

FS 1000. GENERAL SAMPLING PROCEDURES

See also the following Standard Operating Procedures:

- FA 1000 and 2000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

FS 1001. Preliminary Activities

1. Begin each sampling trip with some planning and coordination. Refer to FM 1000 for recommendations and suggestions on laboratory selection and communication, and field mobilization.

1.1. DEP recommends that a minimum of two people be assigned to a field team. In addition to safety concerns, the process of collecting the samples, labeling the containers and completing the field records is much easier if more than one person is present.

1.2. If responding to incidents involving hazardous substances, DEP recommends that four or five people be assigned to the team.

2. EQUIPMENT

2.1. Select appropriate equipment based on the sampling source (see FS 2000 to FS 8200), the analytes of interest and the sampling procedure.

2.1.1. If properly cleaned, sample containers may be used as collection devices or intermediate containers.

2.2. The equipment construction must be consistent with the analytes or analyte groups to be collected (see Tables FS 1000-1 and FS 1000-2).

2.3. Bring precleaned equipment to the field or use equipment that has been certified clean by the vendor or laboratory.

3. DEDICATED EQUIPMENT STORAGE

3.1. Store all dedicated equipment (except dedicated pump systems or dedicated drop pipes) in a controlled environment.

3.2. If possible, store equipment in an area that is located away from the sampling site. If equipment other than dedicated pumps or dedicated drop pipes is stored in monitoring wells, suspend the equipment above the formation water.

3.3. Securely seal the monitoring well in order to prevent tampering between sampling events.

3.4. Decontaminate all equipment (except dedicated pumps or drop pipes) before use according to the applicable procedures in FC 1000.

4. SAMPLE CONTAINERS

4.1. The analyses to be performed on the sample determine the construction of sample containers.

4.2. Inspect all containers and lids for flaws (cracks, chips, etc.) before use. Do not use any container with visible defects or discoloration.

FS 1002. *Contamination Prevention and Sample Collection Order*

1. CONTAMINATION PREVENTION

1.1. Take special effort to prevent cross contamination and contamination of the environment when collecting samples. Protect equipment, sample containers and supplies from accidental contamination.

1.1.1. Do not insert pump tubing, measurement probes, other implements, fingers, etc. into sample containers or into samples that have been collected for laboratory analysis.

1.1.1.1. If it is necessary to insert an item into the container or sample, ensure that the item is adequately decontaminated for the analytes of interest to be analyzed in the sample.

1.1.2. If possible, collect samples from the least contaminated sampling location (or background sampling location) to the most contaminated sampling location.

1.1.2.1. Collect the ambient or background samples first and store them in separate ice chests or shipping containers.

1.1.3. Collect samples in flowing water from downstream to upstream.

1.1.4. Do not store or ship highly contaminated samples (concentrated wastes, free product, etc.) or samples suspected of containing high concentrations of contaminants in the same ice chest or shipping container with other environmental samples.

1.1.4.1. Isolate these sample containers by sealing them in separate, untreated plastic bags immediately after collecting, preserving, labeling, etc.

1.1.4.2. Use a clean, untreated plastic bag to line the ice chest or shipping container.

2. SAMPLE COLLECTION ORDER

2.1. Sampling order is a recommendation to be modified depending on site circumstances. Unless field conditions justify other sampling regimens, collect samples in the following order:

- Volatile Organics and Volatile Inorganics
- Extractable Organics, Petroleum Hydrocarbons, Aggregate Organics and Oil & Grease
- Total Metals
- Dissolved Metals
- Inorganic Nonmetallics, Physical and Aggregate Properties, and Biologicals
- Radionuclides
- Microbiological

Note: If the pump used to collect groundwater samples cannot be used to collect volatile or extractable organics, then collect all other parameters, withdraw the pump and tubing, and collect the volatile and extractable organics.

3. COMPOSITE SAMPLES

- 3.1. Do not collect composite samples unless required by permit or DEP program.
- 3.2. If compositing is required, use the following procedure:
 - 3.2.1. Select sampling points from which to collect each aliquot.
 - 3.2.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.
 - 3.2.3. Record the approximate amount of each aliquot (volume or weight).
 - 3.2.4. Add preservative(s), if required.
 - 3.2.5. Label container and make appropriate field notes (see FD 1000-9000).
 - 3.2.6. Notify the laboratory that the sample is a composite sample.
 - 3.2.7. When collecting soil or sediment samples, combine the aliquots of the sample directly in the sample container with no pre-mixing. Notify the laboratory that the sample is an unmixed composite sample, and request that the laboratory thoroughly mix the sample before sample preparation or analysis.
 - 3.2.8. When collecting water composites see FS 2000, section 1.3 or pertinent sections of other water matrix SOPs for specific details on collection.

FS 1003. *Protective Gloves*

1. Gloves serve a dual purpose to:
 - Protect the sample collector from potential exposure to sample constituents
 - Minimize accidental contamination of samples by the collector
2. The DEP recommends wearing protective gloves when conducting all sampling activities. They must be worn except when:
 - The sample source is considered to be non-hazardous
 - The samples will not be analyzed for trace constituents
 - The part of the sampling equipment that is handled without gloves does not contact the sample source
3. Do not let gloves come into contact with the sample or with the interior or lip of the sample container.
4. Use clean, new, unpowdered and disposable gloves.
 - 4.1. DEP recommends latex gloves, however, other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.
 - 4.2. Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. Many vendor catalogs provide information about the permeability of different gloves and the circumstances under which the glove material might be applicable.
 - 4.3. The powder in powdered gloves can contribute significant contamination and DEP does not recommend wearing powdered gloves unless it can be demonstrated that the powder does not interfere with the sample analysis.

5. If gloves are used, change:
 - After preliminary activities such as pump placement;
 - After collecting all the samples at a single sampling point; or
 - If torn, or used to handle extremely dirty or highly contaminated surfaces.
6. Properly dispose of all used gloves.

FS 1004. *Container and Equipment Rinsing*

When collecting aqueous samples, rinse the sample collection equipment with a portion of the sample water before taking the actual sample. Sample containers do not need to be rinsed. In the case of petroleum hydrocarbons, oil & grease or containers with premeasured preservatives, the sample containers cannot be rinsed.

FS 1005. *Fuel-Powered Equipment and Related Activities*

1. Place all fuel-powered equipment away from, and downwind of, any site activities (e.g., purging, sampling, decontamination). If field conditions preclude such placement (i.e., the wind is from the upstream direction in a boat), place the fuel source(s) as far away as possible from the sampling activities and describe the conditions in the field notes.
2. Handle fuel (i.e., filling vehicles and equipment) prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves. Dispense all fuels, dispose of gloves downwind, and well away from the sampling activities.
3. If sampling at active gas stations, stop sample collection activities during fuel deliveries.

FS 1006. *Preservation, Holding Times and Container Types*

1. Preserve all samples according to the requirements specified in Tables FS 1000-4 through FS 1000-10.
 - 1.1. The information listed in the above-referenced tables supersedes any preservation techniques, holding time or container type that might be discussed in individual analytical methods.
 - 1.2. If samples are collected only for total phosphorus and are not for NPDES compliance, thermal preservation (ice) is not required if the sample containers are pre-preserved with acid.
2. The preservation procedures in the referenced tables specify immediate preservation. "Immediate" is defined as "within 15 minutes of sample collection." Perform all preservation on-site (in the field).
 - 2.1. Preservation is not required if samples can be transported back to the laboratory within 15 minutes of collecting the sample and
 - 2.1.1. The laboratory begins sample analysis within the 15-minute window and documents the exact time the analysis began, or
 - 2.1.2. The laboratory adds the appropriate preservatives (including thermal preservation) within 15 minutes of sample collection and documents the exact time that the preservation was done.
3. PRESERVING COMPOSITE WATER SAMPLES

3.1. If the sample preservation requires thermal preservation (e.g., $<6^{\circ}\text{C}$), the samples must be cooled to the specified temperature.

3.1.1. Manually collected samples to be composited must be refrigerated at a temperature equal to or less than the required temperature.

3.1.2. Automatic samplers must be able to maintain the required temperature by packed ice or refrigeration.

3.2. When chemical preservation is also required, begin the preservation process within 15 minutes of the last collected sample.

3.3. Holding Times for Automatic Samplers:

3.3.1. If the collection period is 24 hours or less, the holding time begins at the last scheduled sample collection;

3.3.2. If the collection period exceeds 24 hours, the holding time begins with the time that the first sample is collected.

4. PH ADJUSTED PRESERVATION - Check the pH of pH-adjusted samples according to these frequencies:

4.1. During the first sampling event at a particular site, check all samples (includes each groundwater monitoring well, surface water location, or influent/effluent sampling location) that are pH-adjusted except volatile organics.

4.2. During subsequent visits to a particular site, check at least one sample per parameter group that must be pH-adjusted.

4.3. If the frequency of sample collection at a specified location is greater than once per month (i.e., weekly or daily), check the pH of at least one sample per parameter group (except volatile organics) according to the following schedule:

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

4.4. 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.

4.5. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

5. THERMAL PRESERVATION

5.1. When preservation requirements indicate cooling to a specific temperature, samples must be placed in wet ice within 15 minutes of sample collection (see 1006, section 2 above). Unless specified, do not freeze samples.

5.2. All supplies (ice, dry ice, etc.) necessary to meet a thermal preservation requirement must be onsite for immediate use.

5.3. Ship samples in wet ice. If samples are cooled to the required temperature before shipment, samples may be shipped with frozen ice packs if the specified temperature is maintained during shipment. The sample temperature must not exceed the specified temperature.

5.4. If immediate freezing is required, dry ice must be available in the field to begin the freezing process.

FS 1007. *Preventive and Routine Maintenance*

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

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

1. Follow the manufacturer's suggested maintenance activities and document all maintenance. At a minimum, DEP recommends the activities listed on Table FS 1000-12.

2. Maintain documentation for the following information for each piece of equipment or instrumentation. See FD 3000 also.

2.1. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number or other unique identification.

2.2. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures and solution or parts replacement for instrument probes.

2.3. Include the calendar date for the procedures performed.

2.4. Record names of personnel performing the maintenance or repair tasks.

2.5. Describe any malfunctions necessitating repair or service.

2.6. Retain vendor service records for all affected instruments.

2.7. Record the following for rented equipment:

- Rental date(s)
- Equipment type and model or inventory number or other description

2.8. Retain the manufacturer's operating and maintenance instructions.

FS 1008. *Documentation and References*

1. REFERENCES: All sampling references must be available for consultation in the field. These include:

- DEP SOPs;
- Internal SOPs;
- Sampling and analysis plans; and/or
- Quality Assurance Project Plans.

2. DOCUMENTATION: Complete and sign all documentation (see FD 1000).

FS 1009. *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

1.1. Document all activities related to a sampling event, including sample collection, equipment calibration, equipment cleaning and sample transport.

1.2. The required documentation related to each sampling or other field activity is specified in the associated SOPs; i.e., FQ 1000, FC 1000, the FS series, and the FT series.

1.3. The documentation requirements are also summarized in FD 1000, Field Documentation. FD 1000 additionally contains a list of example forms published with the SOPs that may be used to document various activities or as templates for creating customized forms.

2. LEGAL CHAIN OF CUSTODY (COC)

The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

Evidentiary sample custody protocols are used to demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

When a client or situation requires legal COC, use the procedures in FD 7000 to document and track all time periods associated with the physical possession and storage of sample containers, samples, and subsamples from point of origin through the final analytical result and sample disposal.

When legal or evidentiary COC is required, samples must be:

- In the actual possession of a person who is authorized to handle the samples (e.g., sample collector, laboratory technician);
- In the view of the same person after being in their physical possession;
- Secured by the same person to prevent tampering; or
- Stored in a designated secure area.

2.1. Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times.

2.1.1. Limit the number of individuals who physically handle the samples as much as practicable.

2.1.2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

2.1.3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

2.1.4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

2.2. Use a Chain of Custody form or other transmittal record to document sample transfers to other parties. Other records and forms may be used to document internal activities if they meet the requirements for legal chain of custody.

2.3. Legal COC begins when the precleaned sample containers are dispatched to the field.

2.3.1. The person who relinquishes the prepared sample kits or containers and the individual who receives the sample kits or containers must sign the COC form unless the same party provides the containers and collects the samples.

2.3.2. All parties handling the empty sample containers and samples are responsible for documenting sample custody, including relinquishing and receiving samples, except commercial common carriers.

2.4. Shipping Samples under Legal COC

2.4.1. Complete all relevant information on the COC transmittal form or record (see FD 7200, section 2).

2.4.2. Internal records must document the handling of the samples and shipping containers in preparation for shipment. The names of all persons who have prepared the shipment must be recorded. All time intervals associated with handling and preparation must be accounted for.

2.4.3. Place the forms in a sealed waterproof bag and place in the shipping container with the samples.

2.4.4. Seal the shipping container with tamper-proof seals (see 2.6 below) so that any tampering can be clearly seen by the individual who receives the samples.

2.4.5. Note: The common carrier does not sign COC records. However, the common carrier (when used) must be identified.

2.5. Delivering Samples to the Laboratory

2.5.1. All individuals who handle and relinquish the sample containers must sign the transmittal form. The legal custody responsibilities of the field operations end when the samples are relinquished to the laboratory.

2.6. Chain of Custody Seals: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

2.6.1. Place the seal so that the closure cannot be opened without breaking the seal.

2.6.2. Record the time, calendar date and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.

2.6.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FS 1010. *Health and Safety*

Implement all local, state and federal requirements relating the health and safety.

FS 1011. *Hazardous Wastes*

Follow all local, state and federal requirements pertaining to the storage and disposal of any hazardous or investigation-derived wastes.

1. Properly manage all investigation-derived waste (IDW) so contamination is not spread into previously uncontaminated areas.
 - 1.1. IDW includes all water, soil, drilling mud, decontamination wastes, discarded personal protective equipment (PPE), etc. from site investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, and abandonment, and other investigative activities. Containerize the IDW at the time it is generated.
 - 1.2. Determine if the IDW must be managed as Resource Conservation and Recovery Act (RCRA) regulated hazardous waste through appropriate testing or generator knowledge. Manage all IDW that is determined to be RCRA regulated hazardous waste according to the local state and federal requirements.
 - 1.3. Properly dispose of IDW that is not a RCRA-regulated hazardous waste but is contaminated above the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality.
 - 1.4. IDW that is not contaminated or contains contaminants below the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality may be disposed of onsite as long as the IDW will not cause a surface water violation.
 - 1.5. Maintain all containers holding IDW in good condition:
 - 1.5.1. Periodically inspect the containers for damage
 - 1.5.2. Ensure that all required labeling (DOT, RCRA, etc.) are clearly visible.

Appendix FS 1000
Tables, Figures and Forms

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Table FS 1000-2	Construction Material Selection for Equipment and Sample Containers
Table FS 1000-3	Equipment Use and Construction
Table FS 1000-4	40 CFR Part 136 Table II: Required Containers, Preservation Techniques, and Holding Times (Water/Wastewater Samples)
Table FS 1000-5	Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times for Analytes not found in 40 CFR Part 136
Table FS 1000-6	Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples.
Table FS 1000-7	Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035
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Table FS 1000-12	Preventive Maintenance Tasks
Figure FS 1000-1	Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump

DEP-SOP-001/01
FS 1000 General Sampling Procedures
Table FS 1000-1
Equipment Construction Materials

Construction Material ¹	Acceptable Analyte Groups	Precautions
Metals		
316 Stainless Steel	All analyte groups. Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted. ²
300-Series Stainless Steel (304, 303, 302)	Suitable for all analyte groups (if used, check for corrosion before use). Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted. ² If corroded, there is a potential for samples to be contaminated with iron, chromium, copper or nickel. Check for compatibility with water chemistry for dedicated applications. Do not use in low pH, high chloride, or high TDS waters.
Low Carbon Steel Galvanized Steel Carbon Steel	Inorganic nonmetallics only.	Coring devices are acceptable for all analyte groups if appropriate liners are used. Use Teflon liners for organics. Use plastic or Teflon liners for metals. Do not use if weathered, corroded or pitted. ² If corroded, there is a potential for samples to be contaminated with iron and manganese. Galvanized equipment will also contaminate with zinc and cadmium. If used to collect large samples (e.g., dredges), collect organic and metal samples may be collected from portions of the interior of the collected material.
Brass	Inorganic nonmetallics only.	Do not use if weathered, corroded or pitted. ²
Plastics³		
Teflon and other fluorocarbon polymers	All analyte groups. Especially recommended for trace metals and organics.	Easily scratched. Do not use if scratched or discolored.
Polypropylene Polyethylene (All Types)	All analyte groups.	Easily scratched. Do not use if scratched or discolored.
Polyvinyl chloride (PVC)	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organics samples.

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FS 1000 General Sampling Procedures
Table FS 1000-1
Equipment Construction Materials

Construction Material¹	Acceptable Analyte Groups	Precautions
Tygon, Silicone, Neoprene	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organic samples. Do not use silicone if sampling for silica.
Viton	All analyte groups except extractable and volatile organics. ⁴	Minimize contact with sample. Use only if no alternative material exists.
Glass		
Glass, borosilicate	All analyte groups except silica and boron.	

Adapted from USGS Field Manual, Chapter 2, January 2000.

¹ Refers to construction material of the portions of the sampling equipment that come in contact with the sample (e.g., housing of variable speed submersible pump must be stainless steel if extractable organics are sampled; the housing of a variable speed submersible pump used to sample metals may be plastic.)

² Corroded/weathered surfaces are active sorption sites for organic compounds.

³ Plastics used in connection with inorganic trace element samples (including metals) must be uncolored or white.

⁴ May be allowable for specialized parts where no alternative material exists (e.g., Viton seals are the best available seal for some dedicated pump systems), however, contact with the sample must be minimized.

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Extractable Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Volatile Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Metals	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass (except silica and boron)
Ultratrace Metals	Teflon Polyethylene (All types) Polypropylene (All types) Polycarbonate Mercury must be in glass or Teflon
Inorganic Nonmetallics	Teflon Stainless steel Low carbon, Galvanized or Carbon steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Brass

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Microbiological samples	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Sterilize all sample containers. Thoroughly clean sampling equipment and rinse several times with sample water before collection. Sampling equipment does not require sterilization Do not rinse sample containers

Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>	<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>		
	<u>HOUSING¹</u>	<u>TUBING</u>				
WATER SAMPLING						
GROUNDWATER						
1 Positive displacement pumps ²						
a. Submersible (turbine, helical rotor, gear driven)	SS, Teflon	SS, Teflon, PE, PP	Purging	All analyte groups	^{3,4,5} ; must be variable speed	
			Sampling	All analyte groups	^{3,4,5} must be variable speed	
	SS, Teflon	Non-inert ⁶	Purging	All analyte groups	^{3,4,5} must be variable speed; polishing required ⁷	
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS	
	Non-inert ⁶	Non-inert ⁶	Purging	All analyte groups	^{3,4,5} must be variable speed; polishing required ⁷	
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS	
	b. Bladder pump (no gas contact)	SS, Teflon, PE, PP or PVC if permanently installed	SS, Teflon, PE, PP	Purging	All analyte groups	^{3,4,5} must be variable speed
				Sampling	All analyte groups	^{3,4} must be variable speed Bladder must be Teflon if sampling for volatile or extractable organics or PE or PP if used in portable pumps
SS, Teflon, PE, PP		Non-inert ⁶	Purging	All analyte groups	^{3,4} must be variable speed; polishing required ⁷ This configuration is <u>not</u> recommended	
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	^{3,4} must be variable speed If sampling for metals, the tubing must be non-metallic if not SS	
Non-inert ⁶		Non-inert ⁶	Purging	All analyte groups	^{3,4} must be variable speed; polishing required ⁷	
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	^{3,4} must be variable speed; polishing required ⁷ If sampling for metals, the tubing must be non-metallic if not SS	

Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u> ¹	<u>TUBING</u>	<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
2. Suction lift pumps					
a. Centrifugal	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	⁴ foot-valve required Must be variable speed
	N/A	Non-inert ⁶	Purging	All analyte groups	⁴ foot-valve required; polishing required/ Must be variable speed
b. Peristaltic	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	⁴ foot-valve required; polishing required/ or continuous pumping required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile organics	⁴ Silicone tubing in pump head Must be variable speed
	N/A	Non-inert ⁶	Purging	All analyte groups	⁴ foot-valve required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	⁴ Silicone tubing in pump head Must be variable speed
3. Bailers	SS, Teflon, PE, PP	N/A	Purging	All analyte groups	None; <u>not recommended</u>
			Sampling	All analyte groups	None; <u>not recommended</u>
	Non-inert ⁶	N/A	Purging	All analyte groups <u>except</u> volatile and extractable organics	None; <u>not recommended</u> If sampling for metals, the tubing must be non-metallic if not SS
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	None; <u>not recommended</u> If sampling for metals, the tubing must be non-metallic if not SS
<u>SURFACE WATER</u>					
1. Intermediate containers such as pond sampler, scoops, beakers, buckets, and dippers	SS, Teflon, Teflon-coated, PE, PP	N/A	Grab sampling	All analyte groups	None
	Glass	N/A		All analyte groups except boron and fluoride	None
	Non-inert ⁶	N/A		All analyte groups <u>except</u> volatile and extractable organics	None

Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
2. Nansen, Kemmerer, Van Dorn, Alpha and Beta Samplers, Niskin (or equivalent)	SS, Teflon, Teflon-coated, PE, PP	N/A	Specific depth grab sampling	All analyte groups	None
	Non-inert ⁶	N/A		All analyte groups <u>except</u> volatile and extractable organics	None
3. DO Dunker	SS, Teflon, glass, PE, PP	N/A	Water column composite sampling	All analyte groups	None
4. Bailers – double valve	SS, Teflon, PE, PP	N/A	Grab sampling	All analyte groups	None
	Non-inert ⁶	N/A	Grab sampling	All analyte groups <u>except</u> volatile and extractable organics	None If sampling for metals, the tubing must be non-metallic if not SS
5. Peristaltic pump	N/A	SS, Teflon, PE, PP	Specific depth sampling	All analyte groups <u>except</u> volatile organics	Silicone tubing in pump head Must be variable speed
	N/A	Non-inert ⁶		All analyte groups <u>except</u> volatile and extractable organics	Silicone tubing in pump head Must be variable speed
<u>FIELD FILTRATION UNITS</u>	N/A		Dissolved constituents	Inorganic nonmetallics and metals in surface water Inorganic nonmetallics in groundwater Metals in groundwater and static wastewater and surface water Metals in moving surface water (i.e., river/stream)	Must use a 0.45 µm filter Must use a 0.45 µm filter Must use in-line, high capacity, one-piece molded filter that is connected to the outlet of a pump; no intermediate vessels; positive pressure PE, PP & Teflon bailers acceptable Must use a 1 µm filter in groundwater, a 0.45 µm filter in surface water Must use positive pressure device, but an intermediate vessel may be used. Use a 0.45 µm filter

Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u> <u>HOUSING</u> ¹	<u>TUBING</u>	<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
SOLID SAMPLING					
SOILS					
1. Core barrel (or liner)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	9, 10, 11
	Non-inert ⁶ nonmetallics	N/A	Sampling	All analyte groups	12
	Non-inert ⁶ metals	N/A	Sampling	All analyte groups	12
2. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups ⁸	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics must grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups <u>except</u> volatile and extractable organics	None Must be nonmetallic if not SS
3. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	11
			Compositing or homogenizing	All analyte groups except volatile organics	11
	Non-inert ⁶	N/A	Compositing or homogenizing	All analyte groups	10, 11, 12 must be nonmetallic if not SS
4. Shovel, bucket auger	SS	N/A	Sampling	All analyte groups ⁸	None
	Non-SS	N/A	Sampling	All analyte groups ⁸	10, 11, 12
5. Split spoon	SS or carbon steel w/ Teflon insert	N/A	Sampling	All analyte groups ⁸	10, 11, 12
6. Shelby tube	SS	N/A	Sampling	All analyte groups ⁸	9
	Carbon steel	N/A	Sampling	All analyte groups	9, 10, 12
SEDIMENT					
1. Coring devices	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	9, 10, 11

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Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u> <u>HOUSING</u> ¹	<u>TUBING</u>	<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	Non-inert ⁶ nonmetallics	N/A	Sampling	All analyte groups	¹²
	Non-inert ⁶ metals	N/A	Sampling	All analyte groups	^{9,10,11}
2. Grab – Young, Petersen, Shipek	Teflon, Teflon-lined, SS	N/A	Sampling	All analyte groups ⁸	None
	Carbon steel	N/A	Sampling	All analyte groups	^{10,11}
3. Dredges – Eckman, Ponar, Petit Ponar Van Veen	SS	N/A	Sampling	All analyte groups ⁸	None
	Carbon steel, brass	N/A	Sampling	All analyte groups	^{10,11}
4. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups ⁸	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics be grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups <u>except</u> volatile and extractable organics	None must be nonmetallic if not SS
5. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	¹¹
			Compositing or homogenizing	All analyte groups except volatile organics	¹¹
	Non-inert ⁶	N/A	Compositing or homogenizing	All analyte groups <u>except</u> volatile and extractable organics	none ¹¹ must be nonmetallic if not SS
WASTE ¹³					
Scoop	SS	N/A	Liquids, solids & sludges	All analyte groups ⁸	Cannot collect deeper phases
Spoon	SS	N/A	Solids, sludges	All analyte groups ⁸	Cannot collect deeper phases
Push tube	SS	N/A	Solids, sludges	All analyte groups ⁸	Cannot collect deeper phases
Auger	SS	N/A	Solids	All analyte groups ⁸	None

Table FS 1000-3
Equipment Use and Construction

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
Sediment sampler	SS	N/A	Impoundments, piles	All analyte groups ⁸	None
Ponar dredge	SS	N/A	Solids, sludges & sediments	All analyte groups ⁸	None
Coliwasa, Drum thief	Glass	N/A	Liquids, sludges	All analyte groups	None
Mucksucker, Dipstick	Teflon		Liquids, sludges	All analyte groups	Not recommended for tanks > 11 feet deep
Bacon bomb	SS	N/A	Liquids	All analyte groups ⁸	Not recommended for viscous wastes
Bailer	SS, Teflon	N/A	Liquids	All analyte groups ⁸	Do not use with heterogeneous wastes Not recommended for viscous wastes
Peristaltic pump	N/A	Teflon, Glass	Liquids	All analyte groups except volatile organics	Do not use in flammable atmosphere Not recommended for viscous wastes
Backhoe bucket	Steel	N/A	Solids, Sludges		Difficult to clean Volatiles and metals must be taken from the interior part of the sample
Split spoon	SS	N/A	Solids	All analyte groups ⁸	
Roto-Hammer	Steel	N/A	Solids	All analyte groups ⁸	Physically breaks up sample Not for flammable atmospheres

Acronyms:

N/A not applicable
SS stainless steel
HDPE high-density polyethylene
PE polyethylene
PVC polyvinyl chloride
PP polypropylene

Table FS 1000-3
Equipment Use and Construction

-
- ¹ Refers to tubing and pump housings/internal parts that are in contact with purged or sampled water (interior and exterior of delivery tube, inner lining of the discharge tube, etc.).
- ² If used to collect volatile or extractable organics, all power cords and other tubing must be encased in Teflon, PE or PP.
- ³ If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.
- ⁴ Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between uses.
- ⁵ In-line check valve required.
- ⁶ "Non-inert" pertains to materials that are reactive (adsorb, absorb, etc.) to the analytes being sampled. For organics, materials include rubber, plastics (except PE and PP), and PVC. For metals, materials include brass, galvanized, and carbon steel.
- ⁷ "Polishing": When purging for volatile or extractable organics, the entire length of tubing or the portion which comes in contact with the formation water must be constructed of Teflon, SS, PE or PP. If other materials (e.g., PVC, garden hoses, etc.) are used, the following protocols must be followed: 1) slowly withdraw the pump from the water column during the last phase of purging, to remove any water from the well that may have contacted the exterior of the pump and/or tubing; 2) remove a single well volume with the sampling device before sampling begins. **Do not use Tygon** for purging if purgeable or extractable organics are of interest. Polishing **is not recommended**; use of sampling equipment constructed of appropriate materials is preferred.
- ⁸ Do not use if collecting for hexavalent chromium (Chromium⁺⁶)
- ⁹ If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.
- ¹⁰ If a non-stainless steel (carbon steel, aluminum) liner, core barrel or implement is used, take the samples for metals, purgeable organics and organics from the interior part of the core sample.
- ¹¹ Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest.
- ¹² If non-inert-liner, core barrel or implement is used, take samples from the interior part of the collected sample.
- ¹³ If disposable equipment of alternative construction materials is used, the construction material must be compatible with the chemical composition of the waste, cannot alter the characteristics of the waste sample in any way, and cannot contribute analytes of interest or any interfering components.

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ^{6, 7}
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
Table IA— Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ⁸	36 hours
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C ⁹	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C ⁹	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C ⁹	48 hours
10. Boron	P, FP, or Quartz	HNO ₃ to pH<2	6 months
11. Bromide	P, FP, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, FP, G	Cool, ≤6 °C ⁹	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6 °C ⁹	48 hours
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C ⁹ , NaOH to pH>12 ¹⁰ , reducing agent ⁵	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
Table IB—Metals:			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C ⁹ , pH = 9.3–9.7 ¹²	28 days
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH<2	28 days

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹³	5 mL/L 12N HCl or 5 mL/L BrCl ¹³	90 days ¹³
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO ₃ to pH<2, or at least 24 hours prior to analysis ¹⁴	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C ⁹	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C ⁹	48 hours
41. Oil and grease	G	Cool, ≤6 °C ⁹ , HCl or H ₂ SO ₄ to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C ⁹ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤6 °C ⁹	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C ⁹	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C ⁹	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C ⁹	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C ⁹	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C ⁹	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C ⁹	7 days
61. Silica	P or Quartz	Cool, ≤6 °C ⁹	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C ⁹	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C ⁹	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C ⁹ , add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes
68. Surfactants	P, FP, G	Cool, ≤6 °C ⁹	48 hours

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to all Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C ⁹	48 hours

Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ¹⁶	14 days ¹⁶
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH to 4–5 ¹⁷	14 days ¹⁷
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
7, 38. Benzidines ^{18,19}	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction ²⁰
14, 17, 48, 50–52. Phthalate esters ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	7 days until extraction, 40 days after extraction
82–84. Nitrosamines ^{18,21}	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
88–94. PCBs ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	1 year until extraction, 1 year after extraction
54, 55, 75, 79. Nitroaromatics and isophorone ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 87. Haloethers ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
29, 35–37, 63–65, 107. Chlorinated hydrocarbons ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	7 days until extraction, 40 days after extraction
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs ¹⁸			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH<9	1 year

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to all Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C ⁹	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C ⁹	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year
Table ID—Pesticides			
Tests: 1–70. Pesticides ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , pH 5–9 ²²	7 days until extraction, 40 days after extraction
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO ₃ to pH<2	6 months
Table IH—Bacterial Tests:			
1. <i>E. coli</i>			
2. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
Table IIH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours. ²³
9. Giardia	LDPE; field filtration	0–8 °C	96 hours ²³

Reference: This table is adapted from Table II, 40 CFR Part 136, 2007

¹ “P” is polyethylene; “FP” is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; “G” is glass; “PA” is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); “LDPE” is low density polyethylene.

² Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample,

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

³ When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the

Table FS1000-4

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date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

⁵ Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$), ascorbic acid, sodium arsenite (NaAsO_2), or sodium borohydride (NaBH_4). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If NaBH_4 or NaAsO_2 is used, 25 mg/L NaBH_4 or 100 mg/L NaAsO_2 will reduce more than 50 mg/L of chlorine (see method “Kelada-01” and/or Standard Method

4500–CN⁻ for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

⁶ Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

⁷ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

⁸ Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

⁹ Aqueous samples must be preserved at $\leq 6^\circ\text{C}$, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “ $\leq 6^\circ\text{C}$ ” is used in place of the “ 4°C ” and “ $< 4^\circ\text{C}$ ” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures ($1/100^{\text{th}}$ of 1 degree); rather, three

Table FS1000-4

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significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

¹⁰ Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) SULFUR: To remove elemental sulfur (S₈), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) SULFIDE: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH

< 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

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12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) SULFITE, THIOSULFATE, OR THIOCYANATE: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA-1677) to preclude cyanide loss or positive interference.

(4) ALDEHYDE: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) CARBONATE: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500-CN.B.3.d).

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40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

(6) CHLORINE, HYPOCHLORITE, OR OTHER OXIDANT: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

¹¹ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

¹² To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

¹³ Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁴ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

¹⁵ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

¹⁶ If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁷ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

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40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

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¹⁸ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤ 6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 19, 20 (regarding the analysis of benzidine).

¹⁹ If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

²⁰ Extracts may be stored up to 30 days at < 0 °C.

²¹ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling

²² The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

²³ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

Analyte	Methods	Reference ¹	Container ²	Preservation ³	Maximum Holding Time ⁴
Bromine	DPD Colorimetric ⁵	SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 ⁶	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G ⁷	Dark 4°C Filtered, dark, -20°C	48 hours chilled until filtration ⁸ , and analyze immediately or 48 hours chilled until filtration ⁸ , and 28 days (frozen) after filtration
Corrosivity	Calculated (CaCO ₃ Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C ⁹	7 days ⁹
FL-PRO	Gas Chromatography	DEP (11/1/95)	G only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	7 days until extraction, 40 days after extraction
Odor	Human Panel	SM 2150	G only	Cool 4°C	6 hours
Salinity	Electrometric ¹⁰ Hydrometric ¹⁰	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days ¹⁰
Taste	Human Panel	SM 2160 B, C, D ASTM E679-91	G only	Cool 4°C	24 hours
Total Dissolved Gases	Direct-sensing Membrane-diffusion	SM 2810	_____	_____	Analyze in-situ
Total Petroleum Hydrocarbons	Gravimetry	EPA 1664	G only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	28 days
Transparency	Irradiometric ¹¹	62-302.200(6), FAC	_____	_____	Analyze in-situ
Un-ionized Ammonia	Calculated ¹²	DEP-SOP ¹³	P, G	Cool 4°C Na ₂ S ₂ O ₃ ¹²	8 hours unpreserved 28 days preserved ¹²
Organic Pesticides ¹⁴	GC and HPLC	EPA (600-series) ¹⁴	¹⁵	¹⁵	¹⁵

¹ SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 20th edition, 1998 and Standard Methods Online.

ASTM XXXX-YY = procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1999.

² P = plastic, G = glass.

³ When specified, sample preservation should be performed immediately upon sample collection.

⁴ The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

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- ⁵ The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).
- ⁶ The Determination of Inorganic Anions in Water by Ion Chromatography", EPA Method 300.0, Revised August 1993, by John D. Pfaff, U. S. EPA Cincinnati, Ohio 45268.
- ⁷ Collect samples in opaque bottles and process under reduced light.
- ⁸ Samples must be filtered within 48 hours of collection. Add magnesium carbonate to the filter while the last of the sample passes through the filter..
- ⁹ Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.
- ¹⁰ The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.
- ¹¹ Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DEP Chapter 62-302, FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.
- ¹² The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on-site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H₂SO₄ to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.
- ¹³ DEP Central Analytical Laboratory, Tallahassee, FL, Revision No. 2, 2-12-2001. The document is available from the DEP Standards & Assessment Section..
- ¹⁴ Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) that are not included in Table ID of 40 CFR Part 136 (July 2007).
- ¹⁵ Container, preservation and holding time as specified in each individual method must be followed.

Table FS 1000-6
Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

Analyte	Methods	References	Container	Preservation	Maximum Holding Times
Volatile Organics	Purge-and-Trap GC and GC-MS	8015, 8260, 8021, 5035	See Table 1000-7		
Semivolatile Organics	GC, HPLC, and GC-MS	8041, 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 8141, 8151, 8270, 8275, 8280, 8290, 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332, 8410, 8430, 8440, FL-PRO	Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C ¹	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C ¹ in dark	30 days until extraction, 45 days after extraction
Total Metals-except mercury and chromium VI methods	Flame AA, Furnace AA, Hydride and ICP	All 7000-series (except 7195, 7196, 7197, 7198, 7470 and 7471), and 6010 (ICP)	Glass or plastic 8 oz widemouth (200 grams sample)	None	6 months
Chromium VI	Colorimetric, Chelation with Flame AA (200 gram sample)	7196 and 7197 (prep 3060)	Glass or plastic, 8 oz widemouth (200 gram sample)	Cool 4° ± 2°C ¹	1 month until extraction, 4 days after extraction ²
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4° ± 2°C ¹	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool 4°C ¹	24 hours
Aggregate Properties			Glass or plastic	Cool 4°C ¹	14 days
Inorganic nonmetallics all except:			Glass or plastic	Cool 4°C ¹	28 days
----- Sulfite, Nitrate, Nitrite & o-phosphate			Glass or plastic		48 hours
----- Elemental Phosphorus			Glass		48 hours

Table FS 1000-6

Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

The term "residuals" include: (1) sludges of domestic origin having no specific requirements in Tables FS-1000-4 or FS-1000-9; (2) sludges of industrial origin; and (3) concentrated waste samples.

¹ Keep soils, sediments and sludges cool at 4°C from collection time until analysis. No preservation is required for concentrated waste samples.

² Storage Temperature is 4°C, $\pm 2^\circ\text{C}$

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

Conc. Level	Sampling Device	Collection Procedure	Sample Container		Preservation	Sample Preparation	Max HT ^①	Determinative Procedure
			Type	Vial Preparation				
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1	NaHSO ₄ / 4°C	5035 - Section 7.2	14 D	Any recognized VOC Method
				5035 - 6.1.1 ^②	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
				5035 - 6.1.1 ^②	4°C / -10°C ^{③,④}	5035 - Section 7.2	48 H / 14 D ^⑤	Any recognized VOC Method
	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{②,⑥,⑦}	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{⑥,⑦}	NaHSO ₄ / 4°C	5035 - Section 7.2 ^⑤	48 H / 14 D ^⑤	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1 ^{②⑥⑦}	4°C / -10°C ^{③,④}	5035 - Section 7.2 ^⑤	48 H / 14 D ^⑤	Any recognized VOC Method
>200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3 ^⑥	EnCore or equivalent	5035 - 6.1.3 ^{⑥,⑦}	4°C	5035 - Sections 7.3.2 & 7.3.3 ^⑥	48 H / 14 D ^⑤	Any recognized VOC Method
>200 ug/kg ^⑧	Coring Device	5035 - Section 6.2.2.3 ^⑧	Glass Vial w/ PTFE-silicone Septum	6.1.3 ^⑧	Methanol/PEG + 4°C	5035 - Section 7.3.4	14 D	Any recognized VOC Method
	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum	6.1.2	4°C	5035 - Sections 7.3.1 - 7.3.3	14 D	Any recognized VOC Method
Oily Waste	Conventional Devices	5035 - Section 6.2.4.2	Glass w/ PTFE-silicone Septum	6.1.4	4°C	5035 - Sections 7.4.1 - 7.4.2	14 D	Any recognized VOC Method
	Conventional Devices	5035 - Section 6.2.4.1	Glass w/ PTFE-silicone Septum	6.1.4	Methanol/PEG + 4°C	5035 - Sections 7.4.3	14 D	Any recognized VOC Method
Dry Wt.	Conventional Devices		Glass with Teflon liner		4°C	5035 - Section 7.5		
Soil Screen	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum		4°C	5035 - Section 7.1	14 D	Any recognized VOC Method

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

- ① Maximum time allowable from time/date of collection to sample analysis.
- ② Eliminate 6.1.1.2; use only organic-free water.
- ③ Contents of sampling device must be transported to the laboratory at 4°C and stored at -10°C.
- ④ In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle to maximize surface area.
- ⑤ Maximum allowable time at 4°C is 48 hours; maximum allowable time to sample analysis is 14 days (from time of sample collection).
- ⑥ Conducted in the laboratory.
- ⑦ Entire contents of sampling device are extruded into the sample analysis vial containing the appropriate solvent.
- ⑧ Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 ug/kg.
- ⑨ Methanolic preservation in the field is not recommended, but may be used if approved by an DEP program.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
MICROBIOLOGICAL-BACTERIA	Cool < 10°C, Na ₂ S ₂ O ₃ ⁵			P or G
Total Coliforms, fecal coliforms & <i>E. coli</i> in drinking water	Cool < 10°C ⁶ , Na ₂ S ₂ O ₃ ⁵	30 Hours ⁷		P or G
Total coliforms and fecal coliforms in source water Heterotrophic bacteria in drinking water	Cool < 10°C, Na ₂ S ₂ O ₃ ⁵	8 hours		P or G
Gross Alpha	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Gross beta	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Strontium-89	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Strontium-90	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Radium-226	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Radium-228	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Cesium-134	Concentrated HCl to pH <<2 ^{8,9}	6 mo		P or G
Iodine-131	None	8 da		P or G
Tritium	None	6 months		G
Uranium	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Photon emitters	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Asbestos	Cool 4°C	48 hours		P or G
Bromate	Ethylenediamine (50mg/L)	28 days		P or G
Cyanide	Cool, 4°C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		P or G
Nitrate	Cool, 4°C	48 hours		P or G
Nitrate (chlorinated source)	Cool, 4°C	14 days		P or G
Odor	Cool 4°C	24 hours		G
502.2	Sodium Thiosulfate or Ascorbic Acid, 4°C HCl pH<2 if Ascorbic Acid is used	14 days		Glass with PTFE Lined Septum

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
504.1	Sodium Thiosulfate Cool, 4°C,	14 days	4°C, 24 hours	Glass with PFTE-Lined Septum
505	Sodium Thiosulfate Cool, 4°C	14 days (7 days for Heptachlor)	4°C, 24 hours	Glass with PFTE-Lined Septum
506	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
507	Sodium Thiosulfate Cool, 4°C, Dark	14 days (see method for exceptions)	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
508	Sodium Thiosulfate Cool, 4°C, Dark	7 days (see method for exceptions)	4°C, dark, 14 days	Glass with PFTE-lined Cap
508A	Cool, 4°C	14 days	30 days	Glass with PFTE-lined Cap
508.1	Sodium Sulfite, HCl pH<2, Cool, 4°C	14 days (see method for exceptions)	30 days	Glass with PFTE-lined Cap
515.1	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 28 days	Amber Glass with PFTE-lined Cap
515.2	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.3	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.4	Sodium Sulfite, HCl pH<2, Cool, ≤10°C for first 48 hours ≤6°C thereafter, Dark	14 days	≤0°C, 21 days	
524.2	Ascorbic Acid, HCl pH<2, Cool 4°C	14 days		Glass with PFTE-lined Septum
525.2	Sodium Sulfite, Dark, Cool, 4°C, HCl pH<2	14 days (see method for exceptions)	≤ 4°C, 30 days from collection	Amber Glass with PFTE-lined Cap
531.1, 6610	Sodium Thiosulfate Monochloroacetic acid, pH<3, Cool, 4°C	Cool 4°C, 28 days		Glass with PFTE-lined Septum
531.2	Sodium Thiosulfate, Potassium Dihydrogen Citrate buffer to pH 4,	28 days		

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
	dark, ≤10°C for first 48 hr, ≤6°C thereafter			
547	Sodium Thiosulfate Cool, 4°C	14 days (18 mo. frozen)		Glass with PTFE-lined Septum
548.1	Sodium Thiosulfate (HCl pH 1.5-2 if high biological activity), Cool, 4°C, Dark	7 days	≤4°C 14 days	Amber Glass with PTFE-lined Septum
549.2	Sodium Thiosulfate (H ₂ SO ₄ pH<2 if biologically active), Cool, 4°C, Dark	7 days	21 days	High Density Amber Plastic or Silanized Amber Glass
550, 550.1	Sodium Thiosulfate Cool, 4°C, HCl pH<2	7 days	550, 30 days 550.1, 40 days Dark, 4°C	Amber Glass with PTFE-lined Cap
551.1	Sodium Thiosulfate, Sodium Sulfite, Ammonium Chloride, pH 4.5-5.0 with phosphate buffer, Cool, 4°C	14 days		Glass with PTFE-lined Septum
552.1	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 48 hours	Amber Glass with PTFE-lined cap
552.2	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 7 days ≤-10°C 14 days	Amber Glass with PTFE-lined cap
555	Sodium Sulfite, HCl, pH ≤ 2, Dark, Cool 4°C	14 days		Glass with PTFE-lined cap
1613B	Sodium Thiosulfate, Cool, 0-4°C, Dark		Recommend 40 days	Amber Glass with PTFE-lined Cap

¹ Preservation, when required, must be done immediately upon sample collection.

² Stated values are the maximum regulatory holding times. Sample processing must begin by the stated time.

³ Stated time is the maximum time a prepared sample extract may be held before analysis.

⁴ (P) polyethylene or (G) or glass. For microbiology, plastic sample containers must be made of sterilizable materials (poly-propylene or other autoclavable plastic).

⁵ Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

- ⁶ Temperature requirement applies only to source water samples, however once received by the laboratory, if sample processing does not begin on the same working day, samples must be refrigerated.
- ⁷ If samples are analyzed after 30 hours, but within 48 hours of collection, the laboratory is to indicate in the analytical report that the data may be invalid because of excessive delay in sample processing. No samples received after 48 hours are to be accepted or analyzed for compliance with the regulations of the Department of Environmental Protection or the Department of Health.
- ⁸ It is recommended that the preservative be added at the time of collection unless suspended solids activity is to be measured. It is also recommended that samples be filtered, if suspended or settleable solids are present, prior to adding preservative, at the time of collection. However, if the sample has to be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.
- ⁹ If HCl is used to acidify samples, which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

Table FS 1000-9
Containers, Preservation and Holding Times for Biosolids Samples and Protozoans

<i>ANALYTE NAME</i>	<i>CONTAINER</i>	<i>PRESERVATION</i>	<i>MAX HOLDING TIME</i>
Fecal Coliform	Plastic or Glass	Cool 4°C	24 hours
Salmonella	Plastic or Glass	< 10°C	24 hours
Enteric Viruses	Plastic or Glass	Up to 25°C	2 hours
Enteric Viruses	Plastic or Glass	2 to 10°C	48 hours
Specific Oxygen Uptake Rate	Plastic or Glass	None	As Soon As Possible
Helminth OVA	Plastic or Glass	< 4°C (Do not Freeze)	24 hours
Cryptosporidium/Giardia	Plastic or Glass	0 - 8°C (Do not Freeze)*	96 Hours
Total Solids	Plastic or Glass	≤6°C (Do not Freeze)	7 days
Metallics	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	
Other Inorganic Pollutants	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	

***Dechlorinate bulk samples when applicable**

Table FS 1000-10
Container Materials, Preservation, and Holding Times for Fish and Shellfish

Analyte	Matrix	Sample Container	Field (Transport to Lab)		Laboratory	
			Preservation	Maximum Shipping Time	Storage	Holding Time
	Whole Organism (Fish, shellfish, etc.	Foil-wrap each organism (or composite for shellfish) and transport in waterproof plastic bag	Cool in wet ice or: ----- Freeze on dry ice	24 hours		
Mercury	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	28 days
Other metals	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE		48 hours	Freeze at <-20°C	6 months
Organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	1 year
Dioxin	Tissue (fillets and edible portions, homogenates)	Amber containers: Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	30 days until extraction, 15 days after extraction
Lipids	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	1 year

PTFE = Polytetrafluoroethylene (Teflon)

Table FS 1000-11
Holding Times for SPLP or TCLP Extraction, Sample Preparation and Determinative Analysis

Holding Time (Days)				
	From: Field Collection	From: SPLP or TCLP Extraction	From: Preparative Extraction	Total Elapsed Time
	To: SPLP or TCLP Extraction	To: Preparative Extraction	To: Determinative Analysis	
Volatiles	14	NA	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Mercury	180	NA	180	360

NA – Not Applicable

Table FS 1000-12
Preventive Maintenance Tasks

INSTRUMENT/ACTIVITY	FREQUENCY
REFRIGERATORS, INCUBATORS, OVENS	
Clean interior	Monthly
Check thermometer temperature against certified thermometer or equivalent	Annually
ANYTICAL BALANCES	
Clean pan and compartment	Daily ¹
Check with Class S weights	Monthly
Manufacturer cleaning and calibration	Annually
pH AND ION SELECTIVE ELECTRODES	
PROBE	
Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	Daily, Replace as necessary
Check response time	Daily ¹
METER	
Check batteries and electronics for loose connections and cracked leads	Daily ¹ , Replace as necessary
TURBIDIMETER	
Clean instrument housing	Monthly
Clean cells	Daily ¹
CONDUCTIVITY METER	
Check batteries and probe cables	Daily ¹
Replatinize Probe	Per manufacturer's recommendations
DISSOLVED OXYGEN METERS	
PROBE	
Check membrane for deterioration; check filling solution	Daily ¹ , Replace as necessary
METER	
Battery level and electronics checked	Daily ¹ , Replace as necessary
THERMOMETERS	
Check for cracks and gaps in the mercury	Daily ¹ , Replace as necessary
TEMPERATURE PROBE	
Check connections, cables	Daily ¹
Check against calibrated thermometer	Daily ¹
AUTOMATIC SAMPLE COLLECTION SYSTEMS (e.g., ISCO, Sigma)	
Check sampler operation (forward, reverse, automatic through three cycles of the purge-pump-purge cycle)	Daily ¹ Prior to Sampling Event
Check purge-pump-purge cycle when sampler is installed	Daily ¹ Prior to Sampling Event
Check the flow pacer that activates the sampler to assure proper operation	Daily ¹ Prior to Sampling Event
Check desiccant	Daily ¹ , Replace as Necessary
Check batteries	Daily ¹ , Replace as Necessary
Check pumping rate against manufacturer's specifications	Daily ¹ , Replace as Necessary

¹Daily is defined as prior to use or a 12-hour period if equipment is run continuously

Figure FS 1000-1
Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump

