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40 CFR Ch. I (7-1-03 Edition)

Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or

deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
Table IA—Bacteria Tests:			
1-4 Coliform, fecal and total	P,G	Cool, 4C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours.
5 Fecal streptococci	P,G	Cool, 4C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours.
Table IA—Aquatic Toxicity Tests:			
6-10 Toxicity, acute and chronic	P,G	Cool, 4 °C <sup>16</sup>	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	.....do	Do.
4. Ammonia	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz.	HNO <sub>3</sub> TO pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	.....do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23-24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid <sup>5</sup> .	14 days. <sup>6</sup>
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO <sub>3</sub> to pH<2, H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
Metals: <sup>7</sup>			
18. Chromium VI <sup>7</sup>	P, G	Cool, 4 °C	24 hours.
35. Mercury <sup>17</sup>	P, G	HNO <sub>3</sub> to pH<2	28 days.
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 <sup>7</sup> .	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H <sub>2</sub> SO4 or H <sub>3</sub> PO4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winkler	.....do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	.....do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	.....do	7 days.
56. Residue, Settleable	P, G	.....do	48 hours.
57. Residue, volatile	P, G	.....do	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	.....do	Do.
65. Sulfate	P, G	.....do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P, G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests <sup>8</sup>			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> .	14 days.

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TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
6, 57, 106. Purgeable aromatic hydrocarbons .....	.....do .....	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , <sup>5</sup> HCl to pH2 <sup>9</sup> .	Do.
3, 4. Acrolein and acrylonitrile .....	.....do .....	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , <sup>5</sup> adjust pH to 4–5 <sup>10</sup> .	Do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols <sup>11</sup> .	G, Teflon-lined cap..	Cool, 4 °C, 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.
7, 38. Benzidines <sup>11</sup> .....	.....do .....	.....do .....	7 days until extraction. <sup>13</sup>
14, 17, 48, 50–52. Phthalate esters <sup>11</sup> .....	.....do .....	Cool, 4 °C .....	7 days until extraction; 40 days after extraction.
82–84. Nitrosamines <sup>11 14</sup> .....	.....do .....	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , <sup>5</sup> store in dark.	Do.
88–94. PCBs <sup>11</sup> .....	.....do .....	Cool, 4 °C .....	Do.
54, 55, 75, 79. Nitroaromatics and isophorone <sup>11</sup> ..	.....do .....	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , <sup>5</sup> store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons <sup>11</sup> .	.....do .....	.....do .....	Do.
15, 16, 21, 31, 87. Haloethers <sup>11</sup> .....	.....do .....	Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	Do.
29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons <sup>11</sup> .	.....do .....	Cool, 4 °C .....	Do.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs <sup>11</sup> .			
aqueous: field and lab preservation. ....	G .....	Cool, 0–4 °C, pH<9, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> .	1 year.
Solids, mixed phase, and tissue: field preservation..	.....do .....	Cool, <4 °C .....	7 days.
Solids, mixed phase, and tissue: lab preservation	.....do .....	Freeze, <–10 °C .....	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides <sup>11</sup> .....	.....do .....	Cool, 4°C, pH 5–9 <sup>15</sup> .....	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium .....	P, G .....	HNO <sub>3</sub> to pH<2 .....	6 months.

Table II Notes  
<sup>1</sup> Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).  
<sup>2</sup> 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.  
<sup>3</sup> 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 (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 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).  
<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 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). 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. The term "analyze immediately" usually means within 15 minutes or less of sample collection.  
<sup>5</sup> Should only be used in the presence of residual chlorine.  
<sup>6</sup> 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.  
<sup>7</sup> Samples should be filtered immediately on-site before adding preservative for dissolved metals.  
<sup>8</sup> Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.  
<sup>9</sup> Sample receiving no pH adjustment 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. 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).  
<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 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.  
<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>Sufficient ice should be placed 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 4°C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not 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.

<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. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987; 55 FR 24534, June 15, 1990; 55 FR 33440, Aug. 15, 1990; 56 FR 50759, Oct. 8, 1991; 57 FR 41833, Sept. 11, 1992; 58 FR 4505, Jan. 31, 1994; 60 FR 17160, Apr. 4, 1995; 60 FR 39588, 39590, Aug. 2, 1995; 60 FR 44672, Aug. 28, 1995; 60 FR 53542, 53543, Oct. 16, 1995; 62 FR 48403, 48404, Sept. 15, 1997; 63 FR 50423, Sept. 21, 1998; 64 FR 4978, Feb. 2, 1999; 64 FR 10392, Mar. 4, 1999; 64 FR 26327, May 14, 1999; 64 FR 30433, 30434, June 8, 1999; 64 FR 73423, Dec. 30, 1999; 66 FR 32776, June 18, 2001; 67 FR 65226, Oct. 23, 2002; 67 FR 65886, Oct. 29, 2002; 67 FR 69971, Nov. 19, 2002]

#### § 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published

studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Analytical Methods Staff, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW., Washington, DC 20460. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976; 62 FR 30763, June 5, 1997]