

APPENDIX II

**REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES FOR SOLID WASTE AND WASTEWATER**

Table II-Required Containers, Preservation Techniques, and Holding Times

Parameter No./name	Matrix	Method	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Bacterial Tests:					
Coliform, total, fecal	Liquid/Solid	AOAC 986.33*, AOAC 991.14*, SM 9221C, SM 9222B, SM 9223B*, AOAC 2000.15*, AOAC/RI-PTM#060702*, AOAC/RI-PTM#110401*	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ^{22,23}
<i>E. coli</i>	Liquid/Solid	CMMEF 35.24, EPA 1603, SM 9221F, SM 9223B, SM 9225C, AOAC/RI-PTM#110402*, BAM 4*, CMMEF 8.935*, OMA 2009.02*, AOAC 991.14*, AOAC 998.08*#, SM 9222G*#	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ^{22,23}
Fecal streptococci	Liquid/Solid/Swab	SM 9230C*#	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
Enterococci	Liquid/Solid/Swab	ASTM D6503-14, ASTM D6503-99, SM 9230C*#	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
Salmonella	Liquid/Solid	AOAC 2000.07*, AOAC/RI-PT#080601*, CMMEF 37.74*, EPA 1682*, SM 9260B*, SM 9260D*, AOAC 967*, AOAC/RI-PT#010803*, AOAC/RI-PTM#071101*,	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
Inorganic Tests:					
Acidity	Liquid/Solid	EPA 305.1*, SM 2310B, SM 2310Bm*	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
Alkalinity	Liquid/Solid	ACS RGT Chemicals*, EPA 310.1*, SM 2320B, Ag HB60-23*, SM 2320B for Sludge	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
Ammonia	Liquid/Solid	EPA 350.3*, SM 4500-NH3 G, SM 4500NH3D, SM 4500NH3-Dm*	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2	28 days.
Ammonia as N	Liquid	ASTM D6919	P	Store at 4°C, Sulfuric acid to pH< 2	28 days.
Anions_IC_DW	Liquid	EPA 300.1	P, G, for Chlorite=must be opaque	50 mg/L EDA; cool to 4 °C for Chlorite	28 days; 14 days for Chlorite
Biochemical oxygen demand	Liquid/Solid	EPA 405.1, SM 5210B, 5210m*	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
Biochemical oxygen demand, carbonaceous	Liquid	EPA 405.1, SM 5210B,	P, FP G	Cool, ≤6 °C ¹⁸	48 hours.
Chemical oxygen demand	Liquid/Solid	EPA 410.4, SM 5220D*, 5220Dm*	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2	28 days.
Chloride	Liquid	ASTM D512-12(B), SM 4500CI--B	P, FP, G	None required	28 days.
Chlorine, total residual	Liquid/Solid	ASTM D2022, EPA 330.5, SM 4500CI-G, ASTM 808-	P, G	None required	Analyze within 15 minutes.

Parameter No./name	Matrix	Method	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
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Chromium, Hexavalent	Liquid/Solid	EPA 218.6, SM 3500Cr B, SW-846 7199*, ISO 17025-2007*, SM 3500 B-MOD*, SW-846 7196A*	G	Store at 4°C, adjust pH to 9-9.5 with buffer solution	Analyze within 24 hours of collection
Color	Liquid/Solid	EPA 110.2, SM 2120B, SM 2120Bm*	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
Cyanide, total or available (or CATC) and free	Liquid/Solid	EPA 335.2, SM 4500 CNC/E, SW-846 9014*, SW-846 9213,	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH>10 ^{5,6} , reducing agent if oxidizer present	14 days.
Fluoride	Liquid	EPA 340.2, SM 4500F--C	P	None required	28 days.
Hardness	Liquid	EPA 130.2, SM 2340B, SM 2340C	P, FP, G	HNO3 or H2SO4 to pH<2	6 months.
Hydrogen ion (pH)	Liquid/Solid	EPA 150.1*, SM 4500 H B, SW-846 9040C, SW-846 9041A, SW-846 9045D*	P, FP, G	None required	Analyze within 15 minutes.
Kjeldahl and organic N	Liquid/Solid	EPA 351.4*, SM 4500NH3D	P, FP, G	Cool, ≤6 °C ¹⁸ , H2SO4 to pH<2	28 days.
Metals⁷:					
Mercury (CVAA)	Liquid/Solid, Tissue	EPA 245.1, SW-846 7470A*, EPA 105*, EPA105m*, SW-846 7471B*#	P, FP, G	HNO3 to pH<2	28 days.
Mercury (CVAFS)	Liquid	EPA 1631E	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷	90 days. ¹⁷
Mercury_Low Level	Liquid	EPA 1631E, EPA 245.1	FP-lined cap, G can be used if Hg is only target analyte	5mL/L of pretested 12NHCl or 5mL/L BrCl	Analyze within 48 hours. Preserved samples up to 90 days of the date of collection.
Nitrate-nitrite	Liquid/Solid	EPA 353.2*, EPA 353.3, SM 4500NO3E	P, FP, G	Cool, ≤6 °C ¹⁸ , H2SO4 to pH<2	28 days.
Nitrite	Liquid	EPA 354.1, SM 4500NO2B	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
Oil and grease	Liquid/Solid	EPA 1664A*, EPA1664B, SW-846 9071B*	G	Cool to ≤6 °C ¹⁸ , HCl or H2SO4 to pH<2	28 days.
Organic Carbon	Liquid/Solid	EPA 415.1/SW-846 9060A	P, FP, G	Cool to ≤6 °C ¹⁸ , HCl, H2SO4, or H3PO4 to pH<2	28 days.
Orthophosphate	Liquid	EPA 365.2, SM 4500P-E	P, FP, G	Cool, ≤6 °C ^{18,24}	Filter within 15 minutes; Analyze within 48 hours.
Oxygen, Dissolved Probe	Liquid/Solid	EPA 360.1, SM 4500O-G, SM 4500O-Gm	G, Bottle and top	None required	Analyze within 15 minutes.
Phenols	Liquid/Solid	EPA 420.1*	G	Cool, ≤6 °C ¹⁸ , H2SO4 to pH<2	28 days.
Phosphorous, total	Liquid/Solid	EPA 365.2, SM 4500P-E	P, FP, G	Cool, ≤6 °C ¹⁸ , H2SO4 to pH<2	28 days.
Residue, Nonfilterable (TSS)	Liquid/Solid	EPA 160.2, SM 2540D, SM 2540Dm*	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
Silica	Liquid	EPA 370.1, SM 4500SiO2-D	P or Quartz	Cool, ≤6 °C ¹⁸	28 days.
Specific conductance	Liquid/Solid	EPA 120.1, SM 2510B, ASTM D4940*	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.

Parameter No./name	Matrix	Method	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Sulfate	Liquid/Solid	EPA 300.0/ASTM C1580-09/ASTM D516	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.
Sulfide	Liquid/Solid	EPA 376.2/SM 4500-S F/SW-846 9034	P, FP, G	Cool, ≤6 °C ¹⁸ , add zinc acetate plus sodium hydroxide to pH>9	7 days.
Sulfite	Liquid/Solid	EPA 377.1/SM4500 SO3-B	P, FP, G	None required	Analyze within 15 minutes.
Temperature	Liquid	SM 2550B	P, FP, G	None required	Analyze.
Turbidity	Liquid/Solid	EPA 180.1, SM 2130B, SM 2130Bm*	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
Organic Tests⁸:					
EDB_DBCP	Liquid	EPA 504.1	G	Cool to 4 °C	14 days.
GOC, GOC_flare	Air	Method 18	Cassette	Store under proper environmental conditions for compounds being analyzed	For air bags the hold time is 72 hours
Perchlorate	Liquid/Solid	EPA 6860/331.0	P, solids=4oz amber glass bottles	None required	28 days
PFAS	Liquid/Solid	EPA 537.1, EPA 537.1m*	P	Cool, ≤6 °C, 5.0 g/L Trizma,	Water extracted within 14 days and analyzed 28 days after extraction
RSK 175	Liquid	RSK 175	G, VOA vials	Acidify with HCl to pH<2 except for CO2, Store at 0.5-6°C	Analyze within 14 days of collection.
Sulfur compounds in Air/liquid	Air/Liquid	ASTM D5504	Tedlar bag	None required	Analyze within 24 hours of collection
SVOC	Liquid/Solid	ASTM- D7065-11, EPA 625.1, SW-846 8270C, SW-846 8270D*	Solid samples must be collected in wide mouth 4oz glass jar. Aqueous samples must be collected in one-liter amber bottle with a Teflon-lined cap.	Cool, ≤6 °C, Sodium thiosulfate, Na2S2O3, should be added to aqueous samples that contain residual chlorine.	Aqueous samples must be extracted with methylene chloride and concentrated for analysis within 7 days of sampling. All solid samples must be extracted within 14 days of sampling.
SVOC for Drinking Water	Liquid	EPA 525.2	G amber bottles fitted with Teflon-lined screw caps.	Store at 4 °C	Must be extracted within 14 days. Sample extracts may be stored for up to 30 days after extraction.
VOC	Liquid/Solid/Swab	EPA 524.2, EPA 624.1, SW-846 8260B*, SW-846 8260C*#	VOA Vial	Store at 4 °C, HCl	Hold Time is 14 days if samples are properly preserved.
Phenols ¹¹	Liquid/Solid	EPA 420.1*	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na2S2O3 ⁵	7 days until extraction, 40 days after extraction.

Parameter No./name	Matrix	Method	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
PCBs ¹¹	Liquid/Solid	EPA 608, EPA 608.3, SW-846 8082*, SW-846 8082A*	G, FP-lined cap	Cool, ≤6 °C ¹⁸	1 year until extraction, 1 year after extraction.
Polynuclear aromatic hydrocarbons ¹¹	Liquid/Solid	SW-846 8310*, EPA 610	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction.
Haloethers ¹¹	Liquid/Solid	EPA 615, SW-846 8151A*	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction.
Pesticides ¹¹	Liquid/Solid	EPA 608.3, SW-846 8081B*	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , pH 5–9 ¹⁵	7 days until extraction, 40 days after extraction.

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.

²Except 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, 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).

³When 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).

⁴Samples 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 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 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.

⁵ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine). Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (22nd Edition) addresses dechlorination procedures.

⁶Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365-09a. There may be interferences that are not mitigated by the analytical test methods or D7365-09a. 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 or the analytical test method must be documented along with supporting data.

⁷For 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.

⁸Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

⁹If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁰The 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.

¹¹When 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).

¹²If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

¹³Extracts may be stored up to 30 days at < 0 °C.

¹⁴For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵The 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% Na₂S₂O₃.

¹⁶Place 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, 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. 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.

¹⁷Samples 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.

¹⁸Aqueous 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 " \leq °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).

¹⁹An 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.

²⁰To 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.

²¹Holding 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.

²²Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

²³For 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.

²⁴The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (*i.e.*, that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (*i.e.*, within 15 minutes of collection).

[38 FR 28758, Oct. 16, 1973]

*Method also used for solids

#Method also used for swabs