

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

Parameter number/name	Container 1	Preservation ^{2 3}	Maximum holding time ⁴
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Table IA - Bacterial Tests

1-4. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ^{22 23}
5. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
6. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
7. <i>Salmonella</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²

Table IA - Aquatic Toxicity Tests

8-11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ¹⁶	36 hours.
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Table IB - Inorganic Tests

1. Acidity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸	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 ¹⁸	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸ , 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 ¹⁸	48 hours.
23-24. Cyanide, total or available (or CATC) and free	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH >10 ^{5 6} , reducing agent if oxidizer present	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.

Parameter number/name	Container 1	Preservation ^{2 3}	Maximum holding time ⁴
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 ¹⁸ , H ₂ SO ₄ to pH <2	28 days.

Table IB - Metals⁷

18. Chromium VI	P, FP, G	Cool, ≤ 6 °C ¹⁸ , pH = 9.3-9.7 ²⁰	28 days.
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH <2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷	90 days. ¹⁷
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 ¹⁹	6 months.
38. Nitrate	P, FP, G	Cool, ≤ 6 °C ¹⁸	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤ 6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days.
40. Nitrite	P, FP, G	Cool, ≤ 6 °C ¹⁸	48 hours.
41. Oil and grease	G	Cool to ≤ 6 °C ¹⁸ , HCl or H ₂ SO ₄ to pH <2	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤ 6 °C ¹⁸ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH <2	28 days.
44. Orthophosphate	P, FP, G	Cool, to ≤ 6 °C ^{18 24}	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 ¹⁸ , H ₂ SO ₄ to pH <2	28 days.
49. Phosphorus (elemental)	G	Cool, ≤ 6 °C ¹⁸	48 hours.
50. Phosphorus, total	P, FP, G	Cool, ≤ 6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days.
53. Residue, total	P, FP, G	Cool, ≤ 6 °C ¹⁸	7 days.
54. Residue, Filterable (TDS)	P, FP, G	Cool, ≤ 6 °C ¹⁸	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤ 6 °C ¹⁸	7 days.

Parameter number/name	Container 1	Preservation ^{2 3}	Maximum holding time ⁴
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C ¹⁸	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C ¹⁸ , 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 ¹⁸	48 hours.
69. Temperature	P, FP, G	None required	Analyze within 15 minutes.
73. Turbidity	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.

Table IC - Organic Tests⁸

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 ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹	14 days. ⁹
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹	14 days. ⁹
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ , pH to 4-5 ¹⁰	14 days. ¹⁰
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction.
7, 38. Benzidines ^{11 12}	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction. ¹³
14, 17, 48, 50-52. Phthalate esters ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸	7 days until extraction, 40 days after extraction.

Parameter number/name	Container 1	Preservation 2 3	Maximum holding time 4
82-84. Nitrosamines ^{11 14}	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.
88-94. PCBs ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone ¹¹	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.
1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹	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.
15, 16, 21, 31, 87. Haloethers ¹¹	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.
29, 35-37, 63-65, 73, 107. Chlorinated hydrocarbons ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸	7 days until extraction, 40 days after extraction.
60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs ¹¹	G	See footnote 11	See footnote 11.
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH <9	1 year.
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C ¹⁸	7 days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C ¹⁸	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 ₂ SO ₄ to pH <2	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX)	G	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , HNO ₃ to pH <2	Hold <i>at least</i> 3 days, but not more than 6 months.

Parameter number/name	Container 1	Preservation ^{2 3}	Maximum holding time ⁴
120. Chlorinated Phenolics	G, FP-lined cap	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , H ₂ SO ₄ to pH <2	30 days until acetylation, 30 days after acetylation.

Table ID - Pesticides Tests

1-70. Pesticides ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , pH 5-9 ¹⁵	7 days until extraction, 40 days after extraction.
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Table IE - Radiological Tests

1-5. Alpha, beta, and radium	P, FP, G	HNO ₃ to pH <2	6 months.
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Table IH - Bacterial Tests

1, 2. Coliform, total, fecal	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
3. <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
4. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²
5. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours. ²²

Table IH - Protozoan Tests

6. <i>Cryptosporidium</i>	LDPE; field filtration	1-10 °C	96 hours. ²¹
7. <i>Giardia</i>	LDPE; field filtration	1-10 °C	96 hours. ²¹

¹ "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.

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 sample (e.g., using a 24-hour composite sample; 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).

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 requirement 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. 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.

⁵ 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.

⁶ 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.

⁷ 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.

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

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

¹⁰ 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.

¹¹ 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).

¹² 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.

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

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

¹⁵ 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% Na₂S₂O₃.

¹⁶ 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.

¹⁷ 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.

¹⁸ 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).

¹⁹ 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.

²⁰ 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.

²¹ 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.

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

²³ 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.

²⁴ The 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]

EDITORIAL NOTE

Editorial Note: For FEDERAL REGISTER citations affecting § 136.3, see the List of CFR Sections Affected, which appears in the Finding Aids section of the printed volume and at www.govinfo.gov.
