

## ***FS 2000. GENERAL AQUEOUS SAMPLING***

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

### **1. COMMON PROCEDURES**

The following procedures are applicable to the collection of all water samples.

1.1. Refer to FS 1000 for procedures that are common to all types of sample collection including general preservation and thermal preservation procedures.

#### **1.2. Grab Samples**

1.2.1.1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15-minute time limit applies to aqueous samples only. No time limit applies to the collection of solid samples (e.g., residuals).

1.2.1.2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- A snapshot of the water quality at a particular instant in time is desired.
- The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow).
- The characteristics of the water or waste stream are known to be constant or nearly so.
- When conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- The sample is to be analyzed for analytes whose characteristics are likely to change significantly with time (e.g., dissolved gases, microbiological tests, pH).
- The sample is to be collected for analytes such as Oil and Grease, bacteriological tests or other parameters listed in number 3 of this section where the compositing process could significantly affect the actual concentration.
- Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.
- When identifying and tracking slug loads and spills.

1.2.1.3. If required, measure the following parameters on grab samples or in-situ.

NOTE: If the permit specifies a composite sample for any of the parameters mentioned below, **FOLLOW THE PERMIT CONDITIONS**

Cyanide	Oil and Grease
Residual Chlorine	pH
Dissolved constituents in field-filtered samples (ortho-phosphorus, metals, etc.)	Specific Conductance
Dissolved Oxygen and other dissolved gases	Un-ionized Ammonia
Microbiological Parameters	Volatile Organic Compounds
TRPHs	Temperature
Total Phenols	

### 1.3. Composite Samples

1.3.1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.

1.3.2. Composite samples are used when stipulated in a permit or when:

- The water or wastewater stream is continuous;
- Analytical capabilities are limited;
- Determining average pollutant concentration during the compositing period;
- Calculating mass/unit time loadings; or
- Associating average flow data to parameter concentrations

1.3.3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow rate or they may be collected proportional to the flow rate. The permit or work plan will specify which composite sample type to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

1.3.3.1. Time Composite Sample: Time composite samples are based on a constant time interval between samples. A time composite sample can be collected manually or with an automatic sampler. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample described in number 3.3 of this section.

1.3.3.2. Flow Proportional Composite Sample: Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. There are two methods used to collect this type of sample:

- Method 1: Collect a constant sample volume per stream flow (e.g., a 200 mL sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately.
- Method 2: Collect a sample by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

1.3.3.3. Sequential Composite Sample: Sequential composite samples are composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited for each hour.

- The 24-hour composite is made up from the individual one-hour composites. Each of the 24 individual samples is manually flow-proportioned according to the flow recorded for the hour that the sample represents. Each flow-proportioned sample is then added to the composite samples. The actual compositing of the samples is done by hand and may be done in the field or the laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to, and handled, in the laboratory. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.
- A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow
- Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period. For example, for pretreatment studies you can visually determine when high strength wastes are being discharged from a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure the pH throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals

1.3.3.4. Continuous Composite Sample: Collected continuously from the stream. The sample may be a constant volume that is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

1.3.3.5. Areal Composite: A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites must be made up of equal volumes of grab samples; each grab sample must be collected in an identical manner. Examples include residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc sample is similar to the flow proportional composite.

#### 1.4. Collection Techniques

1.4.1. When filling a sample container that already contains premeasured preservative, slowly pour the sample down the side of the container so that the preservative does not

splatter. If the preservative is concentrated acid, and the sample water is added too quickly, the reaction between the water and the acid can generate enough heat to burn unprotected skin or could splatter and cause acid burning.

1.4.2. Collect grab samples (single, discrete samples) unless directed by permit, program, or approved sampling plan or work plan to collect composite samples.

1.4.3. Except for volatile organic compounds and sulfide, leave ample headspace in the sample bottle to allow for expansion, effervescence and proper mixing at the laboratory.

#### 1.5. Collecting Filtered/Dissolved Samples

1.5.1. Certain studies or projects require collection of dissolved (i.e., filtered) samples. Identify all analytes in samples that are filtered as “dissolved” or “filtered” in field notes or laboratory transmittal forms and on final reports.

1.5.2. Collect both filtered and unfiltered samples from the same water in a collection device (e.g., bailer, intermediate container) or consecutively if sampling from a pump.

1.5.3. Collect dissolved metals in groundwater according to the procedures discussed in FS 2225. **Do not** collect filtered samples for metals from groundwater sources unless:

1.5.3.1. The DEP has required or approved the protocol and the DEP program allows the use of the procedure; or

1.5.3.2. The organization is documenting that a filtered groundwater sample is as or more representative of the groundwater quality. In this case, collect **both** unfiltered and filtered samples for analysis. Submit the results of both samples the DEP for review.

1.5.4. Filtration, when performed, must begin within 15 minutes of sample collection.

1.5.5. Collect dissolved groundwater samples for metals with a one-piece molded construction 1 µm filter unless otherwise specified by a DEP program. Use a 0.45 µm filter when filtering all other constituents **including** metals in surface water.

1.5.6. The filter must be compatible with the analyte to be filtered (e.g., zero carbon content for carbon analysis; non-protein binding filters for nitrogen).

1.5.7. Equipment blanks, when collected, must be processed through the filtration apparatus and analyzed for the analytes of interest.

1.5.8. Filters and filtration equipment are intermediate devices and therefore must be adequately rinsed per FS 2110 section 1.1.2.1.

#### THE FOLLOWING ARE SPECIAL CONSIDERATIONS FOR VARIOUS ANALYTE GROUPS:

#### **FS 2001.**     *pH-Preserved Samples*

##### 1. SAMPLE CONTAINERS

1.1. Use properly cleaned sample containers (see FC 1300).

1.2. Inspect all containers for visual defects or contamination. Discard if defects are present or containers do not appear clean.

##### 2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. If the preservative is added after the sample is collected, (the container is not prepreserved), do not fill the container to the rim.

### 3. PRESERVATION

3.1. Preserve the sample within 15 minutes of sample collection or filtration (if applicable) unless collected as a composite sample (see FS 1006, section 1.3) or for analysis of lead and copper for drinking water compliance (see FS 2310, section 2).

3.2. Preserve the sample with the chemical specified by the method or preservation tables (Tables FS 1000-4 to FS 1000-10).

3.2.1. The chemical reagents must be pure enough so that the reagent does not contribute contamination or interferences to the analytes of interest.

3.3. Preserve the sample by adding an accurately measured amount of preservative to the container. Premeasured vials of the preservative, or a graduated container or pipet, may be used.

3.3.1. Tightly cap the sample container and gently tip the container two to three times to distribute the chemical.

3.4. The pH of the preserved sample must meet the pH criterion of the applicable preservation tables (see Tables FS 1000-4 to FS 1000-10). **Do not over preserve the sample.**

3.4.1. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH meets the required level. **Do not put the pH paper directly into the sample container.**

3.4.2. If the pH does not meet the required level, add additional measured amounts of preservative and test with narrow range pH paper (see section 3.4.1 above) until the pH meets the pH requirement.

3.4.3. Record the total amount of preservative that was added to the sample. This documentation is necessary for the next site visit, since additional acid may be needed to adequately preserve the sample on subsequent visits.

3.5. Cooling to less than 6°C with wet ice (see FS 1006, section 5) may be required.

3.6. Protect from direct sunlight.

3.7. Preserve at least one of the equipment blanks with the **greatest** amount of preservative that was required in the sample set and note the amount in field documentation.

3.8. After the sample has been preserved, screw the cap on tightly.

4. Verifying pH-Preserved Samples: Verify the pH of all pH-preserved samples (except volatile organics) in the field (see FS 2001, section 3.4). If samples are routinely collected from the same sample location, a pH check is not required each time samples are collected.

4.1. If the frequency of sample collection at a specified location is once per month or greater (e.g., weekly or daily), check the pH of **at least one** sample per parameter group according to the following schedule:

- 4.1.1. Weekly sampling: 1 pH check per month
- 4.1.2. Daily sampling: 1 pH check per week
- 4.2. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.
  - 4.2.1. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.
  - 4.2.2. For all other sample collection frequencies, pH checks may be reduced as follows:
    - 4.2.2.1. During the first sampling event at a particular site, check **all** samples (except volatile organics) that are pH-adjusted, and
    - 4.2.2.2. During subsequent visits to a particular site, check **at least one** sample per parameter group that must be pH-adjusted.
5. DOCUMENTATION
  - 5.1. Complete the sample container label and stick firmly on the container.
  - 5.2. Complete the field notes.
  - 5.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment or preservation problems.

## **FS 2002.     *Metals***

1. SAMPLE CONTAINERS
  - 1.1. Use properly cleaned containers (see FC 1300).
  - 1.2. Inspect the containers and caps for visual defects or contamination. Do not use containers if defects are present or if they do not appear clean.
2. SAMPLE COLLECTION PROCEDURES
  - 2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.
  - 2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.
3. PRESERVATION - Follow preservation procedures outlined in FS 2001 above.
  - 3.1. Requirements for specific metals:
    - 3.1.1. For boron or cold-vapor atomic absorption Mercury with a grade of nitric acid (HNO<sub>3</sub>) that is suitable for use for metals analysis. Use concentrated HNO<sub>3</sub> or 1:1 HNO<sub>3</sub> to lower the pH of less than 2 S.U., but greater than 1.62 S.U.
    - 3.1.2. For Chromium VI add sufficient ammonium sulfate buffer solution specified per Table FS 1000-4 to the sample to raise the pH of the sample to a pH of 9.3 – 9.7 and place in ice (see FS 2002).
    - 3.1.3. Trace Level Mercury
      - 3.1.3.1. Collect samples for trace level mercury (<100 ug/L) in tightly-capped fluoropolymer or glass bottles.

3.1.3.2. If the samples cannot be received by the laboratory within 48 hours of sample collection, preserve the sample with BrCl or HCl solution.

3.1.3.3. For dissolved trace level mercury, samples must be filtered through a 0.45 µm filter within 24 hours of sample collection. If the samples cannot be transported to the laboratory within 24 hours, follow the procedures in FS 8200 for field filtration.

3.1.4. Samples collected for lead and copper for drinking water compliance and metals other than those listed above do not require immediate acid preservation.

3.1.4.1. When samples are not acidified with acid, the transmittal form to the laboratory must:

- Clearly state that the samples are unpreserved; and
- Request that the laboratory preserve the samples.

3.1.4.2. If samples are acidified, use concentrated HNO<sub>3</sub> or 1:1 HNO<sub>3</sub> to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.2. After the sample has been preserved, screw the cap on tightly.

#### 4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. On the transmittal form, clearly identify samples that must be acidified by the laboratory (FS 2002, 3.1.3 or 3.1.4 above).

### **FS 2003. *Extractable Organics***

#### 1. SAMPLE CONTAINERS

1.1. Most samples are collected in glass containers with Teflon-lined caps. Note: Teflon containers are also acceptable. There are some exceptions such as collecting samples in amber glass (e.g., nitroamines, nitroaromatics, etc.). If in doubt, verify the proper container type in Tables FS 1000-4 through FS 1000-10.

1.2. Inspect glass bottles to assure that there are no visual glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.

1.3. Collect composite samples from automatic sample collection devices in refrigerated glass or Teflon containers through Teflon, polyethylene or polypropylene tubing.

#### 2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container without touching the interior Teflon liner.

2.2. Carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. Fill bottle with sample to almost full capacity.

#### 3. PRESERVATION

- 3.1. In general, these types of samples must be preserved by cooling to 4°C.
  - 3.1.1. Some analyte groups require a chemical preservation. See Tables FS 1000-4 through FS 1000-10 for any additional preservation.
  - 3.1.2. If the samples for pesticides cannot be extracted within 72 hours of collection, the sample pH must be in the range of 5 to 9. If needed, adjust sample to the specified pH range with sodium hydroxide or sulfuric acid.
  - 3.1.3. Add sodium thiosulfate if residual chlorine is present.
- 3.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).
4. DOCUMENTATION
  - 4.1. Complete the sample container label and stick firmly on the container.
  - 4.2. Document when samples were placed in wet ice immediately (see FS 1006, section 5).
  - 4.3. Complete the field notes.
  - 4.4. Make notes on the lab transmittal form and the field records about any sample that appears highly contaminated or exhibits other abnormal characteristics (i.e., foaming, odor, etc.).

## **FS 2004.**     *Volatile Organics*

1. SAMPLE CONTAINERS
  - 1.1. Use a screw cap glass sample vial that is sealed with a Teflon-coated septum.
  - 1.2. Collect **at least two** vials of each sample. Some laboratories may require three or more vials, therefore verify the laboratory's policy on the number of vials they require unless the laboratory provides the sampling kit.
  - 1.3. Inspect the vials for glass or septum defects (e.g., rim must not have nicks or visible depressions and the septum must not be deformed). Do not use containers if defects are present or if they do not appear clean.
2. SAMPLE COLLECTION PROCEDURES
  - 2.1. Special precautions for petroleum sources:
    - 2.1.1. If possible, transport and store fuels in a separate vehicle from sampling equipment, empty vials and collected samples. If these items must be transported in the same vehicle as fuel, store the fuels as far away from the vials as possible.
    - 2.1.2. Place all fuel or exhaust sources downwind of the sampling location.
    - 2.1.3. Position all petroleum-fueled engines (including the vehicle) downwind of the sampling operations.
  - 2.2. Do not allow the sampling equipment or hands to touch the rim of the sample container.
  - 2.3. Do not remove septum caps from VOC vials until just prior to filling. Cap vials immediately after filling with sample.
  - 2.4. **DO NOT PRERINSE VOC VIALS.**



2.5. Do not aerate the sample during sample collection. If collecting from a spigot or pump, reduce the flow rate to less than 100 mL/min.

2.6. If preservation is required, proceed to section 3 below unless the laboratory supplied vials with premeasured quantities of acid, and the sample does not need to be dechlorinated (see 3.2 below).

2.6.1. If no preservation is required or if the vials are prepreserved (see 2.5 above), slowly and carefully allow the sample to flow down the **side** of the vial to minimize turbulence. Fill the vial until the surface tension holds the water in a "convex meniscus".

2.6.2. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

2.6.3. If using a bailer, the bailer must be equipped with a controlled flow bottom assembly.

### 3. PRESERVATION

3.1. Preserve the sample **during** the sample collection process.

3.2. Dechlorination: Some treated water samples (drinking water and treated wastewater) may contain residual chlorine that must be removed with a dechlorination agent such as sodium thiosulfate or ascorbic acid. This process must occur **before** any additional preservatives (i.e., acid) are added. The dechlorination agent must be **in the vial** before the sample is added.

3.2.1. Laboratories may supply vials with premeasured quantities of dechlorination agent. If acid preservation **is not required**, fill the vials (see section 2.5.1 above) and proceed to section 4 below.

3.2.2. For chlorinated drinking water samples, add 3 mg sodium thiosulfate per 40 mL vial.

3.2.3. If the chlorine level is unknown, the concentration must be measured (see FT 2000). For sources other than drinking water (e.g., chlorinated effluent), 10 mg sodium thiosulfate per 40 mL vial will remove up to 5 ppm  $\text{Cl}_2$ .

### 3.3. Acid Preservation

#### 3.3.1. Chlorinated Samples

3.3.1.1. If acid preservation is required, carefully fill the vial with sample, but not to a convex meniscus as described in section 2.5.1 above.

3.3.1.2. Add four drops of concentrated HCl (more acid may be needed if the sample is known to contain high levels of bicarbonate or is otherwise buffered).

3.3.1.3. Add additional sample to create a convex meniscus.

NOTE: If the sample reacts with the acid by generating gas, do not submit preserved samples for analysis. Instead, collect unpreserved samples (seven-day holding time must be met).

#### 3.3.2. Unchlorinated Samples

3.3.2.1. The laboratory may supply vials with premeasured quantities of acid. In this case, proceed to section 2.5.1 above. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

3.3.2.2. If the samples are preserved in the field, follow the procedure in section 3.3 above.

#### 4. CAPPING THE VIAL

4.1. Fill the vial so that the sample surface is above the container rim (convex meniscus).

4.1.1. **Do not pour** sample into cap.

4.1.2. Fill vial from the original source (tubing, spigot, etc.) **Do not fill vial from sample collected in the cap.**

4.2. **Immediately** cap the vial with the Teflon seal contacting the sample. Some sample may overflow while tightening the cap.

4.3. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.

4.4. Turn the vial over and tap it to check for the presence of bubbles.

4.4.1. If bubbles are present, and the total volume of the bubbles is less than 5 mm in diameter, the sample may be submitted.

4.4.2. If the total volume of the bubbles is greater than 5 mm in diameter, discard the vial and fill a new one.

4.4.3. **Do not open a vial to add additional sample.**

#### 5. SAMPLE PACKING

5.1. Label each vial with an appropriate field ID number and preservation (e.g., preserved with acid, sodium thiosulfate/acid, etc.).

5.2. Wrap each vial in a protective material (e.g., bubble wrap).

5.3. Place the set of vials in a small, sealable, untreated plastic bag unless the laboratory supplies an alternate method of packing.

5.4. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.5. Protect samples from environmental contamination during storage and transport to the laboratory.

5.6. As an added measure, DEP recommends wrapping the set of replicate samples in bubble wrap and sealing them in a container. This procedure will add further protection from potential contamination.

#### 6. DOCUMENTATION

6.1. Label all the vials.

6.2. Complete field records.

6.3. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.

### **FS 2005.** *Bacteriological Sampling*

#### 1. SAMPLE CONTAINERS

1.1. Collect the samples in properly sterilized containers.

- 1.1.1. Presterilized Whirl-pak bags (or equivalent) are generally used.
- 1.1.2. If Whirl-pak bags are not used, the sample container must have a volume of at least 125 mL.
- 1.1.3. If using bottles, the caps must be sterilized. If the caps are lined, there must be documentation to show that the liner does not produce toxic compounds when sterilized.
- 1.1.4. Bottles and caps must be sterilized according to procedures in FC 1320 or purchased presterilized from a commercial vendor.

## 2. SAMPLE COLLECTION PROCEDURES

- 2.1. Unless a composite is specified by permit, all samples must be grab samples.
- 2.2. Do not open the container once it has been sealed.
- 2.3. Do not rinse sample container before collecting the sample.
- 2.4. Use aseptic techniques to collect the sample:
  - 2.4.1. If an intermediate device is used, thoroughly rinse with sample water. To ensure proper rinsing, DEP recommends that microbiological samples be the last sample collected with the sampling device.
  - 2.4.2. Do not put fingers into the mouth of the container or on the interior of the cap.
  - 2.4.3. Do not disinfect the sample equipment or sampling port.
- 2.5. Rinse the sampling equipment with sample water before collecting the sample. Therefore, collect microbiological samples at the end of a sampling sequence.
- 2.6. Wells with In-Place Plumbing, Spigots and/or Faucets
  - 2.6.1. Do not disinfect the spigot with bleach, alcohol or heat. Turn on spigot and flush at maximum velocity (see FS 2310).
  - 2.6.2. After flushing, reduce the water flow to approximately 500 mL/min and allow the water to flow for a few minutes before collecting samples. If other samples (metals, nutrients, etc.) are to be collected, collect these samples first.
  - 2.6.3. **Do not stop the flow before or during the filling process.**
- 2.7. Direct Grab Sample Collection
  - 2.7.1. Hold a rigid container near the base and plunge neck downward, below the surface. Turn container until the neck points slightly upward with the mouth directed toward the current. Fill to within about 1/2 inch of the top and cap immediately.
  - 2.7.2. Whirl-pak bags (or equivalent)
    - Open the bag by zipping off the top and pulling the white tabs to open the bag. Hold the bag behind the wire ties, and plunge neck downward and up in one sweeping arc; or
    - Zip off the top of the bag. Hold bag so that the mouth and wire ties are in front of the hands and fingers. Immerse the bag, and open the bag into the current.
    - The above procedures may also be accomplished by attaching the bag to a pole.
  - 2.7.2.1. Bring the bag to the surface, and press out excess water.

2.7.2.2. Seal the bag by folding the open ends at least three times and securely twisting the wire ties.

## 2.8. Intermediate Device Collection

2.8.1. When using an intermediate sampling device (bailer, DO dunker, niskin bottle, etc.), obtain sufficient sample in the sample collection device to completely fill the sample container. Begin pouring sample out of the device **BEFORE** collecting into the container. Continue to pour sample out of the device, place container under flowing stream, and fill. **Do not stop the flow before or during the filling process.**

## 3. PRESERVATION

3.1. Preserve samples according to Tables FS 1000-4 through FS 1000-10.

3.2. Place all samples in wet ice immediately after sample collection (see FS 1006, section 5).

3.3. When the sample contains residual chlorine, add a dechlorinating agent such as sodium thiosulfate to the sample container.

3.3.1. The final concentration of sodium thiosulfate must be approximately 100 milligrams per liter (mg/L) in the sample (add 0.1 mL of a 10% solution of thiosulfate to a 125 mL sample).

3.3.2. Some vendors or laboratories provide sterile containers with premeasured amounts of dechlorinating agent. Determine if the source of the field containers already contain a dechlorinating agent.

3.3.3. **Do not use containers with dechlorinating chemicals** when collecting samples from sources that are known to be free from residual chlorine.

## 4. HOLDING TIME

4.1. The holding time for microbiological samples is very short. Let the laboratory know the approximate time that samples will be collected and when they are expected to be delivered to the laboratory.

4.2. The holding time begins at the time (hours and minutes) the sample is collected and ends at the time that the sample is placed on the applicable growth media.

4.3. Consult Tables FS 1000-4, -6, -8, and -9 for holding times.

## 5. DOCUMENTATION

5.1. Label each sample container with an appropriate field ID number.

5.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.3. Complete field records.

5.4. Make note in the field records of any unusual sample appearances or sampling conditions.

## **FS 2006.** *Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPHs)*

### 1. SAMPLE CONTAINERS

1.1. Collect samples for O&G and TRPHs in 1-liter wide mouth amber glass bottles.

- 1.2. The cap must have a Teflon liner.
- 1.3. Visually inspect glass bottles and caps for defects. Do not use container if defects are present or if they do not appear clean.
2. SELECTION OF SAMPLING POINTS
  - 2.1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.
    - 2.1.1. Select a point of greatest mixing.
    - 2.1.2. For compliance samples at a facility, collect samples from a point that best represents oil and grease concentrations.
3. SAMPLE COLLECTION PROCEDURES
  - 3.1. All samples must be grab samples.
    - 3.1.1. If composite data are required, collect individual grab samples over the specified time period.
    - 3.1.2. Submit all samples for analysis.
    - 3.1.3. Average the concentrations of the results to determine the average concentration over time.
  - 3.2. Do not collect the sample by skimming the surface.
  - 3.3. Collect a discrete sample that will be used for analysis. Do not use this sample for any other test.
  - 3.4. Remove the cap from the glass bottle without touching the interior of the container or lid.
  - 3.5. Do not rinse the sampling device or the sample container with sample water.
  - 3.6. Collect the sample directly into the container.
    - 3.6.1. If intermediate sampling equipment is needed, do not allow the sampling equipment to touch the rim of the sample container.
    - 3.6.2. Do not use automatic samplers to collect these types of samples.
    - 3.6.3. Fill the bottle with the sample water to almost full capacity.
    - 3.6.4. Add preservatives (see section 4 below).
    - 3.6.5. Quickly cap the container and tighten securely.
4. PRESERVATION
  - 4.1. Preserve the sample within 15 minutes of sample collection.
  - 4.2. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**
  - 4.3. Preserve the sample by adding an accurately measured amount of sulfuric or hydrochloric acid to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.
    - 4.3.1. Tightly cap the sample container and shake to distribute the acid.

4.3.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

4.3.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 4.3.2 above) until the pH has been reduced to below 2 pH units.

4.3.4. Record the total amount of acid that was added to the sample.

4.4. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

4.5. After the sample has been preserved, screw the cap on tightly.

4.6. Immediately place the sample in **wet** ice after preserving with acid (see FS 1006, section 5).

## 5. DOCUMENTATION

5.1. Label each vial with an appropriate field ID number.

5.2. Protect glass container from breakage ("bubble wrap" is recommended).

5.3. Complete field records.

5.4. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

## **FS 2007.**     *Radiological Sampling (Excludes Radon)*

### 1. SAMPLE CONTAINERS

1.1. Use polyethylene, polyvinyl chloride (PVC), or Teflon containers.

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

### 2. SAMPLE COLLECTION PROCEDURES

2.1. On unknown sites, survey the area with a beta-gamma survey instrument, such as a Geiger-Müller meter.

2.1.1. If radiation levels are above instrument background, consult a radiation safety specialist to determine appropriate safety procedures.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

### 3. PRESERVATION

3.1. Preserve the sample with a suitable grade of nitric acid (HNO<sub>3</sub>).

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

3.4. If the preservative is added after the sample is collected (the container is not prepreserved), do not fill the container to the rim.

3.5. Preserve the sample by adding an accurately measured volume of concentrated  $\text{HNO}_3$  or 1:1  $\text{HNO}_3$  to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

3.5.1. Tightly cap the sample container and shake to distribute the acid.

3.5.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

3.5.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 3.5.2 above) until the pH has been reduced to just below 2 pH units.

3.5.4. Record the total amount of acid that was added to the sample.

3.5.5. Cooling to 4°C is not required.

3.6. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

3.7. After the sample has been preserved, screw the cap on tightly.

#### 4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

### **FS 2008.**     *Radon Sampling*

Radon is a gas and is easily removed from water sources. Therefore, follow the same precautions and care used to collect volatile organic samples. Minimize contact with air during sample collection. Other sample collection techniques may be appropriate, depending on the analytical method or as specified in the project data quality objectives.

#### 1. SAMPLE CONTAINERS

1.1. Use glass sample vials containing a premeasured portion of the scintillation "cocktail."

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

1.3. Collect at least two samples.

2. PRESERVATION: The scintillation cocktail is the only required preservative.

3. SAMPLE COLLECTION PROCEDURES Obtain specific sample collection instructions from the laboratory that will analyze the samples. These instructions must include proper handling as well as sample size and packing instructions. The following are general instructions for collecting the samples:

3.1. Carefully fill a syringe (usually 10 mL) with sample water so that air bubbles are not pulled in with the sample before, during or after filling.

3.2. Place the tip of the syringe **BELOW** the scintillation cocktail and slowly dispense the sample **BENEATH** the cocktail surface.

- 3.3. Replace the lid and cap tightly.
  - 3.4. Generally, the vial is used in the laboratory analytical instrument and labels or ID numbers on the sides of the containers may interfere with the analysis. Check with the laboratory for proper placement of labels or field ID numbers.
  - 3.5. Ship in an upright position in the shipping containers that have been provided by the laboratory. If none are provided, protect vials from breakage ("bubble wrap" is recommended), segregate replicate samples in separate plastic bags, and ship to the laboratory in an upright position.
4. DOCUMENTATION
    - 4.1. Complete the field notes.
    - 4.2. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

### **FS 2009.**     *Cyanide Sampling*

Cyanide is a very reactive and unstable species and is highly toxic. Samples suspected of containing cyanide must be handled very carefully.

1. SAMPLE CONTAINERS
  - 1.1. Use polyethylene or glass sample containers.
  - 1.2. Use properly cleaned containers (see FC 1300).
  - 1.3. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.
2. SAMPLE COLLECTION PROCEDURES
  - 2.1. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.
3. PRESERVATION
  - 3.1. Many different analytes interfere with the cyanide analysis (e.g., sulfides). If any interferences are known to be present, pretreat the sample for interferences by following the applicable footnotes in Table FS 1000-4.
  - 3.2. Preserve the sample within 15 minutes of sample collection.
  - 3.3. Preserve samples with sodium hydroxide to a pH greater than 12.
  - 3.4. Preserve the sample by adding an accurately measured amount of a sodium hydroxide solution or sodium hydroxide pellets to the container. Use a graduated container or pipet to add the solution.
    - 3.4.1. Tightly cap the sample container and shake to distribute the preservative.
    - 3.4.2. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is greater than 12. **Do not put the pH paper directly into the sample container.**
    - 3.4.3. If the pH is less than 12, add additional measured amounts of the preservative and test with narrow range pH paper (see section 3.4.2 above) until the pH has been raised to above 12 pH units.



- 3.4.4. Record the total amount of preservative that was added to the sample.
- 3.5. After the sample has been preserved, screw the cap on tightly.
- 3.6. Immediately put the sample in **wet** ice (see FS 1006, section 5).
- 3.7. Preserve at least one of the equipment blanks with all the reagents and the **greatest** amount of sodium hydroxide that was required in the sample set and note the amount in field documentation.
4. DOCUMENTATION
  - 4.1. Complete the sample container label and stick firmly on the container.
  - 4.2. Complete the field notes.
  - 4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.
  - 4.4. Ensure that all preservation measures are part of the field notes.

#### **FS 2010** *Sulfide Sampling*

1. Analyze samples within 15 minutes of collection, or the preserve the sample within 15 minutes for later analysis. If preservation is required add the zinc acetate and sodium hydroxide to the container **before** filling with sample.
2. Avoid aerating the sample during collection. Slowly pour the sample slowly and carefully allow the sample to flow down the **side** of the container to minimize turbulence.
3. Check the pH (if necessary) before completing the filling process.
4. Complete the filling process. **Do not leave a head space.**