

TECHNICAL APPENDIX C

**REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, HOLDING TIMES AND
40 CODE OF FEDERAL REGULATIONS (CFR) PART 136**

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES			
Parameter	Container(1)	Preservative (2), (3)	Maximum Holding Time (4)
Bacterial Tests			
Coliform, fecal and total	P, G	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	6 hours
Fecal streptococci	P, G	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	6 hours
Inorganic Tests			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia	P, G	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Biochemical oxygen demand	P, G	Cool, 4°C	48 hours
Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical oxygen demand	P, G	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Chloride	P, G	None required	28 days
Chlorine, total residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C NaOH to pH>12 0.6g ascorbic acid (5)	14 days (6)
Fluoride	P	None required	28 days
Hardness	P, G	HN0 ₃ to pH<2 H ₂ SO ₄ to pH<2	6 months
Hydrogen ion (pH)	P, G	None required	Analyze immediately
Kjeldahl and organic Nitrogen	P, G	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Metals (7)			
Chromium VI	P, G	Cool, 4°C	28 hours
Mercury	P, G	HN0 ₃ to pH<2	28 hours
Metals, except above	P, G	HN0 ₃ to pH<2	6 months
Nitrate	P, G	Cool, 4°C	48 hours

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Parameter	Container(1)	Preservative (2), (3)	Maximum Holding Time (4)
Nitrate-nitrite	P, G	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Nitrite	P, G	Cool, 4°C	48 hours
O&G	G	Cool, 4°C H ₂ SO ₄ or HCl to pH<2	28 days
Organic carbon	P, G	Cool, 4°C HCl or H ₂ SO ₄ to pH<2	28 days
Orthophosphate	P, G	Filter immediately Cool, 4°C	48 hours
Oxygen, Dissolved Probe	G bottle and top	None required	Analyze immediately
Dissolved oxygen, Winkler method	G bottle and top	Fix on site and store in dark	8 hours
Phenols	G only	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total	P, G	Cool, 4°C H ₂ SO ₄ to pH<2	28 days
Residue, total	P, G	Cool, 4°C	7 days
Residue, filterable	P, G	Cool, 4°C	7 days
Residue, nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, settleable	P, G	Cool, 4°C	48 hours
Residue, volatile	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days
Sulfide	P, G	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
Sulfite	P, G	None required	Analyze immediately
Surfactants	P, G	Cool, 4°C	48 hours
Temperature	P, G	None required	Analyze
Turbidity	P, G	Cool, 4°C	48 hours

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Parameter	Container(1)	Preservative (2), (3)	Maximum Holding Time (4)
Organic Tests (8)			
Purgeable halocarbons	G, Teflon-lined septum	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	14 days
Purgeable aromatics	G, Teflon-lined septum	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	14 days
Acrolein and acrylonitrile	G, Teflon-lined septum	HCl to pH<2 (9) Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	14 days
Phenols (11)	G, Teflon-lined cap	Adjust pH to 4-5 (10) Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
Benzidines (11)	G, Teflon-lined cap	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction (13)
Phthalate esters (11)	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines (11), (14)	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
PCBs (11) acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitroaromatics and isophorone (11)	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons (11)	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
Haloethers (11)	G, Teflon-lined cap	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons (11)	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD (11)	G, Teflon-lined cap	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
Pesticides Tests			
Pesticides (11)	G, Teflon-lined cap	Cool, 4°C pH 5-9 (15)	7 days until extraction, 40 days after extraction
Radiological Tests			
Alpha, beta, and radium	P, G	HNO ₃ to pH<2	6 months

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- (1) Polyethylene (P) or Glass (G).
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States Mails, 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 weights 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 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 the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). 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 knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- (7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- (9) Sample receiving no pH adjustment 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.

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- (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. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°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 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re 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 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- (14) For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- (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% $\text{Na}_2\text{S}_2\text{O}_3$.

Source: 40 CFR 136.3 Table II