

DEP-SOP-001/01
FS 1000 General Sampling Procedures

Table FS1000-4

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

Parameter No./Name (refers to parameter number on Tables IA, B, C, D, E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours ^{22, 23}
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
Table IA— Aquatic Toxicity Tests:			
9–12. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ¹⁶	36 hours
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 reducing agent if oxidizer is present. ^{5, 6}	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO ₃ 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 ¹⁸ , H ₂ SO ₄ to pH<2	28 days
Table IB—Metals⁷:			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C ¹⁸ , pH = 9.3–9.7 ²⁰	28 days / 24 hrs without preservative

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Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH<2	28 days
35. Mercury (CVAFS)	FP, G; and FP-lined cap	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
Table IB—Inorganic Tests (continued):			
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, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C ¹⁸ H ₂ SO ₄ , or H ₃ PO ₄ to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤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. Phosphorous (elemental)	G	Cool, ≤6 °C ¹⁸	48 hours
50. Phosphorous, 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	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
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

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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
73. Turbidity	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours

Table IC—Organic Tests⁸			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C 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	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	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
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, 107. Chlorinated hydrocarbons ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸	7 days until extraction, 40 days after extraction

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Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs 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 ¹⁸ , 0.008% Na ₂ S ₂ O ₃ HNO ₃ to pH<2	Hold at least 3 days, but not more than 6 months
120. Chlorinated Phenolics		Cool, <6 °C ¹⁸ , 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
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO ₃ to pH<2	6 months
Table IH—Bacterial Tests:			
1. <i>E. coli</i>	PA, G, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ Na ₂ S ₂ O ₃ ⁵ Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ²² hours ⁶
2. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ Na ₂ S ₂ O ₃ ⁵	8 hours ²²
Table IH—Protozoan Tests:			
8. <i>Cryptosporidium</i>	LDPE; field filtration	0–10 °C	96 hours. ²¹
9. <i>Giardia</i>	LDPE; field filtration	0–10 °C	96 hours ²¹

¹⁶ ¹⁷ ¹⁸ ¹⁹ ²⁰ ²¹ ²² ²³ ²⁴

*Reference: This table is adapted from Table II, 40 CFR, Ch.I, Part 136.3, *Identification of Test Procedures*, 7-1-13 Edition, including all footnotes listed below.

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

² Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤ 6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤ 6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

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

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. 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 Sec. 136.3(e). For a grab sample, the holding

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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 22.21(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 headspace.

⁵ ASTM 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 (20th and 21st editions) addresses dechlorination procedures.

⁶ Sampling, 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.

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.

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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 19, 20 (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 $<0^{\circ}$ C.

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% $\text{Na}_2\text{S}_2\text{O}_3$.

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

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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 " ≤ 6 °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.

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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).

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Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times

- For Analytes not Found in 40 CFR 136*

Analyte	Methods	Reference ¹	Container ²	Preservation ³	Maximum Holding Time ⁴
Bromine	DPD Colorimetric ⁵	SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 ⁶	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G ⁷	Dark 4°C Filtered, dark, 20°C	48 hours chilled until filtration ⁸ , and analyze immediately or 48 hours chilled until filtration ⁹ , and 28 days (frozen) after filtration
Corrosivity	Calculated (CaCO ₃ Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C ⁹	7 days ⁹
Cyanotoxin	ELISA and LC/MSMS		FP, G	Cool 6°C ¹⁶	7 days until extraction, 40 days after extraction
FL-PRO	Gas Chromatography	DEP (11/1/95) ¹⁸	G, PTFE lined cap only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	7 days until extraction, 40 days after extraction
Odor	Human Panel	SM 2150	G only	Cool 4°C	6 hours
Salinity	Electrometric ¹⁰ Hydrometric ¹⁰	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days ¹⁰
Taste	Human Panel	SM 2160 B, C, D ASTM E679-91	G only	Cool 4°C	24 hours
Total Dissolved Gases	Direct-sensing Membrane-diffusion	SM 2810	_____	_____	Analyze in-situ
Total Petroleum Hydrocarbons	Gravimetry	EPA 1664 ¹⁷	G only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	28 days
Transparency	Irradiometric ¹¹	62-302.200(6), FAC	_____	_____	Analyze in-situ
Un-ionized Ammonia	Calculated ¹²	DEP-SOP ¹³	P, G	Cool 4°C Na ₂ S ₂ O ₃ ¹²	8 hours unpreserved 28 days preserved ¹²
Organic Pesticides ¹⁴	GC and HPLC	EPA (600-series) ¹⁴	¹⁵	¹⁵	^{15 16}

*40 CFR, Ch. I, Part 136.3, *Identification of Test Procedures*, 7-1-13 Edition. Reference provided for informational purposes only.

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

¹ SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater"; see Standard Methods Online (<http://www.standardmethods.org/store/>). Reference methods are listed for informational purposes only.

ASTM XXXX-YY = procedure from "Annual Book of ASTM Standards", Water and Environmental Technology, Volumes 11.01 and 11.02 (Water I and II). See American Society for Testing and Materials (ASTM International), <http://www.astm.org/Standard/index.shtml>. Reference methods are listed for informational purposes only.

² P = plastic, G = glass.

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

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

⁵ The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).

⁶ "The Determination of Inorganic Anions in Water by Ion Chromatography", EPA Method 300.0, Revision 2.1., Revised August 1993, by John D. Pfaff, U. S. EPA Cincinnati, Ohio 45268. Reference methods are listed for informational purposes only.

⁷ Collect samples in opaque bottles and process under reduced light. A secondary device, such as a Van Dorn/Niskin or bucket, may be used to collect the sample and then expeditiously transfer into an opaque bottle.

⁸ Samples must be filtered within 48 hours of collection. Add magnesium carbonate to the filter while the last of the sample passes through the filter.

⁹ Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.

¹⁰ The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

¹¹ Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DEP Chapter 62-302, FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.

¹² The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on-site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H₂SO₄ to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

¹³ Calculation of Un-ionized Ammonia in Fresh Water, Chemistry Laboratory Methods Manual, Florida Department of Environmental Protection, Revision 2, 2/12/2001. The document is available from the DEP Standards & Assessment Section. Reference method listed for informational purposes only.

¹⁴ Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) that are not included in Table ID of 40 CFR Part 136.3 (7-1-13 Edition). Reference methods and CFR citation listed for informational purposes only.

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

¹⁵ Container, preservation and holding time as specified in each individual method must be followed.

¹⁶ Sample preservation procedures, container material and maximum allowable holding times for analytes not specified in DEP-SOP-001/01 (March 1, 2014) shall follow the preservation, container and holding time requirements specified in the selected analytical method. If no method-specified requirements exist, the best available scientific knowledge shall be used as guidance for determining the appropriate procedures for use, per 62-160.400(2), F.A.C.

¹⁷ Method 1664, n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. Revision A, February 1999. EPA-821-R-98-002, and, Revision B, February 2010. EPA-821-R-10-001. Reference methods listed for informational purposes only.

¹⁸ FL-PRO - Method for Determination of Petroleum Range Organics, Revision 1, November 1, 1995, Florida Department of Environmental Protection

DEP-SOP-001/01
FS 1000 General Sampling Procedures

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

Analyte	Methods	References*	Container	Preservation	Maximum Holding Times
Volatile Organics	Purge-and-Trap GC and GC-MS	8015, 8260, 8021, 5035	See Table 1000-7		
Semivolatile Organics	GC, HPLC, and GC-MS	8041, 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 8141, 8151, 8270, 8275, 8280, 8290, 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332, 8410, 8430, 8440, FL-PRO, MADEP, TPHWG	Glass, 8 oz widemouth with Teflon® -Lined lid	Cool ≤6°C ¹	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool ≤6°C ¹ in dark	30 days until extraction, 45 days after extraction
Total Metals-except mercury and chromium VI methods	Flame AA, Furnace AA, Hydride and ICP	All 7000-series (except 7195, 7196, 7197, 7198, 7470 and 7471), 6010 (ICP) and 6020 (ICP)	Glass or plastic 8 oz widemouth (200 grams sample)	None	6 months
Chromium VI	Colorimetric, Chelation with Flame AA (200 gram sample)	7196 and 7197 (prep 3060)	Glass or plastic, 8 oz widemouth (200 gram sample)	Cool 4°± 2°C ¹	1 month until extraction, 4 days after extraction ²
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4°± 2°C ¹	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool ≤6°C ¹	24 hours
Aggregate Properties			Glass or plastic	Cool ≤6°C ¹	14 days
Inorganic nonmetallics all except:			Glass or plastic	Cool ≤6°C ¹	28 days
Cyanide			Glass or plastic		14 days
Sulfite, Nitrate, Nitrite & o-phosphate			Glass or plastic		48 hours
Elemental Phosphorus			Glass		48 hours

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

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

¹ Keep soils, sediments and sludges cool at $\leq 6^{\circ}\text{C}$ from collection time until analysis. No preservation is required for concentrated waste samples.

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

* Reference method numbers are listed for informational purposes only and are found in SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (<http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm>), except for the additional informational method sources listed below:

FL-PRO - Method for Determination of Petroleum Range Organics, Revision 1, November 1, 1995, Florida Department of Environmental Protection

MADEP – Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Revision 1.1, May 2004, Massachusetts Department of Environmental Protection

MPN – Microbiological test methods utilizing Most Probable Number procedures

TPHWG - TPH Working Group Series

DEP-SOP-001/01
FS 1000 General Sampling Procedures

Table FS 1000-7

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

Conc. Level	Sampling Device	Collection Procedure*	Sample Container		Preservation	Sample Preparation*	Max HT [Ⓣ]	Determinative Procedure [^]
			Type	Vial Preparation*				
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1	NaHSO ₄ / 4°C (see 5035 – 6.4.3)	5035 - Section 7.2.3	14 D	Any recognized VOC Method (see 5035 – 7.2)
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1 [Ⓣ]	4°C	5035 - Section 7.2.3	48 H	Any recognized VOC Method (see 5035 – 7.2)
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1 [Ⓣ]	4°C / -10°C ^{Ⓣ,Ⓢ}	5035 - Section 7.2.3	48 H / 14 D [Ⓢ]	Any recognized VOC Method (see 5035 – 7.2)
≤200 ug/kg	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{Ⓣ,Ⓢ,Ⓣ}	4°C	5035 - Section 7.2.3	48 H	Any recognized VOC Method (see 5035 – 7.2)
≤200 ug/kg	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{Ⓢ,Ⓣ}	NaHSO ₄ / 4°C (see 5035 – 6.4.3)	5035 - Section 7.2.3 [Ⓢ]	48 H / 14 D [Ⓢ]	Any recognized VOC Method (see 5035 – 7.2)
≤200 ug/kg	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1 ^{Ⓣ,Ⓢ,Ⓣ}	4°C / -10°C ^{Ⓢ,Ⓣ}	5035 - Section 7.2 [Ⓢ]	48 H / 14 D [Ⓢ]	Any recognized VOC Method (see 5035 – 7.2)
>200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3 [Ⓢ]	EnCore or equivalent	5035 - 6.1.3 ^{Ⓢ,Ⓣ}	4°C	5035 - Sections 7.3.2 & 7.3.3 [Ⓢ]	48 H / 14 D [Ⓢ]	Any recognized VOC Method (see 5035 – 7.3)
>200 ug/kg [Ⓢ]	Coring Device	5035 - Section 6.2.2.3 [Ⓢ]	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.3 [Ⓢ]	Methanol/PEG + 4°C	5035 - Section 7.3.4	14 D	Any recognized VOC Method (see 5035 – 7.3)
>200 ug/kg [Ⓢ]	Conventional Devices	DEP SOP - Section 5.	Glass w/ PTFE-silicone Septum	5035 - 6.1.2	4°C	5035 - Sections 7.3.1 - 7.3.3	14 D	Any recognized VOC Method (see 5035 – 7.3)
Oily Waste	Conventional Devices	5035 - Section 6.2.4.2	Glass w/ PTFE-silicone Septum	5035 - 6.1.4	4°C	5035 - Sections 7.4.1 - 7.4.2	14 D	Any recognized VOC Method (see 5035 – 7.4)
Oily Waste	Conventional Devices	5035 - Section 6.2.4.1	Glass w/ PTFE-silicone Septum	5035 - 6.1.4	Methanol/PEG + 4°C	5035 - Sections 7.4.3	14 D	Any recognized VOC Method (see 5035 – 7.4)
Dry Wt.	Conventional Devices	5035 - Sections 6.2.1.6, 6.2.2.7,	Glass with Teflon	5035 – 4.4.1	4°C	5035 - Section 7.5	Not applicable	5035 - Section 7.5

Table FS 1000-7

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

		6.2.3	liner					
Soil Screen	Conventional Devices	5035 - Sections 6.2.1.6, 6.2.3	Glass w/ PTFE-silicone Septum	5035 – 4.4.1	4°C	5035 - Section 7.1	14 D	Any recognized VOC Method (see 5035 – 7.1)

¹Maximum time allowable from time/date of collection to sample analysis.

²Eliminate 6.1.1.2; use only organic-free water.

³Contents of sampling device must be transported to the laboratory at 4°C and stored at -10°C.

⁴In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle to maximize surface area.

⁵Maximum allowable time at 4°C is 48 hours; maximum allowable time to sample analysis is 14 days (from time of sample collection).

⁶Conducted in the laboratory.

⁷Entire contents of sampling device are extruded into the sample analysis vial containing the appropriate solvent.

⁸Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 ug/kg.

⁹Methanolic preservation in the field is not recommended, but may be used if approved by DEP for a project.

*See method 5035 in SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (<http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm>) and DEP SOP FS 3000, Soil, in DEP-SOP-001/01, 9/19/12.

^See 62-160.320, F.A.C., Approved Laboratory Methods

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

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

***Dechlorinate bulk samples when applicable**

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

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

NA – Not Applicable