recovers sufficiently, the first sample should be tested for pH, temperature, and conductivity. When recovery exceeds two hours, the sample should be collected as soon as sufficient volume is available. **If recovery is longer than 10 hours, the well should not be tested.** The project manager may wish to review available information to determine if obtaining a representative sample is possible.

#### 1.7.4.2 Procedure for Well Evacuation: Teflon® Bailer

1. Clear the area around the well pad; cover with plastic if necessary.
2. Slowly lower the bailer to the water surface and remove it when full.
3. Reel or pull bailer to the surface using precaution to not allow the lanyard (cable or string) to touch the ground.
4. Use the bailer volume and number of bails removed to determine volume of water removed. Hazardous material should be poured into a container for later disposal.
5. Repeat steps 2 and 3 until 1.5 well volumes have been removed.
6. Begin monitoring for pH, temperature, and conductivity. Record on Monitoring Well Data Sheet. Discard the sample into the collection pail. Purge until the change between samples of each parameter is less than 5 percent.
7. Continue until at least three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
8. Record date and time the well was purged on the Monitoring Well Data Sheet.

**NOTE: For wells sampled in the State of Florida, three well volumes will be purged prior to pH, temperature, and conductivity screening. Following evacuation of three well volumes, purge water will be screened for these parameters at regular intervals until two consecutive measurements are within 5 percent. The intervals may be time-based (at least 5 min) or represent a portion of the well volume (at least 0.5 well volume) Compliance with more stringent local, State, or Regional guidelines will be maintained where required.**

#### 1.7.4.3 Procedure for Well Evacuation: Peristaltic Pump

1. Clean area around the well pad.
2. Install the appropriate length of Tygon or Teflon® tubing into the pump mechanism.
3. Insert the uncontaminated sampling end of the tubing into the well surface.
4. Connect the pump to power supply.
5. Operate the pump at a rate that does not cause excessive agitation of the replacement water.
6. Determine the pump flow rate.
7. Purge until 1.5 well volumes have been evacuated.

8. Collect samples at a rate of one per well volume evacuated. Monitor these samples for pH, temperature, and conductivity. Record these measurements on the Monitoring Well Data Sheet. Monitor until the difference in each parameter is less than 5 percent.
9. Continue purging until three well volumes have been evacuated and the parameters pH, temperature, and conductivity are within 5 percent, or until a low yield well has been evacuated to dryness.
10. Record the date and time the well was purged on the Well Sampling Field Data Sheet.

### 1.7.5 Purging Techniques: Wells with In-Place Plumbing

#### 1.7.5.1 General

The volume to be purged is dependent on whether the pumps are running continuously or intermittently and how close to the source samples can be collected. If storage/pressure tanks are present, a volume must be purged to totally exchange the volume of water in the tank.

#### 1.7.5.2 Continuously Running Pumps

For continuously running pumps, the well should be purged by opening the valve and allowing it to flush for 15 minutes if the well volume is unknown. If the sample is collected after a holding tank, the volume of the tank should also be purged.

#### 1.7.5.3 Intermittently Running Pumps

Wells shall be purged at maximum rate for at least 15 minutes. Monitoring of field parameters will continue until two consecutive measurements within 5 percent are measured at 5-minute intervals.

### 1.7.6 Sample Withdrawal

Technique for withdrawal is dependent on the parameters to be analyzed. To collect a representative sample and minimize the possibility of sample contamination:

- Use Teflon® or stainless steel sampling devices when organics are a concern.
- Use dedicated tubing or samplers for each well. If a dedicated sampler is not available, clean the sampler between sampling events. Analyze equipment blanks to ensure cross-contamination has not occurred.

The preferred sample collection order is as follows (decreasing volatility):

1. Volatile organic compounds (VOCs)
2. Extractable Organics (includes Total Recoverable Petroleum Hydrocarbons [TRPH], Oil & Grease, Pesticides and Herbicides)
3. Total metals
4. Dissolved metals
5. Microbiological
6. Inorganic (includes Nutrients, demands, and Physical Properties)
7. Radionuclide

The following items are acceptable sampling devices for all parameters:

- A gas-operated, Teflon® or stainless steel squeeze pump (also referred to as a bladder pump with adjustable flow control) should be dedicated or completely cleaned between sampling events. If it is dedicated, the protocols on its use, flow rates and use of flow controls should be discussed.
- A Teflon® bailer with check valves and a bottom-emptying device. Dedicated or disposable bailers should not be cleaned between purging and sampling operations.

EMServices generally supplies sampling devices for wells sampled by EMServices. However, some clients have wells equipped with dedicated sampling devices. All dedicated equipment will be cleaned between sampling events with the exception of dedicated pump systems or dedicated pipes that are never removed. EMServices will evaluate the device and the project manager shall approve/disapprove of the dedicated device prior to sampling. If sampling includes dissolved parameters, samples shall be filtered in the field in the following manner:

1. Use a one piece, molded, in-line high capacity disposable 1.0-micron filter when collecting samples for dissolved trace metals analysis. Use a 0.45-micron filter when sampling for all other (i.e., orthophosphorous, silica, etc.) dissolved parameters.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the positive pressure side of the peristaltic pump.
4. If well is deeper than 25 feet, a submersible bladder pump may be necessary to bring the sample to the surface. Samples shall not be collected in an intermediate container.
5. At least one filtered equipment blank using deionized water must be collected and analyzed.
6. The sample shall be preserved as required following filtration.
7. Unfiltered samples will be collected in conjunction with filtered samples.

**NOTE:** Filtered samples will be collected only at the request of DER and will not be collected for turbid samples only.

#### 1.7.1.1 Sample Removal: With In-Place Plumbing

Samples should be collected following purging from a valve or tap as near to the well as possible, and ahead of all screens, aerators, filters, etc. Collected shall be directly into the containers. Flow rate should not exceed 500 ml/min.

#### 1.7.1.2 Sample Removal: Without In-Place Plumbing

1. Following purging, collect the sample and pour it directly from the bailer into the sample container. If a peristaltic pump is used, pump the directly into the container. Collect the samples in order of decreasing volatilization sensitivity.
2. Measure the conductivity, pH, and temperature of the samples, and record the results on the Monitoring Well Data Sheet.
3. If a bailer is not dedicated, clean equipment using standard procedures. Collect blanks at a rate of one per type of equipment field cleaned. If a piece of equipment is cleaned more than twenty times, collect blanks at a rate of 10 percent. An equipment blank must be taken and preserved for each analyte method group.
4. If a bailer is used to collect samples, replace the bailer string. Take precautions not to allow the string to touch the ground. Dispose of the used string properly. If Teflon® or stainless steel cable is used, clean according to standard procedures and do not let it touch the ground.
5. Replace the well cap, close, and lock the protective casing lid.

#### 1.7.7 Split and Duplicate Sample Collection

Split samples measure variability between analysts, methods, and/or laboratories and are obtained as subsamples of a single sample. Duplicate samples measure variability inherent in the collection method or waste stream and are obtained during the same sampling event.

##### 1.7.7.1 Split Sample Collection

1. Collect sufficient sample in a container constructed of appropriate materials.
2. Preserve as necessary.
3. Mix well.
4. Alternately, decant 10% of the sample volume into each container and mix well.

5. Continue until each container is filled with an adequate sample volume.
6. Seal the containers, assign a field number, and complete the chain of custody.

#### 1.7.7.2 Duplicate Sample Collection

1. Collect two samples in rapid succession into separate containers.
2. Preserve as necessary.
3. Mix well.
4. Seal the containers, assign a field number, and complete the chain of custody.

#### 1.7.8 Drinking Water Sampling

##### 1.7.8.1 General Concerns

The containers and preservatives must be selected prior to sampling. These shall comply with Federal, State and Local guidelines. It is recommended that the appropriate preservative be added to the container by the laboratory.

##### 1.7.8.2 Sampling Drinking Water Wells

Purging and sampling should be from a spigot closest to the wellhead. (The spigot should be before the holding tank and filters. If this is not possible, the holding tank must also be purged), All aerators and filters should be removed if possible. Depending on the running schedule of the well and the placement of the pressure tank, the system will be purged as described in Section 1.7.5. If volume of the pressure tank is not known, the well is purged for at least 15 minutes at maximum rate. The flow is reduced to approximately 500 ml/minute.

Sample containers with no preservatives:

- a. The interior of the cap or the container should not be exposed to anything.
- b. The sample container is rinsed and the water is discarded.
- c. Containers are not rinsed if collecting for oil and grease, total recoverable hydrocarbons, volatile organics (including trihalomethanes) or microbiologicals.
- d. The container should be tilted to minimize agitation.

Sample containers with preservatives:

- a. The above protocol is followed but **DO NOT** rinse the container.
- b. The open end of the container should be held away from the face while filling.
- c. The container should be gently tipped several times to mix the preservatives
- d. Place the bottle in a plastic bag and cool to 4° C.

1.7.8.3 Sampling Drinking Water for the Lead/Copper Rule

1. The sampling point is dependent on if the sample is being taken to monitor compliance with Drinking Water Regulations for Lead and Copper. If so, the sample must be taken from a cold-water tap in the kitchen or bathroom of residential housing or from an interior tap where water is used for consumption in a non-residential building.
2. Samples must be collected after the water has stood in the pipes for at least six hours.
3. **THE SYSTEM SHOULD NOT BE FLUSHED.**
4. The first flush should be collected immediately into the sample container. **DO NOT RINSE THE CONTAINER PRIOR TO COLLECTING THE SAMPLE.**
5. The container should be tilted to minimize agitation.
6. If the container contains preservative, hold the open end away from the face.
7. Add preservative as needed.
8. Replace cap and gently tip the container several times to mix the preservatives.
9. Place in a plastic sample bag.

1.7.8.4 Drinking Water Supply Sampling

1. When sampling for compliance, the sampling point is normally designated by permit or the municipality.
2. Each sample shall have stood in the line for at least six hours. Samples shall be collected in one of the following ways:
  - a. At the tap, after flushing the volume of water between the tap and the lead service line. The volume of water shall be calculated based upon the inner diameter and length of the pipe between the tap and the service line.
  - b. By tapping directly into the service line.

- c. In a single-family residence, allow the water to run until a significant temperature change indicates water standing in the service line is being sampled.
3. The flow shall be reduced to less than 500 ml/min before collecting samples.
4. Test for the presence of residual chlorine using starch iodide paper or a Hach DR-100 chlorine analyzer.
5. If residual chlorine is present and the parameter being analyzed requires removal of chlorine, collect the sample in the appropriate sample container(s) using the required preservatives.
  - a. Add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$  or 100 mg of  $\text{Na}_2\text{S}_2\text{O}_3$  per 1 liter of sample water directly into the sample container.
  - b. After replacing the cap, tip the container several times to mix the preservative.

## 1.8 SOIL SAMPLING

Soil samples are preserved as per Section 1.12. When compositing subsamples, the quantity of each subsample used shall be measured and recorded in the field logbook.

### 1.8.1 Sampling Equipment

Hand Auger  
Encore™ Sampler  
Split Spoons  
Trowel  
Spoons  
Shovel  
Mixing

### 1.8.2 Hand Auger Sampling Procedure

This procedure is used in when only relatively shallow samples are required or in instances where use of heavy equipment is not justified. The hand auger may be used to collect samples of soils or other materials at various depths by adding extensions as necessary.

1. Remove surface debris from the location of the sampling hole using a clean shovel or spoon.
2. Disturbed portions of soil should be discarded and not taken as part of the sample.
3. Using a clean auger, drill to the desired sample depth. Confirm depths using a tape measure or other appropriate device.
4. Use a clean planer auger to clean and level the bottom of the boring.

5. All grab samples should be mixed thoroughly prior to containerization (except VOCs).
6. Using a clean auger, extract the desired sample. Subsampling is performed for VOC sample collection using an Encore™ sampling device. Once the core sample is collected, additional samples should be taken using an Encore™ sampler, either 5g or 25g, capped, sealed, and immediately cooled to 4°C. The holding time for this method is 48 hours. Alternatively, weigh 5g of sample into a pre-weighed vial (with a Teflon lined screw cap) containing, 5ml sodium bisulfate solution and a magnetic stir bar, cap, and then ice to 4°C. The holding time for this method is 14 days.
7. If less than the collected volume of material is desired or if multiple containers are required, subsampling shall be conducted. The collected material shall be placed in a clean mixing pan and thoroughly mixed using a clean, stainless steel spoon. The mixed material will then be quartered, removed and recombined before samples are collected. For clay soils, aliquots representative of the entire sample should be removed from the auger using stainless steel spoons. Samples for chemical analyses shall not be collected from auger flights or cuttings from hollow stem auger flights. Samples used for vapor meter determinations will not be used for trace contaminant analyses.
8. Samples should then be labeled. The depth range from which the samples were taken should be included in the sample description.
9. Repeat steps (2) through (6) as necessary to obtain samples at all desired depths.
10. When preparing composite samples, the quantity of each subsample shall be measured and recorded in the field logbook.

### 1.8.3 Split and Duplicate Sample Collection

Split samples measure variability between analysts, methods, and laboratories and are taken as subsamples from a single sample. Duplicate samples are collected simultaneously to measure variability inherent in the sampling method. True split samples are difficult to collect for soil, sediment and sludge under field conditions. Split samples for these materials are therefore considered duplicate samples. The collection procedure is as follows:

1. Collect the appropriate volume of sample into a clean disk constructed of a non-reactive material.
2. Mix the material with a clean utensil and separate into 4 to 10 equal portions.
3. Alternate placing a portion of the subdivided material into each container.
4. Repeat until each container is filled.
5. Assign each container a field sample number and complete the chain of custody.

## **1.9 WASTE SAMPLING**

### 1.9.1 Sampling Equipment

Shovel  
Split Spoons  
Trowel  
Spatula  
Spoons  
Drum Pump  
Coliwassa tubes

### 1.9.2 General

This section discusses the collection of samples from drums, tank trucks, and storage tanks, and samples from waste piles and landfills. Sampling of closed containers is considered a hazardous operation by all EMServices personnel.

#### 1.9.2.1 Specific Quality Control Procedures for Sampling Equipment

Sampling equipment used during waste sampling must be cleaned as specified in Section 1.10 of this manual before being returned from the field to minimize contamination. Contaminated disposable equipment must be disposed of as specified in the sampling plan. All field equipment shall be cleaned and repaired before being stored at the conclusion of a field study. Special decontamination procedures may be necessary in some instances and will be developed on a case-by-case basis. Any deviation from standard cleaning procedures and all field repairs shall be documented in field logbooks. Equipment that has not been properly cleaned must be tagged and labeled.

#### 1.9.2.2 Collection of Supplementary Information

The collection of supplementary data is important when collecting waste samples. Any field analyses shall be recorded in field logbooks. Sketches of sampling locations and layout shall be documented in the logbooks. Photographs shall be used extensively.

### 1.9.3 Open and Closed Container Sampling

#### 1.9.3.1 General

When sampling containers, open containers should be sampled first since they generally present less hazard than closed containers. Closed containers must be considered as extremely hazardous. Because of the

dangers involved with container sampling, the sampling of drums or other containers containing either unknown materials or known hazardous materials shall be considered a hazardous duty assignment.

One problem with container sampling is stratification and/or phase separation. Care must be taken to ensure that the sample collected is representative. If only one layer or phase is sampled, this should be noted when interpreting analytical results. If no stratification is present, representative samples may be composited by depth. When a drum or cylindrical container is standing vertically, depth compositing provides a good quantitative estimate of the containers contents. In other cases where containers are tipped, horizontal, deformed, etc., and stratification may not be present, vertical compositing will at least provide a qualitative sample.

#### 1.9.3.2 Sampling Equipment

The following equipment is available for use in collecting waste samples: barrel bung wrenches, adjustable wrenches, etc.; coliwassa samplers for drum sampling; and peristaltic pumps for liquid waste sampling from containers.

#### 1.9.3.3 Sampling Techniques

Containers with unknown materials or known hazardous materials shall be opened using only spark proof opening devices from a grounded container. The coliwassa sampler is a single use glass sampler, consisting of an outer glass tube with one end tapered, and a separate inner glass tube with a small bulb on one end. The outer tube is slowly lowered into the drum, tapered end first. Slowly lowering the tube allows the liquid phases in the drum to remain in equilibrium. The inner glass tube is inserted into the outer tube. After both inner and outer tubes are inserted into the drum to be sampled, the inner tube bulb end is pressed gently against the tapered end of the outer tube, forming a seal. Both tubes are withdrawn from the drum and the ends of the tubes are held over the sample container.

Drum samples can also be collected using a length of glass tube (1/2-inch or less inside diameter). The tube is inserted into the drum as far as possible and the open end is sealed to hold the sample in the tube. The sample is then placed in the appropriate container. Sample volumes shall be the absolute minimum required. Tank truck and storage tank samples may be collected from access ports on top of these tanks or trucks using the above techniques. Tank trucks are often compartmentalized, and each compartment should be sampled. Sampling from discharge valves is not recommended due to stratification possibilities and possibilities of sticking or broken valves. If the investigator must sample from a discharge valve,

the valving arrangement of the particular tank truck being sampled must be clearly understood to ensure that the contents of the compartments of interest are sampled. The investigator must realize that samples obtained from valves may not yield representative samples. If stratification or phase separation of waste samples is suspected, the sample collected should be representative of container contents. Samples should be depth composited when possible and number and types of layers shall be noted when interpreting analytical results.

#### 1.9.4 Waste Piles and Landfills

##### 1.9.4.1 General

Waste piles that consist of sludges and other solid wastes, liquid wastes mixed with soil or slag, or any type of wastes mixed with construction debris, household garbage, etc. The sampling personnel must be aware that landfills were not and are often still not selective in the types of materials accepted. Sampling at landfills could involve sampling operations that are potentially dangerous to sampling personnel.

##### 1.9.4.2 Sampling Locations

Sampling locations should be selected that will yield a representative sample of the waste. Exceptions are situations in which representative samples cannot be collected safely or when the team is purposely determining worst-case scenarios.

###### 1.9.4.2.1 Waste Piles

A representative sample from a small waste pile can be obtained by collecting a single sample. Collecting representative samples from large waste piles requires a statistical approach in selecting both the numbers of samples and sample location. A discussion of statistical methods is outlined in the Test Methods for Evaluating Solid Waste (SW-846) issued by the EPA Office of Solid Waste and Emergency Response.

###### 1.9.4.2.2 Landfills

Representative samples from landfills are difficult to due to the heterogeneous nature of the wastes. A statistical approach should be used in selecting both the number of samples and the sample location. Statistical methods are given in Test Methods for Evaluating Solid Waste (SW-846) issued by the EPA Office of Solid Waste and emergency Response. Landfills often generate

leachate at one or more locations down gradient of the fill material that can provide some insight of the materials in a landfill that are migrating via groundwater.

#### 1.9.4.3 Sampling Techniques

All samples collected should be placed into a Pyrex® or aluminum-mixing pan and mixed thoroughly. Samples for volatile organic compounds analyses must not be mixed or composited. Stainless steel spoons or scoops should be used to clear away surface materials before samples are collected. Near surface samples can then be collected with a clean stainless steel spoon. Depth samples can be collected by digging to the desired depth with a carbon steel shovel or scoop and removing the sample with a stainless steel spoon.

### **1.10 STANDARD CLEANING PROCEDURES**

#### 1.10.1 General

##### 1.10.1.1 Introduction

Procedures outlined in this section are used by EMServices personnel in cleaning field equipment prior to use. Ideally, a sufficient amount of clean equipment is carried to the field so that the project can be conducted without the need for field cleaning. This will not always be the case. EMServices policy regarding cleaning field equipment is as follows:

1. Equipment to be used in the field must be thoroughly cleaned in a controlled environment using prescribed procedures. This minimizes the potential for contaminants being transferred to equipment, vehicles, and the laboratory.
2. All equipment will be rinsed immediately with tap water after use, even if it is to be field cleaned for other sites.
3. If equipment is used only once (i.e., not cleaned in the field), it will be tagged in the field and transported separate from clean equipment and labeled as dirty or contaminated equipment.
4. All cleaning procedures shall be documented. In-field decontamination shall be documented in the field records. These records will specify the type of equipment cleaned and the specific protocols that are used. In-house cleaning records must identify the type of equipment, date it was cleaned, SOP used, and person that cleaned it.

5. Unless justified through documentation (i.e., company written protocols and analytical records) and historic data (i.e., absence of analytes of interest in equipment blanks), the protocols in Sections 1.10.1.2 through 1.10.7.9 shall be followed without modification.
6. All field sampling equipment shall be pre-cleaned in-house prior to arrival onsite unless otherwise justified.

#### 1.10.1.2 Cleaning Materials

Laboratory detergent is a phosphate-free, laboratory detergent such as Liquinox®. The use of any other detergent is noted in field logbooks and summary reports. A 10% nitric acid solution shall be made from reagent-grade nitric acid and deionized water. Standard cleaning solvent used will be pesticide-grade isopropanol. Other solvents (acetone and/or hexane) may be substituted as necessary. The use of other solvents must be documented in field logbooks and summary reports. Tap water may be used from any potable water system. Untreated water is not an acceptable substitute for tap water. Deionized water is tap water that has been passed through a deionizing resin column and should contain no inorganic compounds at or above analytical detection limits. Organic-free water is tap water that has been de-ionized and treated with activated carbon. Organic-free water should contain no detectable levels of organic compounds, and less than 5 ug/l of VOCs.

Analyte-free water is water in which all the analytes of interest and all interferences are below the method detection limits. Analyte-free water is always used for blank preparation and for the final in-house decontamination rinse. Substitution of higher-grade water (i.e., deionized or organic-free water for tap water) is permitted and need not be recorded. Solvent, nitric acid, detergent, and rinse water used to clean equipment shall not be reused.

#### 1.10.1.3 Marking Clean Equipment

Equipment that is cleaned by these methods shall be marked with the date and time that the equipment was cleaned.

#### 1.10.1.4 Marking Contaminated or Damaged Field Equipment

Field equipment that needs repair will be tagged and repairs or symptoms noted on the tag. Field equipment that needs cleaning **will not** be stored with clean equipment. All wrapped equipment not used in the field may be placed back in stock after equipment is inspected to ensure that contamination has not taken place.

#### 1.10.1.5 Decontamination of Equipment Used With Hazardous Waste

Equipment used to collect hazardous or toxic wastes or materials from hazardous waste sites, RCRA facilities, or in-process waste streams shall be decontaminated prior to leaving the site. This decontamination procedure shall consist of washing with laboratory detergent and rinsing with tap water. Procedures that are more stringent may be required depending on the waste sampled.

If equipment is heavily contaminated, an acetone or acetone/hexane/acetone pre-rinse may be necessary prior to regular decontamination procedures. It is not recommended that this type of cleaning be performed in the field.

#### 1.10.1.6 Disposal of Cleaning Materials

See Section 1.14.

#### 1.10.1.7 Safety Procedures for Cleaning Operations

All applicable safety procedures shall be followed during cleaning operations. The following precautions shall be taken during cleaning operations:

- Safety glasses or goggles, gloves, and protective clothing will be worn during all cleaning operations.
- Solvent rinsing operations will be conducted under a hood or in an open, well-ventilated area.
- No eating, smoking, drinking, chewing, or hand to mouth contact shall be permitted during cleaning operations.

#### 1.10.1.8 Storage of Field Equipment

All clean field equipment shall be stored in a designated, contaminant-free area.

### 1.10.2 Quality Control Procedures for Cleaning

#### 1.10.2.1 General

This section establishes quality control methods to monitor the effectiveness of the equipment cleaning procedures. The results of these methods will be monitored by the EMServices Quality Assurance Officer (QAO). All quality control procedures are recorded in a logbook and maintained in a quality assurance file. If contamination problems are

detected, the EMServices QAO shall determine the cause(s) of the problem(s) and take immediate corrective action.

#### 1.10.2.2 Rinse Water

The quality of water used shall be monitored once per quarter by placing water in standard, precleaned, sample containers and submitting them to a laboratory for analysis. Organic-free water will also be submitted for analyses of the various organic compounds.

#### 1.10.3 Procedures for Cleaning Teflon® or Glass Equipment Used in the Collection of Samples for Trace Organic Compounds and/or Metals Analyses

1. Equipment will be washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove materials are on the equipment, an acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.
2. Rinse the equipment with hot tap water.
3. Rinse equipment with a 10 percent nitric acid solution. If nitrogen-containing compounds are of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse. If necessary, the equipment can be soaked in fresh nitric acid solution.
4. Rinse equipment with tap water.
5. Rinse equipment with deionized water.
6. Rinse equipment twice with solvent and allow to dry.
7. If equipment cannot be cleaned effectively, discard of properly.
8. Wrap equipment in aluminum foil. Seal in plastic and date.

#### 1.10.4 Procedures for Cleaning Stainless Steel or Metal Sampling Equipment Used in Trace Organic and/or Metals Sample Collection

1. Equipment will be washed with laboratory detergent and hot water using a brush to remove any particulate matter or surface film. If oil, grease, or other hard to remove materials are on the equipment, an acetone/hexane/acetone pre-wash and/or steam cleaning may be necessary.
2. Rinse equipment with hot tap water.
3. Rinse equipment with deionized water.
4. Rinse equipment twice with solvent and allow to dry.
5. If equipment cannot be cleaned effectively, discard of properly.
6. Wrap equipment in aluminum foil. Seal in plastic and date.

#### 1.10.5 Cleaning Procedures for Automatic Sampling Equipment

##### 1.10.5.1 Automatic Wastewater Samplers

1. The exterior and accessible interior portions of automatic samplers will be washed with Liquinox and rinsed with tap water.
2. The electronics casing will be cleaned with a clean damp cloth.
3. All vinyl sample tubing will be discarded after each use.
4. Teflon® tubing will be cleaned using procedures found in Section 1.10.1.2
5. Silastic pump tubing will be cleaned and re-used after each use if possible. Tubing will be cleaned using cleaning procedures specified in Section 1.10.1.1 of this document. Tubing shall be checked on a regular basis and will be changed if it has become discolored or has lost its elasticity.

#### 1.10.5.2 Reusable Glass Composite Sample Containers

1. If containers are used to collect samples that contain hard to remove materials (i.e., oil and grease), it is rinsed as necessary with reagent grade acetone prior to the detergent wash. If material cannot be removed, the container is discarded.
2. Wash containers thoroughly with hot tap water and Liquinox and rise thoroughly with hot tap water.
3. If metals are to be sampled, rinse with 10% nitric acid. If nutrients are to be sampled, follow with a 10% hydrochloric acid rinse.
4. Rinse thoroughly with tap water.
5. Rinse thoroughly with DI water.
6. If organics are to be sampled, rinse twice with isopropanol and allow to air-dry for 24 hours or more. Cap the container with the decontaminated Teflon® lined lid.
7. After use, rinse with tap water in the field and cover to prevent drying of material onto the interior surface.
8. Containers that have a visible scale, film, or discoloration after cleaning or that were used at a chemical manufacturing facility should be properly discarded at the conclusion of the sampling activities.

#### 1.10.5.3 Reusable Plastic Composite Sample Containers

1. Wash containers with hot tap water and laboratory detergent, using a bottlebrush to remove particulate matter and surface film.
2. Rinse containers with hot tap water.
3. Rinse containers with 10 percent nitric acid. If nitrogen-containing compounds are of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse containers with tap water.
5. Rinse containers with deionized water.
6. Cap with aluminum foil.

7. Plastic sample containers used at facilities that produce toxic compounds will be properly disposed of at the conclusion of the sampling activities. Containers that have a visible film, scale, or other discoloration remaining after cleaning will be discarded.

#### 1.10.5.4 Plastic Sequential Sample Bottles for Automatic Sampler Base

1. Rinse bottles in field with potable or de-ionized water when possible.
2. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and deionized water rinse cycles. Alternatively, hand wash using the same procedures.
3. Rinse with 10 percent nitric acid. If nitrogen-containing compounds are of concern, hydrochloric acid must be used as a substitute or subsequent equipment rinse.
4. Rinse with tap water.
5. Replace bottles in sampler base; cover with aluminum foil before storing.

#### 1.10.6 Cleaning Procedures for Sampling Tubing

##### 1.10.1.1 Silastic Rubber Pump Tubing Used In Automatic Samplers

Silastic pump tubing used in automatic samplers need not be replaced in pumps where the sample does not contact the tubing, where the sampler is being used solely for purging purposes (i.e., not being used to collect samples). Tubing must be changed on a regular basis if used for sampling purposes and should be cleaned in the following manner:

1. Flush tubing with laboratory grade detergent and hot tap water.
2. Rinse thoroughly with hot tap water.
3. Rinse thoroughly with DI water.
4. If used to collect metals samples, the tubing shall be flushed with 1+5 nitric acid, followed by a thorough rinsing with DI water.
5. Install the tubing in the automatic wastewater sampler.
6. Cap both ends with aluminum foil or equivalent.

Tubing should always be replaced at automatic sampler manufacturer's recommended frequencies. If tubing cannot be adequately cleaned, it shall be discarded.

##### 1.10.1.2 Teflon® Tubing

New Teflon® tubing shall be pre-cleaned as follows:

1. Rinse outside of the tubing with pesticide-grade solvent.
2. Flush interior of the tubing with pesticide-grade solvent.
3. Let dry overnight in drying oven or equivalent.
4. Wrap tubing in aluminum foil and seal in plastic.

Reused tubing shall be transported to the field in pre-cut and pre-cleaned sections. Field cleaning of Teflon® is not recommended. The following steps prescribe cleaning procedures:

1. Exterior of tubing must be cleaned first by soaking in hot, soapy water in a stainless steel or non-contaminating sink. Particulate may be removed with a brush.
2. Clean inside of tubing ends with a small bottlebrush.
3. Rinse surfaces and ends with tap water.
4. Rinse surfaces and ends with nitric acid, tap water, isopropanol, and analyte-free water.
5. Place on fresh aluminum foil; connect all sections with Teflon® couplings.
6. Cleaning configuration:
  - a. Cleaning solutions are placed in a clean, 2-liter glass jar.
  - b. Place one end of tubing in the solution, the other in the **INFLUENT** end of a peristaltic pump.
7. Effluent from the pump can be recycled through the glass cleaning solution jar. All cleaning solutions can be recycled EXCEPT the final isopropanol and analyte-free water rinses.
8. The above configuration is used as follows:
9. Pump generous amounts of hot, soapy water through the tubing.
10. Follow this with tap water, 10% nitric acid, tap water, isopropanol, and analyte-free water.
11. The nitric acid and isopropanol rinses should be allowed to remain in the tubing for 15 minutes with the pump shut off then continue with subsequent rinses
12. Leave any couplings in and connect or cover the remaining ends.
  - a. After cleaning the interior, rinse the exterior with analyte-free water.
  - b. The cleaned lengths are wrapped in aluminum foil and stored in a clean, dry area until use.

#### 1.10.7 Field Equipment Cleaning Procedures

##### 1.10.7.1 General

It is the responsibility of field personnel to properly clean equipment in the field. The following procedures shall be observed when cleaning equipment in the field.

#### 1.10.7.2 Conventional Equipment Use

Remove deposits with a brush if necessary. If only inorganic anions are of interest, equipment should be rinsed with analyte-free water and with the sample at the next sampling location prior to collection. Clean equipment for the collection of samples for organic compounds or trace inorganic analyses according to Section 1.10.7.3.

#### 1.10.7.3 Equipment Used to Collect Organic Compounds and Trace Metals Samples

1. Clean with tap water and laboratory detergent. If necessary, use a brush to remove particulate and surface films.
2. Rinse with tap water.
3. Rinse with 10 to 15 percent nitric acid solution followed by 10 percent hydrochloric acid rinse (unless equipment is made of metal).
4. Rinse with tap water.
5. Rinse with deionized water.
6. Rinse twice with solvent.
7. Rinse with organic-free water and allow to air dry.
8. If organic-free water is unavailable, let air dry. Do not rinse with deionized or distilled water.
9. Wrap with aluminum foil or plastic.

#### 1.10.7.4 Teflon®, Glass, Stainless Steel or Metal Equipment Used to Collect Samples for Metal Analyses

1. Remove particulate matter and surface films. Clean with laboratory detergent and tap water.
2. Rinse with tap water.
3. 10 percent nitric acid solution (skip 3 and 4 if equipment is made of metal and/or stainless steel).
4. Rinse with tap water.
5. Rinse with deionized water.
6. Let air dry.

#### 1.10.7.5 Instruments Used to Measure Groundwater Levels

Wash with laboratory detergent and tap water.  
Rinse with tap water.  
Rinse with deionized water.  
Allow to dry.

#### 1.10.7.6 Field Filtration Apparatus

A new, disposable filtration unit will be used for each site. Filter pore size will be dependent on parameter being monitored as per Section 1.7.1. The peristaltic pump is cleaned as described in Section 1.10.7.7. Silastic pump tubing will be cleaned as described in Section 1.10.1.1. If Teflon® tubing is used; it will be cleaned as described in Section 1.10.1.2. Other tubing types must be cleaned following the appropriate regimen described in Section 1.10.1. In general, non-Teflon® type tubing (e.g., HDPE) will not be re-used.

#### 1.10.7.7 Flow Meters, Above Ground Pumps, Bladder Pumps and Other Field Instrumentation

The exterior of equipment such as flow meters should be washed with a mild detergent and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth. Other field instrumentation should be wiped with a clean, damp cloth. Meter probes should be rinsed with deionized water before storage. Equipment desiccant should be checked and replaced as necessary. Peristaltic pumps used for purging must be free of oil and grease on the exterior. Peristaltic pumps used for sampling must be cleaned on the outside with Liquinox and rinsed with tap water followed by DI water.

#### 1.10.7.8 In-Field Decontamination for Submersible Purging Pump and Tubing

EMServices uses the submersible bladder pump listed in Section 1.7.1 for purging and sample collection. The pump and tubing shall be decontaminated between wells in the following manner:

1. Interior of the pump and tubing shall be thoroughly flushed with a soapy water solution.
2. Wipe or scrub the exterior of the pump and tubing as necessary with the appropriate soap solution.
3. Rinse exterior and interior of pump and tubing thoroughly with tap water followed by a deionized water rinse.
4. Allow remaining water to drain from tubing and pump and allow to air dry as long as possible in a contaminant free area as long as possible before purging the next well.

#### 1.10.7.9 Shipping Containers

All reusable shipping containers shall be washed with laboratory detergent, rinsed with tap water, and air dried before storage or re-use. Extremely contaminated shipping containers shall be cleaned as thoroughly as possible and properly disposed.

#### 1.10.7.10 Analyte Free Water Containers

Analyte-free water containers can be made of glass, Teflon®, polypropylene, or high-density polyethylene (HDPE). Inert glass or Teflon® is recommended for holding organic-free sources of water. Polypropylene can be used when organics are not analytes of concern. HDPE is not normally recommended but is acceptable for use. Water should not be stored in these containers for extended periods. Containers of water should only be used for a single event and should be disposed of at the end of the sampling day. The procedure for cleaning analyte-free water containers is as follows:

1. For new containers, follow instructions in Section 1.10.3 of this manual. Delete the solvent rinse if containers are made of plastic.
2. Cap with Teflon® film, aluminum foil, or the Teflon® lined bottle cap (aluminum foil or Teflon® film may also be used as a cap liner).

If water is being stored in reused containers, the following cleaning procedures should be followed:

1. After emptying, cap the container. Wash exterior of the container with Liquinox and rinse with DI water.
2. Rinse the interior twice with isopropanol unless the container is made of plastic.
3. Rinse the interior thoroughly with analyte-free water.
4. Invert and allow to dry.
5. Fill the container with analyte-free water and cap with aluminum foil, Teflon® film, or a Teflon® lined bottle cap.
6. Water shall not be stored prior to a sampling event for more than 3 days.

#### 1.10.7.11 Vehicles

Field vehicles used by EMServices personnel should be washed at the conclusion of each field-sampling event. This should reduce the risk of

contamination due to transport on a vehicle. When vehicles are used at hazardous waste sites or on studies where pesticides, herbicides, organic compounds, or other toxic materials are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of the site visit. Vehicles are equipped with trash containers. EMServices personnel are responsible for cleanliness of each vehicle.

## **1.11 SAMPLE HISTORY**

Sample chronology is recorded and kept on the EMServices chain of custody, field log.

## **1.12 SAMPLE CONTAINERS, PRESERVATION METHODS AND HOLDING TIMES**

### 1.12.1 General Considerations

For information pertaining to sample containers, preservation methods, and holding times, Refer to SW-846, Table II-1 and Chapter 3, Page 3 for solid waste and RCRA projects and 40 CFR Part 136, Table II for water and wastewater projects.

The provisions of 40 CFR Part 136, Table II shall take precedence over requirements given in any approved method when sampling in the State of Florida for water and wastewater. Proper sample preservation is the responsibility of the sampling team and it is their responsibility to assure that all samples are preserved according to 40 CFR Part 131. For the purposes of this manual, "immediately" will be defined as within 15 minutes. Sample preservation is accomplished either by obtaining pre-preserved containers from an acceptable source or by adding preservatives in the field. It is the responsibility of the field team accepting pre-preserved containers to make sure that the proper preservatives are used and desired results are achieved. The laboratory shall also supply additional preservatives from the same source in suitable containers.

### 1.12.2 Sample Preservation

The following protocols apply for sample containers preserved in the field after sample has been added:

1. Preservatives shall be at least reagent grade or higher. The acid for metals shall be suitable for trace metals analyses.
2. Fresh preservatives shall be obtained prior to each sampling event. Remaining preservatives that are not sealed must be discarded in an acceptable manner.
3. Preservatives are transported in pre-measured glass ampules and added directly to the sample.

4. A corresponding amount of preservative shall be added to associated equipment blanks.
5. The pH is checked on all pH-preserved samples with the exception of VOC, oil and grease, and TRPH. Effectiveness of pH adjustment is made in the following manner:
  - a. Narrow range pH paper is used to test a small aliquot of the preserved sample.
  - b. A small portion of sample is placed into a container, checked with pH paper, and compared against color chart.
  - c. Discard the aliquot properly, but do not pour back into the sample container.
  - d. If pH is acceptable, document in field log and prepare for transport to laboratory

If pH is unacceptable, continue to add additional preservative in measured increments using the methods described above until an acceptable pH has been reached. Record the total amount of preservative used in the field log. Always use additional preservative from the same source as the initial preservation attempt. In some cases, an extra dummy sample can be used to test pH preservation on. Content should be suitably discarded.

If equipment blanks or field blanks are used, the maximum amount of preservative that was used to preserve any single sample in the set shall be added to the equipment or field blank. Samples that need to be preserved at 4°C shall be cooled. The cooler shall be checked to ensure that the ice has not melted. A temperature surrogate bottle will be placed in each cooler to verify that required sample preservation temperature is maintained within acceptable limits.

#### 1.12.3 Sample Containers

EMServices does not clean and re-use sample containers. EMServices receives all sample collection containers from commercial laboratories as precleaned containers. All sampling containers are discarded after use. Cleaning grades of all containers meet EPA analyte specific requirements.

#### 1.12.4 Field Reagent Handling

Reagents, cleaning materials, and preservatives that are maintained by a field team will be stored transported and handled in such a way as to prevent and/or minimize contamination.

The following storage and use protocols will be observed:

1. Chemicals will be stored in-house and transported to the field segregated according to reactivity.
2. Acids are stored in an acid storage cabinet and solvents are stored in a vented, explosion proof solvent storage cabinet.
3. All chemicals transported to the field are stored in bottles and packed to avoid breakage.
4. When reagents are transferred from an original container, the transport container must be pre-cleaned and of compatible material as the original container.
5. Chemicals shall be separated from sample containers and samples to avoid reaction and possible contamination.
6. Analyte free water shall be segregated from solvents to prevent contamination.

#### 1.12.5 Sample Transport

In the majority of situations, samples will be delivered directly to the laboratory by the field sampling team or field courier following standard chain of custody protocols. Samples will be preserved immediately (i.e., within 15 minutes) and packed with ice prior to transport. The field team will relinquish custody to the log-in sample custodian upon arrival at the laboratory. Certain situations will require that the field sampling team ship samples to the laboratory utilizing common carrier (UPS, FEDEX). If samples are sent by common carrier, all documentation (transmittal form, chain of custody, field data, analyses request, etc.) shall be placed in a zip lock bag and placed inside the sample container. The container is then sealed closed and sent to the laboratory in the required time frame to meet requirements of time sensitive analyses (BOD, hexavalent chromium).

### **1.13 SAMPLE DISPATCH**

Samples collected during field investigations or in response to a hazardous materials incident must be classified by the project manager, prior to shipping, as either environmental or hazardous material samples. The shipment of samples designated as environmental samples are not regulated by the U.S. Department of Transportation. Samples collected from certain process streams, drums, bulk storage tanks, soil, sediment, or water samples from suspected areas of high contamination may need to be shipped as hazardous. These regulations are promulgated by the US-DOT and described in the Code of Federal Regulations (49 CFR 171 through 177). The guidance for complying with USDOT regulations in shipping environmental laboratory samples is given in the "National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

#### 1.13.1 Shipment of Environmental Samples

Samples packaged for shipment by EMServices shall be segregated by sample type, preservation requirements, and potential contaminant level. During sampling events in which large numbers of samples will be collected, samples will also be segregated by analyses to be conducted. If multiple sites are to be sampled, or if specific and separate areas of interest can be identified, samples will be further segregated for packaging prior to shipment. Environmental samples shall be packed prior to shipment using the following procedures:

1. Select a cooler (clean and strong). Line the cooler with a large heavy-duty plastic bag.
2. Allow sufficient headspace (except VOCs or others with zero headspace requirements) to compensate for any pressure and temperature changes.
3. Be sure the lids on all bottles are tight.
4. Place all bottles in appropriately sized polyethylene bags.
5. Place VOC vials in foam material transport sleeves.
6. Place foam padding in the bottom of the cooler and then place the bottles in the cooler with sufficient space to allow for the addition of more foam between the bottles.
7. Put ice on top of and/or between the samples.
8. Place chain of custody in a baggie and into the cooler. Close the cooler and securely tape the top of the cooler shut. The chain of custody seals should be affixed to the top and sides of the cooler so that the cooler cannot be opened without breaking the seal.
9. The shipping containers must be marked "THIS END UP". The name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials are not permitted to be on the outside of the container used to transport environmental samples and shall not be used.

## **1.14 INVESTIGATION WASTE**

### **1.14.1 General**

Field surveys conducted by EMServices may generate waste materials. Some of these waste materials may be hazardous wastes that must be properly disposed in accordance with EPA regulations.

#### **1.14.1.1 Types of Investigation Derived Wastes (IDW)**

Materials that may fall under the IDW category are:

- Personnel protective equipment (PPE)
- Disposable sampling equipment (DE)
- Soil cuttings
- Groundwater obtained through well purging

- Spent cleaning and decontamination fluids
- Spent calibration standards

#### 1.14.1.2 Managing Non-hazardous IDW

Disposal of non-hazardous IDW should be addressed prior to initiating work at a site. Facility personnel should be consulted and wastes handled in an appropriate manner as directed by the client. For development and purge water generated in the State of Florida, specific disposal requirements apply. The water shall be contained on-site in temporary storage until it is characterized. Appropriate disposal and/or treatment methods will then be determined.

Possible disposal options are:

- Direct discharge on-site to infiltrate the same or a more contaminated source
- Transportation to an off-site facility

In no case shall the water be discharged into any surface water unless permitted.

#### 1.14.1.3 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW (as defined in 40 CFR 261.30-261.33 or displaying the characteristics of ignitability, corrosivity, reactivity, or toxicity) must be specified in the sampling plan. Hazardous IDW must be disposed in compliance with USEPA regulations. If appropriate, these wastes may be taken to a facility waste treatment system. These wastes may also be disposed of in the source area from which they originated, if state regulations allow this. If on-site disposal is not feasible, appropriate tests must be conducted to determine if the waste is hazardous. If yes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Weak acids and bases may be neutralized in lieu of disposal as hazardous wastes. Neutralized wastewaters may be flushed into a sanitary sewer. If possible, arrangements for proper containerization, labeling, transportation, and disposal/treatment of IDW should be anticipated beforehand. Investigation derived wastes should be kept to a minimum. Most of the routine studies conducted by EMServices should not produce any IDW that are hazardous. Many of the above PPE and DE wastes can be deposited in municipal dumpsters if care is taken to keep them segregated from hazardous waste contaminated materials.

Disposable equipment can often be cleaned to render it non-hazardous, as can some PPE, such as splash suits. The volume of spent solvent waste produced during equipment decontamination can be reduced or eliminated by applying only the minimum amount of solvent necessary.

## 1.15 SAMPLING BIBLIOGRAPHY

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, February 1, 1991, US EPA Region IV, Environmental Services Division.

RCRA Ground Water Monitoring Technical Enforcement Guidance Document (GPO#5500000260-6), US EPA, September 1981.

Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Office of Solid and Emergency Response, US EPA, November 1981.

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December 1988.

Florida Department of Environmental Regulation (DER) Quality Assurance Section (QAS) Guidance Documents:

#89-01 Equipment Material Construction, revised April 7, 1989

#89-02 Field QC Blanks, revised April 28, 1989

#89-03 Teflon/Stainless Steel Bladder Pumps, revised May 10, 1988

#89-04 Field Cleaning Procedures, revised August 10, 1989

DER Manual for Preparing Quality Assurance Plans, DER-QA-001/90, revised September 30, 1992.

NPDES Compliance Inspection Manual, United States Environmental Protection Agency, Enforcement Division, Office of Water Enforcement and Permits, EN-338, 1988.

Handbook for Monitoring Industrial Wastewater, United States Environmental Protection Agency, Technology Transfer, 1973.

EPA Primary Drinking Water Regulations, 40 CFR 141.

Rapid Bioassessment Protocols for Use in Streams and Rivers, United States Environmental Protection Agency, Office of Water, EPA/444/4-89-001, May 1989.

Environmental Sampling and Analysis: A Practical Guide. Lawrence H. Keith, Ph.D., 1991. Lewis Publishers.