
 <b>GUAM WATERWORKS AUTHORITY</b>	<b>STANDARD OPERATING PROCEDURE</b>	No.	SOP-1300-LAB-001
	<b>Sample Collection, Preservation and Storage</b>	Effective Date	<b>4.9.2022</b>
		Final Approver	 Miguel C. Bordallo, P.E. General Manager
		Revision Letter	B

## 1.0 Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide verifiable direction for proper water and wastewater sample collection, including its handling, preservation, storage, and transportation, for GWA compliance with regulations. The objective of GWA compliance sampling is to collect representative samples and to ensure that collected samples are handled in a manner that does not significantly change the composition of the sample between collection and analysis, and therefore results are valid.

## 2.0 Scope

This SOP applies to water and wastewater sample collection by GWA lab personnel and other authorized individuals. Samples are for compliance monitoring but may include special sample collection in the interest of GWA.

Water samples include the following: potable water (routine, repeat, complaint); finished water; source water. Locations and frequencies are set according to regulatory requirements.

Wastewater samples include the following: treatment plant influent, effluent, and discharge receiving waters. Locations and frequencies are specified in each NPDES permit.

## 3.0 Policy

Laboratory Management plans for microbial and chemical analyses in compliance with required Safe Drinking Water Act (SDWA), Guam Primary Drinking Water Regulations (GPDWR), Clean Water Act (CWA) and Clean Air Act (CAA) regulatory monitoring programs. Accordingly, they assess the selection of sampling sites, analyses required, manpower, and equipment needed. Creating an effective sampling plan takes into account known sources of pollution, impacts of seasons, weather, and other variables. The selection and sampling frequency of fixed sites for drinking water sampling of the potable water distribution system is done in conjunction with the Guam Environmental Protection Agency (GEPA) Safe Drinking Water Program. These sites are subject to change with the approval of GEPA and were initially set up to meet the Stipulated Order for Preliminary Relief filed in the District Court of Guam on June 05, 2003, but have subsequently come under the amendments to the SDWA and GPDWR.

Wastewater sampling sites are set according to the GWA NPDES Permit Requirements as amended and renewed from time to time.

## 4.0 Definitions

- 4.1. **Chain of Custody Document:** A chronological documentation or paper trail that records the sequence of custody, control, transfer, analysis, and disposition of materials.
- 4.2. **Complaint Potable Sample:** A sample collected from a consumer's residence upon receiving a water quality complaint from the consumer. The sample is tested for bacteria and other contaminants associated with the water quality complaint.

- 4.3. **Holding Time:** The length of time a sample can be held after collection and prior to analysis without significantly affecting the water quality properties. Holding times vary with the analyte, sample matrix, and analytical methodology.
- 4.4. **Personal Protective Equipment (PPE):** Equipment is worn to minimize exposure to hazards that cause serious workplace injuries and illnesses.
- 4.5. **Preservative:** Chemical reagent or storage condition which preserves and maintains the water sample state at which it was sampled.
- 4.6. **Routine Potable Sample:** A bi-weekly sampling route of distribution system sites selected for system evaluation (bacteriological test). Distribution sample locations include dead-end sections to demonstrate bacteriological quality throughout the network and to ensure that localized contamination does not occur through cross-connections, breaks in the distribution lines, or reduction of positive pressure. Sample locations may include: 1) public sites (government buildings, schools); 2) commercial sites (stores, restaurants, gas stations, and office buildings); and 3) private residences. These sample sites and frequencies are approved by the GEPA and are representative of the distribution system based on the population served.
- 4.7. **Repeat Potable Sample:** A set of repeat samples collected within 24 hours when a total coliform test is positive. All total coliform positive cultures are tested for the presence of either fecal coliforms or E. coli. At least one repeat sample from the sampling tap where the original total coliform positive sample was taken and at least one sample at a tap within five service connections both upstream and downstream of the original sampling site must be taken.
- 4.8. **Sample Collector:** Individual with training and/or experience in water sample collection who is authorized by GWA and Guam Environmental Protection Agency. These individuals may be any GWA employee, client representative (e.g., GIAA), or contractor representative.

## 5.0 Roles and Responsibilities

5.1	General Manager (GM)	Approves this SOP and all its subsequent changes.
5.2	AGM Compliance & Safety (AGM-C&S)	Oversee the development, revision, and implementation of this SOP as the Policy Owner.  Monitors GWA Laboratory and its implementation to this SOP.  Endorses to SOP Committee any amendment(s) needed to this SOP.
5.3	Utility Compliance Lab Administrator	Reviews this SOP annually and recommends necessary changes to the AGM-C&S for consideration.
5.4	Lab Tech Supervisor / Lab Tech Leader	Ensures Sample Collectors adhere to the sampling procedures as outlined herein when collecting samples.

5.5	Lab Certified Sample Collector	Adheres to sampling procedures as outlined herein when collecting samples.  Collects water samples from designated locations as scheduled and assigned.
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## 6.0 Procedure Description

- 6.1. **Method Summary:** The GWA Compliance Water and Wastewater samples are collected, transported, stored, and preserved as described in *40 CFR, Part 136*, and *Part 141*, respectively.

Water samples collection complies with the *23<sup>rd</sup> Edition of Standard Methods for the Examination of Water and Wastewater § 1020 QUALITY ASSURANCE and §1060 COLLECTION AND PRESERVATION OF SAMPLES: the Monitoring and Analytical Requirement in 40 CFR, Part 141* and procedures in EPA's *Quick Guide to Drinking Water Sample Collection*.

Wastewater samples collection complies with the *23<sup>rd</sup> Edition of Standard Methods for the Examination of Water and Wastewater § 1020 QUALITY ASSURANCE and § 1060 COLLECTION AND PRESERVATION OF SAMPLES: the guidelines Establishing Test Procedures or the Analysis of Pollutants in 40 CFR, Part 136*. In addition, sample collections are based on the requirements of the NPDES permits.

- 6.2. **Health and Safety Warnings:** Sample bottles may contain hazardous materials. Sample collectors must handle and fill sample bottles with care to prevent fumes exposure or physical contact with the hazardous material. Proper PPE must be used during collection as stated in the *23<sup>rd</sup> Edition of Standard Methods for the Examination of Water and Wastewater § 1090 LABORATORY OCCUPATIONAL HEALTH AND SAFETY*.

Wastewater contains pathogens, including infectious agents. Appropriate safety precautions must be exercised when handling wastewater as stated in the *23<sup>rd</sup> Edition of Standard Methods for the Examination of Water and Wastewater § 1090 LABORATORY OCCUPATIONAL HEALTH AND SAFETY*.

- 6.3. **Chain-of-Custody Form (CoC):** Sample collectors or designees must prepare a CoC prior to sampling. A CoC documents field activities and laboratory sample handling activities from the time of collection through analysis. It ensures sample integrity from collection through data reporting. It also demonstrates sample control when the data are to be used for regulatory reporting or litigation. (See the *23<sup>rd</sup> Edition of Standard Methods for the Examination of Water and Wastewater § 1020 QUALITY ASSURANCE and § 1060 COLLECTION AND PRESERVATION OF SAMPLES*.) Where litigation is not involved, chain-of-custody procedures are essential for routine control of the required QA/QC (Quality Assurance / Quality Control) of the samples. Chain-of custody information must include:

- 6.3.1. Unique sample or log number;
- 6.3.2. Date and time of sample collection;

- 6.3.3. Sample information (sample point name/location, system/plant name and sample source, and type);
  - 6.3.4. Preservative used;
  - 6.3.5. Analyses required;
  - 6.3.6. Name of sample collectors;
  - 6.3.7. Date and time of sample transfer at each sample handoff with name and initials of the person receiving the sample;
  - 6.3.8. Pertinent field data; and
  - 6.3.9. Comments.
- 6.4. **Handling & Preservation:** Sample collection must be performed by authorized collectors.
- 6.4.1. Water sample handling and preservation adhere to requirements in Table 1060:1 of the *Standard Methods for the Examination of Water and Wastewater*, 23<sup>rd</sup> Edition, American Public Health Association (APHA), Water Environment Federation (WEF) and American Water Works Association (AWWA), 2019 and Table IV-6 of the *Manual for the Certification of Laboratories in Analyzing Drinking Water: Criteria and Procedures Quality Assurance*, 5<sup>th</sup> Edition, EPA, January 2005. These tables contain information for the types of sample containers, preservatives, maximum holding times, and the volumes of sample needed for each parameter. See **Attachments 1 & 2**.
  - 6.4.2. Wastewater sample handling and preservation adheres to requirements in Table II of 40CFR, Part 136.3 I. **Attachment 3**.
  - 6.4.3. Sample preservation may be achieved by using pre-preserved bottles or containers obtained from the laboratory. Sample preservation may also be achieved in the field by following the protocols as stated in the respective SOPs and EPA methods.
- 6.5. **Interferences:** Sampling techniques should be properly followed to avoid cross-contamination.
- 6.5.1. **Water bacteriological testing:** Avoid sampling from leaking taps. Remove devices such as aerators on faucets before sampling. Always ensure that holding containers and work areas are properly sterilized before using.
  - 6.5.2. **Water/wastewater chemical testing:** Ensure sample containers and preservatives are appropriate for the chemical to be tested. Use high purity grade preservatives.
  - 6.5.3. **Wastewater:** Ensure that all composite samplers are in good order and are functioning correctly. Check the sample timer and sample volumes regularly.

6.6. **Apparatus and Materials:** The following lists the apparatus and materials needed for sample collection:

- 6.6.1. Sample containers pre-sterilized (containing preservatives where required);
- 6.6.2. Cooler with ice packs;
- 6.6.3. Temperature Monitoring Devices;
- 6.6.4. Free and Total Chlorine Test Kit (for water distribution sampling);
- 6.6.5. Autosamplers (wastewater sampling); and
- 6.6.6. Other equipment as specified in “*STANDARD METHODS FOR THE EXAMINATION OF WATER AND WASTEWATER, 23<sup>rd</sup> Edition, 2019*” and/or U.S. Environmental Protection Agency methods.

6.7. **Quality Control and Limits:** QC activities are performed by sample collectors to ensure sample integrity.

- 6.7.1. Sample collectors wear gloves when collecting samples;
- 6.7.2. Field control blanks are included during water sampling, specifically for volatile organic compounds;
- 6.7.3. Bacteriological samples are placed in sterile containers, of which sterility is confirmed as described in *Section 13* of the *GWA Compliance Lab Quality Assurance Plan*;
- 6.7.4. Temperature checks are performed after samples have arrived from the field;
- 6.7.5. Samples are collected in appropriate containers with respective preservatives;
- 6.7.6. Collected samples are placed in coolers with ice packs; and
- 6.7.7. Wastewater QA/QC sampling follows the QA/QC procedures for each appropriate type of sample as specified in “*STANDARD METHODS FOR THE EXAMINATION OF WATER AND WASTEWATER*” – (23<sup>rd</sup> Edition; 2019). These include:
  - 6.7.7.1. Autosamplers are regularly inspected and maintained; and
  - 6.7.7.2. Samples are collected as specified in the NDPES permit, i.e., sample type and specified locations.

6.8. **Bacteriological Contaminant Sampling**

6.8.1. **Routine Potable Water Sampling:**

- 6.8.1.1. If the water sample is to be taken from a distribution-system tap without attachments, select a tap that is supplying water from a service pipe directly connected with the main, and is not, for example, served from a cistern, filtration systems, water softener or any other point of use pretreatment device or storage tank.
  - 6.8.1.2. Open tap fully and let the water run to waste for 2 to 5 minutes (for a time sufficient to permit clearing of the service line and receive water from the water main), then reduce water flow to permit filling of the bottle without splashing.
  - 6.8.1.3. If tap cleanliness is questionable unless it can be thoroughly cleaned to the satisfaction of the individual collecting the sample, choose another tap prior to routine disinfection. If a questionable tap is required for special sampling purposes, use extra disinfection of the faucet (inside and outside) by repeatedly applying a solution of sodium hypochlorite (100 mg NaOCl/L) to the faucet before sampling:
    - 6.8.1.3.1. Let the water run for an additional 2 to 3 minutes after treatment.
    - 6.8.1.3.2. Do not sample from leaking taps that allow water to flow over the outside of the tap.
  - 6.8.1.4. If sampling from a mixing faucet, remove faucet attachments such as a screen or splash guard, run hot water for 2 minutes, then cold water for 2 to 5 minutes, before collecting the sample.
  - 6.8.1.5. Keep sample bottles closed until it is time to be filled.
  - 6.8.1.6. Check chlorine residuals before unsealing sample containers to ensure that the service line has cleared.
  - 6.8.1.7. Remove cap, being careful not to contaminate the inner surface of cap and neck of the bottle.
  - 6.8.1.8. Fill the container to the mark without rinsing and replace the cap immediately.
- 6.8.2 **Repeat Potable Water Sampling:** When a routine sample is a total coliform positive, within 24 hours of a positive result, collect at least one repeat sample from the sampling tap where the original total coliform positive sample was taken and at least one from both upstream and downstream locations (GEPA approved/specified) of the original sampling site. Continued repeat sampling is performed until analyses yield a negative result. If chlorine residuals are below 0.2 ppm, resamples must also be collected along with an upstream and downstream sample. When corrective actions have been made (usually chlorinators repairs), and there is a sufficient amount of chlorine detected in the

system (0.2 ppm to 4 ppm) then sampling may discontinue. Full chain of custody records must be kept.

6.8.3 **Complaint Potable Water Sampling:** When a water quality complaint is received, water sample collection is scheduled (day and time) with the consumer. Prepare appropriate sample bottles. Complaint sample custody sheets must contain the following information:

6.8.3.1 System name;

6.8.3.2 Consumer's name;

6.8.3.3 Telephone no.;

6.8.3.4 Address or any contact information;

6.8.3.5 Nature of complaint;

6.8.3.6 Date of the complaint;

6.8.3.7 Time sampled;

6.8.3.8 Chlorine residual;

6.8.3.9 Turbidity (if applicable);

6.8.3.10 Who reported the complaint;

6.8.3.11 Investigator;

6.8.3.12 Other pertinent information used for collecting the sample; and

6.8.3.13 Analyses that are not performed at the GWA Laboratory are contracted out to a GEPA certified laboratory to perform the required analysis. Proper documentation must be complete (chain of custody and shipping papers if necessary). The complainant is notified of the results after confirmation by the responding lab technician or the lab supervisor. If GWA's line is found to be the problem, the lab coordinates with other divisions to rectify the problem.

## 6.9 Chemical Contaminant Sampling:

6.9.1 **Asbestos and classical chemistry constituents (wet chemistry):** Rinse the bottle and cap with sample water three times. Fill the bottle within two inches from the top and cap.

6.9.2 **Disinfection By-products:**

6.9.2.1 **HAA5s:** Do not rinse vials as they contain preservatives. Slowly fill vials through gentle flow ensuring that no air bubbles pass through the samples while filling the vials. Fill vials to the point where a

meniscus is formed at the mouth, almost overflowing. Seal vials with no headspace by allowing excess water to spill down the sides of the vial.

6.9.2.2 **TTHMs:** Do not rinse vials as they contain preservatives. Slowly fill vials through gentle flow ensuring that no air bubbles pass through the samples while filling the vials. Fill vials to the point where a meniscus is formed at the mouth, almost overflowing. Seal vials with no headspace by allowing excess water to spill down the sides of the vial.

6.9.3 **Inorganic Chemicals and radionuclides:** Do not rinse bottles as they contain preservatives. Carefully uncap the bottle, fill it within two inches from the top, and recap.

6.9.4 **Lead and Copper:**

6.9.4.1 Samples must be collected after an extended period of stagnant water conditions (no water usage) within interior piping. A minimum of 6-8 hours of no water usage must be achieved prior to sampling.

6.9.4.2 Collect samples from a kitchen or bathroom cold water faucet. Do not intentionally flush the waterline before the start of the 6-hour period.

6.9.4.3 Place a wide-mouth 1-liter bottle directly under the faucet and gently open the cold-water tap filling to the 1-liter mark. Preserve samples by adding nitric acid (HNO<sub>3</sub>) to pH <2 (around 2 mL 1:1 nitric acid solution). Unpreserved samples must be delivered to the laboratory within 14 days.

6.9.5 **PFAS:**

6.9.5.1 **Sampling Precautions:** Avoid wearing clothing or boots containing Gore-Tex or using materials containing Tyvek. Avoid using cosmetics, moisturizers, heavy fabric softeners on clothes the day of the sampling. Sample PFAS first if your cooler contains other sample collection bottles. Do not use chemical ice packs, felt-tip pens, and permanent markers, adhesive products, plastic binders, and hardcovers. The sampler must wash hands prior to wearing Nitrile gloves in order to limit contamination.

6.9.5.2 **Sampling Procedure:** Flush cold water sampling line for approximately 15 minutes and slow the water stream before sampling. Fill the sample bottle to the neck, replace the cap and tighten leaving space for the preservative to mix. Do not overfill and do not flush out the preservative. Invert 5 times to mix.

6.9.5.3 **\*PFAS** field blanks are usually provided (1 empty bottle and 1 filled with water). Fill the empty bottle with the contents of the filled bottle at the sampling site.

- 6.9.5.4 **Shipping Procedure:** Eurofins Labs provide PFAS coolers specially prepared for shipping. Place the two HDPE plastic bottles into one Ziplock bag and expunge air in the bag before completely sealing. Line cooler with plastic liner, then place samples in Ziplock into the cooler placing bubble wrap between bottles. Fill the lined cooler with wet, loose ice cubes.
- 6.9.6 **Synthetic Organic Compounds:** Do not rinse bottles as they contain preservatives. Allow the system to flush until water temperature has stabilized (usually about two minutes). Carefully fill bottles to within two inches from the top unless containers are vial. Fill vials to the point where a meniscus is formed at the mouth, almost overflowing. Seal vials with no headspace by allowing excess water to spill down the sides of the vial.
- 6.9.7 **UCMR Contaminants:** The sample collection procedure will depend on contaminants being monitored. Instructions will be provided when available.
- 6.9.8 **Volatile Organic Compounds:** Do not rinse vials as they contain preservatives. Slowly fill vials through gentle flow ensuring that no air bubbles pass through the samples while filling the vials. Fill vials to the point where a meniscus is formed at the mouth, almost overflowing. Seal vials with no headspace by allowing excess water to spill down the sides of the vial. Invert the vials several times to check that there are no bubbles.

### 6.10 Wastewater Sampling:

- 6.10.1 Weekly sample collections are performed by treatment plant operators in accordance with the treatment plant's procedures.
- 6.10.2 Quarterly and annual sample collection (offshore sampling):
  - 6.10.2.1 Follow water sampler manual for the proper use and collect samples at specified locations noted in the field sheets per NDPES permits; and
  - 6.10.2.2 Fill containers for each contaminant to be tested as described in Section 6.9.

## 7.0 Document Approvals

Role	Position	Name of Approver	Approval Signature	Date Approved
Authors	Assistant General Manager - Compliance and Safety (AGM-C&S); and	Paul J. Kemp, M. S.; and	Approval on File	On File
	Utility Compliance Lab Administrator	Jennifer O. Cruz, M.S.,		
Policy Owner	Assistant General Manager for Compliance & Safety (AGM-C&S)	Paul J. Kemp, M. S,	Approval on File	On File
Final Approver	General Manager	Miguel C. Bordallo, P.E.	Page 1	Page 1

In accordance with existing Guam and Federal laws, the contents of this SOP were reviewed thoroughly by its policy owner and was found to be:

☒ appropriate for publication on the GWA website without compromising the security of GWA's system or the public's health and safety.

☐ not appropriate for publication on the GWA website because it might jeopardize the security of GWA's system or the public's health and safety.

## 8.0 Records of Revisions

All suggestions for improvement shall be directed to the Policy Owner indicated below. The Policy Owner will consider input received, develop recommendations on how to address the suggestions, and obtain authorization to make the recommended changes. Updates, revisions, corrections, and waivers to this SOP shall be made in writing and be approved by the GM.

8.1 Policy Owner: Assistant General Manager for Compliance & Safety (AGM-C&S)

8.2 Authorization: General Manager

Effective Date	Revision Letter	Document Author	Description of Change
Page 1	B	Paul J. Kemp, M.S, Jennifer O. Cruz, M.S.	Amended Policy/Procedure

## 9.0 References

- 9.1 American Public Health Association (APHA), Water Environment Federation (WEF) and American Water Works Association (AWWA) "STANDARD METHODS FOR THE EXAMINATION OF WATER AND WASTEWATER", 23<sup>rd</sup> Edition, 2019
- 9.2 APHA, WEF, AWWA, STANDARD METHODS ONLINE (2022)  
<<https://www.standardmethods.org/>>
- 9.3 Eurofins Eaton Analytical," EPA 537/537.1 PFAS Sampling Instructions".

- 9.4 GWA Lab, SOP # 3 - Sample Collection, Preservation, and Storage
- 9.5 U.S. Environmental Protection Agency, Microbiological Methods for Monitoring the Environment, EPA-600/8-78-017, December 1978.
- 9.6 U.S. Environmental Protection Agency, National Primary Drinking Water Regulations, 40 CFR Part 141, "Analytical Methods for Regulated Drinking Water Contaminants," 12/5/94.
- 9.7 U.S. Environmental Protection Agency, Manual for the Certification of Laboratories Analyzing Drinking Water, Fourth Edition, EPA 815-B-97-001, March 1997.
- 9.8 U.S. Environmental Protection Agency, Quick Guide to Drinking Water Sample Collection, Second Edition Update, September 2016.
- 9.9 <<https://www.osha.gov/personal-protective-equipment>>

**Attachment 1: Sample Containers, Preservation, and Holding Times for Parameters**

**Table IV-6 Sample Containers, Preservation and Holding Times for Regulated Parameters**

Parameter/ Method	Preservative	Sample Holding Time	Extract Holding Time and Storage Conditions	Suggested Sample Size	Type of Container
Metals (except Hg)	HNO <sub>3</sub> pH<2	6 months		1 L	Plastic or Glass
Mercury	HNO <sub>3</sub> pH<2	28 days		100 mL	Plastic or Glass
Alkalinity	Cool, 4C	14 days		100 mL	Plastic or Glass
Asbestos	Cool, 4C	48 hours		1 L	Plastic or Glass
Chloride	none	28 days		100 mL	Plastic or Glass
Residual Disinfectant	none	immediately		200 mL	Plastic or Glass
Color	Cool, 4C	48 hours		100 mL	Plastic or Glass
Conductivity	Cool, 4C	28 days		100 mL	Plastic or Glass
Cyanide	Cool, 4C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		1 L	Plastic or Glass
Fluoride	none	1 month		100 mL	Plastic or Glass
Foaming Agents	Cool, 4C	48 hours			
Nitrate (chlorinated)	Cool, 4C non-acidified	14 days		100 mL	Plastic or Glass
Nitrate (non chlorinated)	Cool, 4C, non-acidified	48 hours		100 mL	Plastic or Glass
Nitrite	Cool, 4C	48 hours		100 mL	Plastic or Glass
Nitrate+ Nitrite	H <sub>2</sub> SO <sub>4</sub> pH<2	28 days		100 mL	Plastic or Glass
Odor	Cool, 4C	24 hours		200 mL	Glass
pH	none	immediately		25 mL	Plastic or Glass
o-Phosphate	Cool, 4C	48 hours		100mL	Plastic or Glass

**Attachment 1: Sample Containers, Preservation, and Holding Times for Parameters (Cont.)**

Parameter/ Method	Preservative	Sample Holding Time	Extract Holding Time and Storage Conditions	Suggested Sample Size	Type of Container
Silica	Cool, 4C	28 days		100 mL	Plastic
Solids (TDS)	Cool, 4C	7 days		100 mL	Plastic or Glass
Sulfate	Cool, 4C	28 days		100 mL	Plastic or Glass
Temperature	none	immediately		1 L	Plastic or Glass
Turbidity	Cool, 4C	48 hours		100 mL	Plastic or Glass
502.2	Sodium Thiosulfate or Ascorbic Acid, 4C, HCl pH<2	14 days		40-120 mL	Glass with PTFE Lined Septum
504.1	Sodium Thiosulfate Cool, 4C,	14 days	4C, 24 hours	40 mL	Glass with PTFE Lined Septum
505	Sodium Thiosulfate Cool, 4C	14 days (7 days for Heptachlor)	4C, 24 hours	40 mL	Glass with PTFE Lined Septum
506	Sodium Thiosulfate Cool, 4C, Dark	14 days	4C, dark 14 days	1 L	Amber Glass with PTFE Lined Cap
507	Sodium Thiosulfate Cool, 4C, Dark	14 days(see method for exceptions)	4C, dark 14 days	1 L	Amber Glass with PTFE Lined Cap
508	Sodium Thiosulfate Cool, 4C, Dark	7 days (see method for exceptions)	4C, dark 14 days	1 L	Glass with PTFE Lined Cap
508A	Cool, 4C	14 days	30 days	1 L	Amber Glass with PTFE Lined Cap
508.1	Sodium Sulfite HCl pH<2 Cool, 4C	14 days (see method for exceptions)	30 days	1 L	Glass with PTFE Lined Cap
515.1	Sodium Thiosulfate Cool, 4C, Dark	14 days	4C, dark 28 days	1 L	Amber Glass with PTFE Lined Cap

**Attachment 2: Collection and Preservation of Samples**

COLLECTION AND PRESERVATION OF SAMPLES (1060)/Collection of Samples

TABLE 1060.I. SUMMARY OF SPECIAL SAMPLING AND HANDLING REQUIREMENTS\*

Determination	Container†	Minimum Sample Size mL	Sample Type‡	Preservation§	Maximum Storage Recommended	Regulatory
Acidity	P, G(B), FP	100	g	Cool, $\leq 6^{\circ}\text{C}$	24 h	14 d
Alkalinity	P, G, FP	200	g	Cool, $\leq 6^{\circ}\text{C}$	24 h	14 d
BOD	P, G, FP	1000	g, c	Cool, $< 6^{\circ}\text{C}$	6 h	48 h
Boron	F, P (PTFE) or quartz	1000	g, c	$\text{HNO}_3$ to $\text{pH} < 2$	28 d	6 months
Bromide	P, G, FP	100	g, c	None required	28 d	28 d
Carbon, organic, total	G (B), P, FP	100	g, c	Analyze immediately; or cool $\leq 6^{\circ}\text{C}$ , and add $\text{HCl}$ , $\text{H}_3\text{PO}_4$ , or $\text{H}_2\text{SO}_4$ to $\text{pH}$	7 d	28 d
Carbon dioxide	P, G	100	g	Analyze immediately	0.25 h	N.S.
COD	P, G, FP	100	g, c	Analyze as soon as possible, or add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$ ; Cool, $\leq 6^{\circ}\text{C}$	7 d	28 d
Chloride	P, G, FP	50	g, c	None required	N.S.	28 d
Chlorine, total, residual	P, G	500	g	Analyze immediately	0.25 h	0.25 h
Chlorine dioxide	P, G	500	g	Analyze immediately	0.25 h	N.S.
Chlorophyll	P, G	500	g	Unfiltered, dark, $4^{\circ}\text{C}$	24–48 h	N.S.
				Filtered, dark, $-20^{\circ}\text{C}$ (Do not store in frost-free freezer)	28 d	
Color	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h
Specific conductance	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Cyanide Total	P, G, FP	1000	g, c	Analyze within 15 min. Add $\text{NaOH}$ to $\text{pH} > 12$ if sample is to be stored, Cool, $\leq 6^{\circ}\text{C}$ , in dark. Add thiosulfate if residual chlorine present	24 h	14 d; 24 h if sulfide present
Amenable to chlorination	P, G, FP	1000	g, c	Remove residual chlorine with thiosulfate and cool $\leq 6^{\circ}\text{C}$	stat	14 d; 24 h if sulfide present
Fluoride	P	100	g, c	None required	28 d	28 d
Hardness	P, G, FP	100	g, c	Add $\text{HNO}_3$ or $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$	6 months	6 months
Iodine	P, G	500	g	Analyze immediately	0.25 h	N.S.
Metals	P(A), G(A), FP (A)	1000	g, c	For dissolved metals filter immediately, add $\text{HNO}_3$ to $\text{pH} < 2$	6 months	6 months
Chromium VI	P(A), G(A), FP (A)	250	g	Cool, $\leq 6^{\circ}\text{C}$ , $\text{pH}$ 9.3–9.7, ammonium sulfate buffer preservative as specified in method 3500-Cr to extend to 28 days HT	28 d	28 d
Copper by colorimetry	—*	—	g, c	—	—	—
Mercury	P(A), G(A), FP(A)	500	g, c	Add $\text{HNO}_3$ to $\text{pH} < 2$ , Cool $\leq 6^{\circ}\text{C}$	28 d	28 d
Nitrogen Ammonia	P, G, FP	500	g, c	Analyze as soon as possible or add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$ , Cool, $\leq 6^{\circ}\text{C}$	7 d	28 d
Nitrate	P, G, FP	100	g, c	Analyze as soon as possible; Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h (14 d for chlorinated samples)
Nitrate + nitrite	P, G, FP	200	g, c	Add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$ , Cool, $\leq 6^{\circ}\text{C}$	1–2 d	28 d
Nitrite	P, G, FP	100	g, c	Analyze as soon as possible; Cool, $\leq 6^{\circ}\text{C}$	none	48 h
Organic, Kjeldahl	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$ , add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$	7 d	28 d
Odor	G	500	g	Analyze as soon as possible; Cool $\leq 6^{\circ}\text{C}$	6 h	24 h (EPA Manual drinking water)
Oil and Grease	G, wide-mouth calibrated	1000	g	Add $\text{HCl}$ or $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$ , Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d

**Attachment 2: Collection and Preservation of Samples (Cont.)**

COLLECTION AND PRESERVATION OF SAMPLES (1060)/Collection of Samples

TABLE 1060-I. CONT.

Determination	Container†	Minimum Sample Size mL	Sample Type‡	Preservation§	Maximum Storage Recommended	Regulatory
Organic Compounds						
MBAS	P, G, FP	250	g, c	Cool $\leq 6^{\circ}\text{C}$	48 h	48 h as per CFR 136
Pesticides*	G(S), PTFE-lined cap	1000	g, c	Cool, $\leq 6^{\circ}\text{C}$ add 1000 mg ascorbic acid/L if residual chlorine present (0.008 % sodium thiosulfate in CFR 136)	7 d	7 d until extraction; 40 d after extraction
Phenols	P, G, PTFE-lined cap	500	g, c	Cool, $\leq 6^{\circ}\text{C}$ , add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$	*	28 d until extraction, 2 d after extraction
Purgeables* by purge and trap	G, PTFE-lined cap	2×40	g	Cool, $\leq 6^{\circ}\text{C}$ ; add HCl to $\text{pH} < 2$ ; add 1000 mg ascorbic acid/L if residual chlorine present (0.008% sodium thiosulfate in CFR 136)	7 d	14 d
Base/neutral & acids	G(S) amber	1000	g, c	Cool, $\leq 6^{\circ}\text{C}$ , 0.008 % sodium thiosulfate in CFR 136 if chlorine is present	7 d	7 d until extraction; 40 d after extraction
Oxygen, dissolved	G, BOD bottle	300	g	Analyze immediately	0.25 h	0.25 h
Electrode				Titration may be delayed after acidification	8 h	8 h
Winkler				Analyze immediately	0.25 h	N.S.
Ozone	G	1000	g	Analyze immediately	0.25 h	0.25 h
pH	P, G	50	g	Analyze immediately	0.25 h	0.25 h
Phosphate	G(A)	100	g	For dissolved phosphate filter immediately; Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h as per EPA manual for DW.
Phosphorus, total	P, G, FP	100	g, c	Add $\text{H}_2\text{SO}_4$ to $\text{pH} < 2$ and cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Salinity	G, wax seal	240	g	Analyze immediately or use wax seal	6 months	N.S.
Silica	F, P (PTFE) or quartz	200	g, c	Cool $\leq 6^{\circ}\text{C}$ , do not freeze	28 d	28 d
Sludge digester gas	G, gas bottle	—	g	—	N.S.	—
Solids <sup>9</sup>	P, G	200	g, c	Cool, $\leq 6^{\circ}\text{C}$	7 d	2–7 d; see cited reference
Sulfate	P, G, FP	100	g, c	Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Sulfide	P, G, FP	100	g, c	Cool, $\leq 6^{\circ}\text{C}$ ; add 4 drops 2N zinc acetate/100 mL; add NaOH to $\text{pH} > 9$	28 d	7 d
Temperature	P, G, FP	—	g	Analyze immediately	0.25 h	0.25 h
Turbidity	P, G, FP	100	g, c	Analyze same day; store in dark up to 24 h, Cool, $\leq 6^{\circ}\text{C}$	24 h	48 h

\* For determinations not listed, use glass or plastic containers; preferably refrigerate during storage and analyze as soon as possible.

† P = plastic (polyethylene or equivalent); G = glass; G(A) or P(A) = rinsed with 1 + 1  $\text{HNO}_3$ ; G(B) = glass, borosilicate; G(S) = glass, rinsed with organic solvents or baked; FP = fluoropolymer (polytetrafluoroethylene (PTFE, Teflon) or other fluoropolymer

‡ g = grab; c = composite.

§ Cool = storage at,  $> 0^{\circ}\text{C}$ ,  $\leq 6^{\circ}\text{C}$  (above freezing point of water); in the dark; analyze immediately = analyze usually within 15 min of sample collection.

|| See citation<sup>10</sup> for possible differences regarding container and preservation requirements. N.S. = not stated in cited reference; stat = no storage allowed; analyze immediately (within 15 min).

Some drinking water (DW) and treated wastewater (WW) matrices may be subject to positive interference as a result of preservation. If such interference is demonstrable, samples should be analyzed as soon as possible without preservation. Do not hold for more than 15 minutes without demonstrating that cyanide (CN) is stable for longer periods in a specific matrix.

NOTE: This table is intended for guidance only. If there is a discrepancy between this table and the method, the information in the current method takes precedence. If performing the method for compliance purposes, be aware that alternative preservation and holding-time requirements may exist. If so, the regulatory requirements should be used.

### Attachment 3: Table II of 40CFR, Part 136.3 (e)

Table II - Required Containers, Preservation Techniques, and Holding Times			
Parameter number/name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
<b>Table IA - Bacterial Tests</b>			
1-4. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22,23</sup>
5. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
6. Enterococci	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
7. <i>Salmonella</i>	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
<b>Table IA - Aquatic Toxicity Tests</b>			
8-11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C <sup>16</sup>	36 hours.
<b>Table IB - Inorganic Tests</b>			
1. Acidity	P, FP, G	Cool, ≤6 °C <sup>18</sup>	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C <sup>18</sup>	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C <sup>18</sup>	48 hours.
10. Boron	P, FP, or Quartz	HNO <sub>3</sub> 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 <sup>18</sup>	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> 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 <sup>18</sup>	48 hours.
23-24. Cyanide, total or available (or CATC) and free	P, FP, G	Cool, ≤6 °C <sup>18</sup> , NaOH to pH >10 <sup>5,6</sup> , reducing agent if oxidizer present	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> 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 <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.
<b>Table IB - Metals <sup>7</sup></b>			
18. Chromium VI	P, FP, G	Cool, ≤6 °C <sup>18</sup> , pH = 9.3-9.7 <sup>20</sup>	28 days.
35. Mercury (CVAA)	P, FP, G	HNO <sub>3</sub> to pH <2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap <sup>17</sup>	5 mL/L 12N HCl or 5 mL/L BrCl <sup>17</sup>	90 days. <sup>17</sup>
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 <sub>3</sub> to pH <2, or at least 24 hours prior to analysis <sup>19</sup>	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C <sup>18</sup>	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C <sup>18</sup>	48 hours.
41. Oil and grease	G	Cool to ≤6 °C <sup>18</sup> , HCl or H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C <sup>18</sup> , HCl, H <sub>2</sub> SO <sub>4</sub> , or H <sub>3</sub> PO <sub>4</sub> to pH <2	28 days.
44. Orthophosphate	P, FP, G	Cool, to ≤6 °C <sup>18,24</sup>	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 <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.
49. Phosphorus (elemental)	G	Cool, ≤6 °C <sup>18</sup>	48 hours.
50. Phosphorus, total	P, FP, G	Cool, ≤6 °C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days.

### Attachment 3: Table II of 40CFR, Part 136.3 (e) (Cont.)

53. Residue, total	P, FP, G	Cool, ≤6 °C <sup>18</sup>	7 days.
54. Residue, Filterable (TDS)	P, FP, G	Cool, ≤6 °C <sup>18</sup>	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C <sup>18</sup>	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C <sup>18</sup>	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C <sup>18</sup>	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C <sup>18</sup>	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C <sup>18</sup>	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C <sup>18</sup>	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C <sup>18</sup> , 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 <sup>18</sup>	48 hours.
69. Temperature	P, FP, G	None required	Analyze within 15 minutes.
73. Turbidity	P, FP, G	Cool, ≤6 °C <sup>18</sup>	48 hours.
<b>Table IC - Organic Tests<sup>8</sup></b>			
13, 18-20, 22, 24, 25, 27, 28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH 2 <sup>9</sup>	14 days. <sup>9</sup>
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH 2 <sup>9</sup>	14 days. <sup>9</sup>
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , pH to 4-5 <sup>10</sup>	14 days. <sup>10</sup>
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	7 days until extraction, 40 days after extraction.
7, 38. Benzidines <sup>11 12</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction. <sup>13</sup>
14, 17, 48, 50-52. Phthalate esters <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup>	7 days until extraction, 40 days after extraction.
82-84. Nitrosamines <sup>11 14</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
88-94. PCBs <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup>	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
29, 35-37, 63-65, 73, 107. Chlorinated hydrocarbons <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup>	7 days until extraction, 40 days after extraction.
60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs <sup>11</sup>	G	See footnote 11	See footnote 11.
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , pH <9	1 year.
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C <sup>18</sup>	7 days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C <sup>18</sup>	24 hours.
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year.
114-118. Alkylated phenols	G	Cool, <6 °C, H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX)	G	Cool, <6 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , HNO <sub>3</sub> to pH <2	Hold at least 3 days, but not more than 6 months.
120. Chlorinated Phenolics	G, FP-lined cap	Cool, <6 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> to pH <2	30 days until acetylation, 30 days after acetylation.
<b>Table ID - Pesticides Tests</b>			
1-70. Pesticides <sup>11</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>18</sup> , pH 5-9 <sup>15</sup>	7 days until extraction, 40 days after extraction.
<b>Table IE - Radiological Tests</b>			
1-5. Alpha, beta, and radium	P, FP, G	HNO <sub>3</sub> to pH <2	6 months.
<b>Table IH - Bacterial Tests</b>			
1, 2. Coliform, total, fecal	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
3. <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
4. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
5. Enterococci	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours. <sup>22</sup>
<b>Table IH - Protozoan Tests</b>			
6. <i>Cryptosporidium</i>	LDPE; field filtration	1-10 °C	96 hours. <sup>21</sup>
7. <i>Giardia</i>	LDPE; field filtration	1-10 °C	96 hours. <sup>21</sup>

## Sample Collection, Preservation and Storage

### Attachment3: Table II of 40CFR, Part 136.3 (e) (Cont.)

<sup>1</sup> "P" is for 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.
<sup>2</sup> 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 sample (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 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 results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.24(f)(14)(iv) and (v) (volatile organics).
<sup>3</sup> 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 requirement of Table II, the Office of Hazardous Materials, Materials Transportation
<sup>4</sup> 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. Samples may be held for longer periods only if the permittee or monitoring laboratory have 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 ATP Coordinator 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 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. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0-6 °C, with minimum head space.
<sup>5</sup> ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analyses. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (23rd edition) addresses dechlorination procedures for microbiological analyses.
<sup>6</sup> Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365-09a (15). There may be interferences that are not mitigated by the analytical test methods or D7365-09a (15). Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365-09a (15) or the analytical test method must be documented along with supporting data.
<sup>7</sup> 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.
<sup>8</sup> Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
<sup>9</sup> If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.
<sup>10</sup> 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.
<sup>11</sup> 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 12, 13 (regarding the analysis of benzidine).
<sup>12</sup> 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.
<sup>13</sup> Extracts may be stored up to 30 days at <0 °C.
<sup>14</sup> For the analysis of diphenylnitrosamine, add 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> and adjust pH to 7-10 with NaOH within 24 hours of sampling.
<sup>15</sup> 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% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .
<sup>16</sup> Place sufficient ice 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, 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. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.
<sup>17</sup> 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.
<sup>18</sup> Aqueous samples must be preserved at ≤6 °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 "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three 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).
<sup>19</sup> 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.

Attachment3: Table II of 40CFR, Part 136.3 (e)(Cont.)

<sup>20</sup>	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.
<sup>21</sup>	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.
<sup>22</sup>	Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.
<sup>23</sup>	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.
<sup>24</sup>	The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus ( <i>i.e.</i> , that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection ( <i>i.e.</i> , within 15 minutes of collection).