

**PART 136**  
**GUIDELINES ESTABLISHING TEST PROCEDURES**  
**FOR THE ANALYSIS OF POLLUTANTS**

[Editor’s Note: This document highlights changes to Part 136 as a result of the 2012 Method Update Rule. Changes are shown in red font, except deleted language is not shown. As with other LABFACTS articles, some non-relevant language is not shown and minor formatting changes have been made to improve readability. Because of the complexity of Table 1B (Inorganic Methods), that table is provided as a separate document. Catalyst has attempted to capture all changes, but if errors are noted, please let us know.] (Revision 1.1; 5/7/12)

**136.1 Applicability.**

The procedures shall be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

- An application for a permit under section 402 of the Clean Water Act of 1977 (CWA), and/or reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under Parts 122 to 125, and,
- Reports required to be submitted by dischargers under the NPDES established by Parts 124 and 125, and,
- Certifications issued by States pursuant to section 401 of the CWA.

The procedure prescribed herein and in part 503 shall be used to perform the measurements required for an application for a sewage sludge permit and for recordkeeping and reporting requirements under part 503.

**136.2 Definitions.**

National Pollutant Discharge Elimination System (NPDES) means the national system for the issuance of permits under section 402 of the Act.

Detection limit means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure at appendix B.

**136.3 Identification of test procedures.**

a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, IG and 1H. **The methods listed in Tables IA, IB, IC, ID, IE, IF, IG, and IH are incorporated by reference, see paragraph (b), with the exception of EPA Methods 200.7, 601–613, 624, 625, 1613, 1624, and 1625. The full texts of Methods 601–613, 624, 625, 1613, 1624, and 1625 are printed in appendix, and the full text of Method 200.7 is printed in appendix C. The full text for determining the method detection limit when using the test procedures is given in appendix B.** In the event of a conflict between the reporting requirements of Parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of Parts 122 and 125 are controlling and will determine a permittee’s reporting requirements. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG and 1H. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, IE, IF, IG and 1H or by any alternate test procedure which has been approved by the Administrator. Under certain circumstances other additional or alternate test procedures may be used.

**Table IA.--List of Approved Biological Methods for Wastewater and Sewage Sludge**

Parameter and units	Method <sup>1</sup>	EPA	Standard Methods	ASTM <sup>10</sup> AOAC <sup>11</sup> USGS <sup>5</sup>	Other
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**Bacteria:**

1. Coliform (fecal), number per 100 mL, or number per gram dry weight	Most Probable Number (MPN), 5 tube 3 dilution, or	p.132 <sup>3</sup> 1680 <sup>11,15</sup> 1681 <sup>11,20</sup>	9221C or E-06		
	Membrane filter (MF) <sup>2</sup> , single step	p.124 <sup>3</sup>	9222D-97	B-0050-85 <sup>5</sup>	
2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p.132 <sup>3</sup>	9221C or E-06		
3. Coliform (total), number per 100 mL	MF <sup>2</sup> , single step <sup>5</sup> MPN, 5 tube, 3 dilution, or	p. 124 <sup>3</sup> p. 114 <sup>3</sup>	9222D-97 9221B-06	B-0025-8 <sup>5</sup>	
4. Coliform (total), in presence of chlorine, number per 100 mL	MF <sup>2</sup> single step or two step MPN, 5 tube, 3 dilution, or	p. 108 <sup>3</sup> p. 114 <sup>3</sup>	9222B-97 9221B-06		
5. <i>E. coli</i> , number per 100 mL <sup>21</sup>	MF <sup>2</sup> with enrichment <sup>5</sup> MPN <sup>6,8,16</sup> multiple tube, or	p. 111 <sup>3</sup>	9222(B+B.5c)-97 9221B.1-06/9221F-06 <sup>12,14</sup> 9223B-04 <sup>13</sup>	991.15 <sup>11</sup>	Colilert <sup>13,18</sup> Colilert-18 <sup>13,17,18</sup> mColiBue 24 <sup>19</sup>
6. Fecal streptococci, number per 100 mL	MPN <sup>7,9,15</sup> multiple tube/multiple well, or MF <sup>2,6,7,8</sup> single step MPN, 5 tube, 3 dilution, or	1603 <sup>22</sup> p.139 <sup>3</sup>	9230B-07		
7. Enterococci, number per 100 mL <sup>22</sup>	MF <sup>2</sup> , or Plate count MPN <sup>6,8</sup> multiple tube/multiple well, or MF <sup>2,6,7,8</sup> single step Plate count	p.136 <sup>3</sup> p.143 <sup>3</sup>	9230C-07	B-0055-85 <sup>4</sup>	
8. Salmonella, number per gram dry weight <sup>11</sup>	MPN <sup>6,8</sup> multiple tube/multiple well, or MPN multiple tube	1600 <sup>25</sup> p.143 <sup>3</sup> 1682 <sup>23</sup>		D6503-99	Enterolert <sup>13,23</sup>
<b>Aquatic Toxicity:</b>					
9. Toxicity, acute, fresh water organisms, LC50, percent effluent	Ceriodaphnia dubia acute	2002.0 <sup>26</sup>			
	Daphnia pulex and Daphnia magna acute	2021.0 <sup>26</sup>			
	Fathead minnow, Pimephales promelas, and Bannerfin shiner, Cyprinella leedsii, acute.	2001.0 <sup>26</sup>			
	Rainbow trout, Oncorhynchus mykiss, and brook trout, Salvelinus fontinalis, acute.	2019.0 <sup>26</sup>			
10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC50, percent effluent	Mysid, Mysidopsis, bahia, acute	2007.0 <sup>26</sup>			
	Sheepshead minnow, Cyprinodon variegatus, acute.	2004.0 <sup>26</sup>			
	Silverside, Menidia beryllina, Menidia menidia, and Menidia peninsulae, acute	2006.0 <sup>26</sup>			
13. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent	Fathead minnow, Pimephales promelas, larval survival and growth	1000.0 <sup>27</sup>			
	Fathead minnow, Pimephales	1001.0 <sup>27</sup>			

	promelas, embryo-larval survival and teratogenicity	
	Daphnia, Ceriodaphnia dubia, survival and reproduction.	1002.0 <sup>27</sup>
	Green alga, Selenastrum capricornutum, growth	1003.0 <sup>27</sup>
14. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC25, percent	Sheepshead minnow, Cyprinodon variegatus, larval survival and growth	1004.0 <sup>28</sup>
	Sheepshead minnow, Cyprinodon variegatus embryo-larval survival and teratogenicity	1005.0 <sup>28</sup>
	Inland silverside, Menidia beryllina, larval survival and growth	1006.0 <sup>28</sup>
	Mysid, Mysidopsis bahia, survival, growth and fecundity.	1007.0 <sup>28</sup>
	Sea urchin, Arbacia punctulata, fertilization	1008.0 <sup>27</sup>

Notes to Table IA:

1 The method must be specified when results are reported.

2 A 0.45 um membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

3 USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8-78/017.

4 USGS. 1989. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of Interior, Reston, Virginia.

5 Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

6 Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

7 When the MF method has not been used previously to test ambient waters with high turbidity, large number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

8 To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

9 ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards--Water and Environmental Technology. Section 11.02. ASTM International.

10 AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17.

11 Recommended for enumeration of target organism in sewage sludge.

12 The multiple-tube fermentation test is used in 9221B.1-2006. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

13 These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme [beta]glucuronidase produced by E. coli.

14 After prior enrichment in a presumptive medium for total coliform using 9221B.1-2006, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F-2006. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

15 Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium. US Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-10-003. April 2010. USEPA.

16 Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert may be enumerated with the multiple-well procedures, Quanti-Tray or Quanti-Tray 2000, and the MPN calculated from the table provided by the manufacturer.

17 Colilert-18 is an optimized formulation of the Colilert for the determination of total coliforms and E. coli that provides results within 18 h of incubation at 35 C rather than the 24 h required for the Colilert test and is recommended for marine water samples.

18 Descriptions of the Colilert, Colilert-18, Quanti-Tray, and Quanti-Tray/2000 may be obtained from IDEXX Laboratories, Inc.

19 A description of the mColiBlue24 test, Total Coliforms and E. coli, is available from Hach Company.

20. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-013. July 2006' USEPA.

21 Recommended for enumeration of target organism in wastewater effluent.

22 Method 1603: Escherichia coli (E. coli) In Water By Membrane Filtration Using Modified membrane-Thermotolerant Escherichia coli Agar modified mTEC). EPA-821-R-09-007. USEPA. July 2009.

23 Method 1682: Salmonella in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-014. July 2006. USEPA.

24 A description of the Enterolert test may be obtained from IDEXX Laboratories, Inc.

25 Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-[beta]-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-09-016. December 2009. USEPA.

26 USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/012.

27 USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition, U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/013.

28 USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/014.

[Editor's Note: Because Table 1 B had the most changes, and because it is just a complex table, this table is contained as a separate document in landscape format.]

**Table IC.--List of Approved Test Procedures for Non-Pesticide Organic Compounds**

<b>Parameter<sup>1</sup></b>	<b>EPA Method Number<sup>2,7</sup></b>	<b>Standard Methods</b>	<b>ASTM</b>	<b>Other</b>
1. Acenaphthene	610, 625, 1625B		D4657-92 (98)	
2. Acenaphthylene	610, 625, 1625B	6410 B-00	D4657-92 (98)	
3. Acrolein	603, 624 <sup>4</sup> , 1624B			
4. Acrylonitrile	603, 624 <sup>4</sup> , 1624B			

5. Anthracene	610, 625, 1625B	6410 B-00	D4657-92 (98)	
6. Benzene	602, 624, 1624B	6200 B and C-97		
7. Benzidine	605, 625 <sup>5</sup> , 1625			Note 3, p.1
8. Benzo(a)anthracene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
9. Benzo(a)pyrene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
10. Benzo(b)fluoranthene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
11. Benzo(g,h,i)perylene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
12. Benzo(k)fluoranthene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
13. Benzyl chloride				Notes 3 p 130, 6 p. S102
14. Benzyl butyl phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
15. Bis(2-chloroethoxy) methane	611, 625, 1625B	6410 B-00		Note 9, p. 27
16. Bis(2-chloroethyl) ether	611, 625, 1625B	6410 B-00		Note 9, p. 27
17. Bis (2-ethylhexyl) phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
18. Bromodichloromethane	601, 624, 1624B	6200 B and C-97		
19. Bromoform	601, 624, 1624B	6200 B and C-97		
20. Bromomethane	601, 624, 1624B	6200 B and C-97		
21. 4-Bromophenylphenyl ether	611, 625, 1625B	6410 B-00		
22. Carbon tetrachloride	601, 624, 1624B			Note 3, p. 130
23. 4-Chloro-3-methylphenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
24. Chlorobenzene	601, 602, 624, 1624B	6200 B and C-97		Note 3 p. 130
25. Chloroethane	601, 624, 1624B	6200 B and C-97		
26. 2-Chloroethylvinyl ether	601, 624, 1624B	6200 B and C-97		
27. Chloroform	601, 624, 1624B	6200 B and C-97		Note 3, p. 130
28. Chloromethane	601, 624, 1624B	6200 B and C-97		
29. 2-Chloronaphthalene	612, 625, 1625B	6410 B-00		Note 9, p. 27
30. 2-Chlorophenol	604, 625, 1625B	6410 B-00		Note 9, p. 27
31. 4-Chlorophenylphenyl ether	611. 625, 1625B	6410 B-00		Note 9, p. 27
32. Chrysene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
33. Dibenzo(a,h)anthracene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
34. Dibromochloromethane	601, 624, 1624B	6200 B and C-97		
35. 1,2-Dichlorobenzene	601, 602 624, 1625B	6200 C-97		Note 9, p. 27
36. 1,3-Dichlorobenzene	601, 602 624, 1625B	6200 C-97		Note 9, p. 27
37. 1,4-Dichlorobenzene	601, 602 624, 1625B	6200 C-97		Note 9, p. 27
38. 3,3' Dichlorobenzidine	625, 1625B, 605	6410 B-00		
39. Dichlorodifluoromethane	601	6200 C-97		
40. 1,1-Dichloroethane	601, 624, 1624B	6200 C-97		
41. 1,2-Dichloroethane	601, 624, 1624B	6200 B and C-97		
42. 1,1-Dichloroethene	601, 624, 1624B	6200 B and C-97		
43. <i>trans</i> -1,2-Dichloroethene	601, 624, 1624B	6200 B and C-97		
44. 2,4-Dichlorophenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
45. 1,2-Dichloropropane	601, 624, 1624B	6200 B and C-97		
46. <i>cis</i> -1,3-Dichloropropene	601, 624, 1624B	6200 B and C-97		

47. <i>trans</i> -1, 3-Dichloropropene	601, 624, 1624B	6200 B and C-97		
48. Diethyl phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
49. 2,4-Dimethylphenol	604, 625, 1625B			Note 9, p. 27
50. Dimethyl phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
51. Di- <i>n</i> -butyl phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
52. Di- <i>n</i> -octyl phthalate	606, 625, 1625B	6410 B-00		Note 9, p. 27
53. 2,3-Dinitrophenol	604, 625, 1625B			
54. 2,4-Dinitrotoluene	609, 625, 1625B	6410 B-00		Note 9, p. 27
55. 2,6-Dinitrotoluene	609, 625, 1625B	6410 B-00		Note 9, p. 27
56. Epichlorohydrin				Note 3, p. 130; Note 6, p. S102
57. Ethylbenzene	602, 624, 1624B	6200 B and C-97		
58. Fluoranthene	610, 625, 1625B, 610	6410 B-00	D4657-92 (98)	Note 9, p. 27
59. Fluorene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
60. 1,2,3,4,6,7,8- Heptachlorodibenzofuran	1613B			
61. 1,2,3,4,7,8,9- Heptachlorodibenzofuran	1613B			
62. 1,2,3,4,6,7,8- Heptachlorodibenzo- <i>p</i> -dioxin	1613B			
63. Hexachlorobenzene	612, 625, 1625B	6410 B-00		Note 9, p. 27
64. Hexachlorobutadiene	612, 625, 1625B	6410 B-00		Note 9, p. 27
65. Hexachlorocyclopentadiene	612, 625 <sup>5</sup> , 1625B	6410 B-00		Note 9, p. 27
66. 1,2,3,4,7,8- Hexachlorodibenzofuran	1613B			
67. 1,2,3,6,7,8- Hexachlorodibenzofuran	1613B			
68. 1,2,3,7,8,9- Hexachlorodibenzofuran	1613B			
69. 2,3,4,6,7,8- Hexachlorodibenzofuran	1613B			
70. 1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	1613B			
71. 1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	1613B			
72. 1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	1613B			
73. Hexachloroethane	616, 625, 1625B	6410 B-00		Note 9, p. 27
74. Indeno(1,2,3- <i>cd</i> ) pyrene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
75. Isophorone	609, 625, 1625B	6410 B-00		Note 9, p. 27
76. Methylene chloride	601, 624, 1624B	6200 C-97		Note 3, p. 130
77. 2-Methyl-4,6-dinitrophenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
78. Naphthalene	610, 625, 1625B	6410 B-00		Note 9, p. 27
79. Nitrobenzene	609, 625, 1625B	6410 B-00		Note 9, p. 27
80. 2-Nitrophenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
81. 4-Nitrophenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
82. <i>N</i> -Nitrosodimethylamine	607, 625 <sup>5</sup> , 1625B	6410 B-00		Note 9, p. 27
83. <i>N</i> -Nitrosodi- <i>n</i> -propylamine	607, 625, 1625B	6410 B-00		Note 9, p. 27
84. <i>N</i> -Nitrosodiphenylamine	607, 625 <sup>5</sup> , 1625B	6410 B-00		Note 9, p. 27
85. Octachlorodibenzofuran	1613B			

86. Octachlorodibenzo-p-dioxin	1613B			
87. 2,2-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether]	611, 625, 1625B	6410 B-00		
88. PCB-1016	608, 625	6410 B-00		Note 3, p. 43
89. PCB-1221	608, 625	6410 B-00		Note 3, p. 43
90. PCB-1232	608, 625	6410 B-00		Note 3, p. 43
91. PCB-1242	608, 625	6410 B-00		Note 3, p. 43
92. PCB-1248	608, 625			Note 3, p. 43
93. PCB-1254	608, 625	6410 B-00		Note 3, p. 43
94. PCB-1260	608, 625	6410 B-00		Note 3, p. 43
95. 1,2,3,7,8-Pentachlorodibenzofuran	1613B			
96. 2,3,4,7,8-Pentachlorodibenzofuran	1613B			
97. 1,2,3,7,8-Pentachlorodibenzo-p-dioxin	1613B			
98. Pentachlorophenol	604, 625, 1625B	6410 B-00		Note 3, p. 140; Note 9, p. 27
99. Phenanthrene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
100. Phenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
101. Pyrene	610, 625, 1625B	6410 B-00	D4657-92 (98)	Note 9, p. 27
102. 2,3,7,8-Tetrachlorodibenzofuran	1613B			
103. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	1613B			
104. 1,1,2-Tetrachloroethane	601, 624, 1624B	6200 B and C-97		Note 3, p. 130
105. Tetrachloroethene	601, 624, 1624B	6200 B and C-97		Note 3, p. 130
106. Toluene	602, 624, 1624B	6200 B and C-97		
107. 1,2,4-Trichlorobenzene	612, 625, 1625B	6410 B-00		Note 3, p. 130
108. 1,1,1-Trichloroethane	601, 624, 1624B	6200 B and C-97		
109. 1,1,2-Trichloroethane	601, 624, 1624B	6200 B and C-97		Note 3, p. 130
110. Trichloroethene	601, 624, 1624B	6200 B and C-97		
111. Trichlorofluoromethane	601, 624	6200 B and C-97		
112. 2,4,6-Trichlorophenol	604, 625, 1625B	6410 B-00, 6420 B-00		Note 9, p. 27
113. Vinyl chloride	601, 624, 1624B	6200 B and C-97		
114. Nonylphenol			D7065-06	
115. Bisphenol A (BPA)			D7065-06	
116. p-tert-Octylphenol (OP)			D7065-06	
117. Nonylphenol Monoethoxylate (NP1EO)			D7065-06	
118. Nonylphenol Diethoxylate (NP2EO)			D7065-06	
119. Adsorbable Organic Halides (AOX)	1650 <sup>11</sup>			
120. Chlorinated Phenolics	1653 <sup>11</sup>			

1. All parameters are expressed in micrograms per liter (ug/L) except for Method 1613 in which the parameters are expressed in picograms per liter (pg/L).

2. The full text of Methods 601-613, 624, 625, 1624B, and 1625B, are given at Appendix A. The full text of Method 1613B is incorporated by reference. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B.

3. Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978.
4. Method 624 may be used for quantitative determination of acrolein and acrylonitrile, provided that the laboratory has documentation to substantiate the ability to detect and quantify these analytes at levels necessary to comply with any associated regulations. In addition, the use of sample introduction techniques other than simple purge-and-trap may be required. QC acceptance criteria from Method 603 should be used when analyzing samples for acrolein and acrylonitrile in the absence of such criteria in Method 624.
5. Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.
6. Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the Fifteenth Edition of Standard Methods (1981).
7. Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601-603, 624, 625, 1624, and 1625B in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.
8. Organochlorine Pesticides and PCBs in Wastewater Using Empore Disk, 3M Corporation Revised 10/28/94.
- 9 USGS Method 0-3116-87 from *Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory--Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments* U.S. Geological Survey, Open File Report 93-125.
- 10 Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.
- 11 Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration. Revision C, 1997. US EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GCMS. Revision A, 1997. US EPA.

**Table ID.--List of Approved Test Procedures for Pesticides<sup>1</sup>**

Parameter	Method	EPA <sup>2,7</sup>	Standard Methods	ASTM	Other
1. Aldrin	GC	608, 617	6630 B- & C-2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> 3M0222 <sup>8</sup>
	GC/MS	625	6410 B-2000		
2. Ametryn	GC	507, 619			Notes 3, 6, O-3106-93 <sup>9</sup>
	GC/MS	525.2			O-1121-91 <sup>14</sup>
3. Aminocarb	TLC				Notes 3, 6,
	HPLC	632			
4. Atraton	GC	619			Notes 3, 6
5. Atrazine	GC	507, 619			Notes 3, 6, O-3106-93 <sup>9</sup>
	HPLC/MS				O-2060-01 <sup>12</sup>
	GC/MS	525.1, 525.2			O-1126-95 <sup>11</sup>
6. Azinphos methyl	GC	614, 622, 1657			Notes 3, 6
	GC/MS				O-1126-95 <sup>11</sup>
	TLC				Notes 3, 6,
7. Barban	HPLC	632			
	GC	608, 617	6630 B- & C-2000	D3086-90, D5812-96 (02)	Note 3, 3M0222 <sup>8</sup>
8.alpha-BHC	GC/MS	625 <sup>5</sup>	6410 B-2000		O-1126-95 <sup>11</sup>
	GC	608, 617	6630 B- & C-2000	D3086-90, D5812-96 (02)	3M0222 <sup>8</sup>
9.beta-BHC	GC/MS	625 <sup>5</sup>	6410 B-2000		
	GC	608, 617	6630 B- & C-2000	D3086-90, D5812-96 (02)	3M0222 <sup>8</sup>
10.delta-BHC	GC/MS	625 <sup>5</sup>	6410 B-2000		
	GC	608, 617	6630 B- & C-2000	D3086-90, D5812-96 (02)	3M0222 <sup>8</sup>



11. gamma-BHC (Lindane)	GC/MS	625 <sup>5</sup>	6410 B-2000	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 <sup>8</sup> .
	GC	608, 617	6630 B- & C- 2000		
12. Captan	GC/MS	625	6410 B-2000	D3086-90, D5812-96 (02)	3M0222 <sup>8</sup> , O-1126-95 <sup>11</sup> Note 3
	GC	617	6630 B-2000		
13. Carbaryl	TLC				Notes 3, 6
	HPLC	531.1, 632			
	HPLC/MS	553			
	GC/MS				
14. Carbophenothion.	GC	617	6630 B-2000	D3086-90, D5812-96 (02)	Notes 4, 6 Notes 3, 4, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
15. Chlordane	GC	608, 617	6630 B- & C- 2000		
16. Chloroprotham	GC/MS	625	6410 B-2000		Notes 3, 6,
	TLC				
17. 2,4-D	HPLC	632			Notes 3, O-3104-83 <sup>4</sup> , O-2060-01 <sup>12</sup>
	GC	615	6640 B-2001		
18. 4,4'-DDD	HPLC/MS			D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC	608, 617	6630 B- & C- 2000		
19. 4,4'-DDE	GC/MS	625	6410 B-2000	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC	608, 617	6630 B- & C- 2000		
20. 4,4'-DDT	GC/MS	625	6410 B-2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC	608, 617	6630 B- & C- 2000		
21. Demeton-O	GC/MS	625	6410 B-2000		Notes 3, 6
	GC	614, 622			
22. Demeton-S	GC	614, 622			Notes 3, 6.
23. Diazinon	GC	507, 614, 622, 1657			Notes 3, 6, O-3104-83 <sup>4</sup>
	GC/MS	525.2			
24. Dicamba	GC	615			O-1126-95 <sup>11</sup> Note 3
	HPLC/MS				
	GC	622.1			
25. Dichlofenthion	GC	622.1			Notes 4, 6
26. Dichloran	GC	608.2, 617	6630 B- -2000		Note 3
27. Dicofol.	GC	617		D3086-90, D5812-96 (02)	O-3104-83 <sup>4</sup>
28. Dieldrin	GC	608, 617	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC/MS	625	6410 B-2000		O-1126-95 <sup>11</sup>
29. Dioxathion	GC	614.1,1657			Notes 4, 6
30. Disulfoton	GC	507, 614, 622, 1657			Notes 3, 6
	GC/MS	525.2			O-1126-95 <sup>11</sup>
31. Diuron	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS	553			
32. Endosulfan I	GC	608, 617	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B-2000		O-2002-01 <sup>13</sup>
33. Endosulfan II	GC	608, 617	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B-2000		O-2002-01 <sup>13</sup>
34. Endosulfan Sulfate	GC/MS	625 <sup>5</sup>	6410 B-2000	D3086-90, D5812-96 (02)	Note 3, 3M0222 <sup>8</sup>
	GC	608, 617	6630 B- & C- 2000		

35. Endrin	GC/MS GC	625 <sup>5</sup> 608, 617, 1656	6410 B-2000 6630 B- & C- 2000	D3086-90, D5812-96 (02)	Note 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
36. Endrin aldehyde	GC/MS GC	525.2, 625 <sup>5</sup> 608, 617	6410 B-2000 6630 C-2000	D3086-90, D5812-96 (02)	3M0222 <sup>8</sup>
37. Ethion	GC/MS GC	625 <sup>5</sup> 614, 614.1,1657			O-2002-01 <sup>13</sup> Notes 4, 6
38. Fenuron	GC/MS TLC HPLC HPLC/MS	632 553			O-2002-01 <sup>13</sup> Notes 3 6
39. Fenuron-TCA.	TLC HPLC	632			O-2060-01 <sup>12</sup> Notes 3, 6
40. Heptachlor	GC	505, 508, 608, 617, 1656	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Notes 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
41. Heptachlor epoxide	GC/MS GC	525.1, 525.2, 625 608, 617	6410 B-2000 6630 C-2000	D3086-90, D5812-96 (02)	O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
42. Isodrin.	GC/MS GC	625 617	6410 B-2000 6630 C-2000		Note 6, O-3104-83 <sup>4</sup>
43. Linuron	GC HPLC HPLC/MS GC/MS	632 553			Notes 3, 6 O-2060-01 <sup>12</sup> O-1126-95 <sup>11</sup>
44. Malathion	GC	614, 1657	6630 B-2000		Notes 3, 6 O-1126-95 <sup>11</sup>
45. Methiocarb	GC/MS TLC HPLC HPLC/MS	632			Notes 3, 6 O-2060-01 <sup>12</sup>
46. Methoxychlor	GC	505, 508, 608.2, 617, 1656	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Notes 3, O-3104-83 <sup>4</sup> , 3M0222 <sup>8</sup>
47. Mexacarbate	GC/MS TLC HPLC	525.1, 525.2 632			O-1126-95 <sup>11</sup> Notes 3, 6
48. Mirex	GC	617	6630 B- & C- 2000	D3086-90, D5812-96(02)	Note 3, O-3104-83 <sup>4</sup>
49. Monuron	TLC HPLC	632			Notes 3, 6
50. Monuron-TCA	TLC HPLC	632			Notes 3, 6
51. Nuburon	TLC HPLC HPLC/MS	632			Notes 3, 6. O-2060-01 <sup>12</sup>
52. Parathion methyl	GC	614, 622, 1657	6630 B-2000		Notes 3, 4
53. Parathion ethyl	GC/MS GC GC/MS	614	6630 B-2000		O-1126-95 <sup>11</sup> Note 3 O-1126-95 <sup>11</sup>
54. PCNB	GC	608.1, 617	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Note 3

55. Perthane	GC	617		D3086-90, D5812-96(02)	O-3104-83 <sup>4</sup>
56. Prometron	GC	507, 619			Notes 3, 6, O-3106-93 <sup>9</sup>
	GC/MS	525.2			O-1126-95 <sup>11</sup>
57. Prometryn	GC	507, 619			Notes 3, 6, O-3106-93 <sup>9</sup>
	GC/MS	525.1, 525.2			O-2002-01 <sup>13</sup>
58. Propazine	GC	507, 619, 1656			Notes 3, 6, O-3106-93 <sup>9</sup>
	GC/MS	525.1, 525.2			
59. Propham	TLC				Notes 3, 6, O-3106-93 <sup>9</sup>
	HPLC	632			
	HPLC/MS				O-2060-01 <sup>12</sup>
60. Propoxur	TLC				Notes 3, 6
	HPLC	632			
61. Secbumeton	TLC				Notes 3, 6
	GC	619			
62. Siduron	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS				O-2060-01 <sup>12</sup>
63. Simazine	GC	505, 507, 619, 1656			Notes 3, 6, O-3106-93 <sup>9</sup>
	GC/MS	525.1, 525.2			O-1126-95 <sup>11</sup>
64. Strobane	GC	608.1, 617	6630 B- & C- 2000		Note 3
65. Swep	TLC				Notes 3, 6
	HPLC	632			
66. 2,4,5-T	GC	615	6640 B-2001		Note 3, O-3104-83 <sup>4</sup>
67. 2,4,5-TP (Silvex)	GC	615	6640 B-2001		Note 3, O-3104-83 <sup>4</sup>
68. Terbutylazine	GC	619, 1656			Notes 3, 6
	GC/MS				O-2002-01 <sup>13</sup>
69. Toxaphene	GC	505, 508, 608, 617, 1656	6630 B- & C- 2000	D3086-90, D5812-96 (02)	Notes 3, 8, O-3105-83 <sup>4</sup>
	GC/MS	525.1, 525.2, 625	6410 B-2000		
70. Trifluralin	GC	627, 1656	6630 B-2000		Notes 3, O-3106-93 <sup>9</sup>
	GC/MS	525.2			O-1126-95 <sup>11</sup>

Notes:

1. Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.
2. The full text of Methods 608 and 625 are given at Appendix A. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B.
3. 'Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,' U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.
4. 'Methods for Analysis of Organic Substances in Water and Fluvial Sediments,' Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).
5. The method may be extended to include a-BHC, g-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
6. Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency.' Supplement to the Fifteenth Edition of Standard Methods (1981).
7. Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be

reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

8. Organochlorine Pesticides and PCBs in Wastewater Using Empore Disk, 3M Corporation, Revised 10/28/94.

9 Method O-3106-93 is in Open File Report 94-37, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors. 1994. USGS..

10 EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, EPA 821-R-92-002, April 1992, US EPA. The full text of Methods 608 and 625 are provided at Appendix A, Test Procedures for Analysis of Organic Pollutants, of this Part 136. EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, 1993, US EPA. EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.0, 1995, US EPA. EPA methods 1656 and 1657 are in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, 1993, US EPA..

11 Method O-1126-95 is in Open-File Report 95-181, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1995. USGS.

12 Method O-2060-01 is in Water-Resources Investigations Report 01-4134, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory-Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry. 2001. USGS.

13 Method O-2002-01 is in Water-Resources Investigations Report 01-4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of moderate-use pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry. 2001. USGS.

14 Method O-1121-91 is in Open-File Report 91-519, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of organonitrogen herbicides in water by solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring. 1992. USGS.

**Table IE.--List of Approved Radiological Test Procedures**

<b>Parameter and units</b>	<b>Method</b>	<b>EPA<sup>1</sup></b>	<b>Standard Methods, 18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup> Ed</b>	<b>Standard Methods Online</b>	<b>ASTM</b>	<b>USGS<sup>2</sup></b>
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	7110 B	7110 B-00	D1943-90, 96	p 75 and 78 <sup>3</sup>
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	7110 B-00	D1943-90, 96	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	7110 B-00	D1890-90, 96	p 75 and 78 <sup>3</sup>
4. Beta-Counting error, pCi per liter	Proportional counter	Appendix B	7110 B	7110 B-00	D1890-90, 96	p 79
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	7500Ra B-01	D2460-90, 97	
(b) 226Ra, pCi per liter	Scintillation counter	903.1	7500Ra C	7500Ra C-01	D3454-91, 97	p 81

Notes:

1 ``Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980), U.S. Environmental Protection Agency, August 1980.

2 Fishman, M.J. and Brown, Eugene, ``Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

3 The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the ``total".

**Table IF.--List of Approved Methods for Pharmaceutical Pollutants**

<b>Pharmaceuticals pollutants</b>	<b>CAS No.</b>	<b>Analytical method number</b>
acetonitrile	75-05-8	1666/1671/D3371/D3695
n-amyl acetate	628-63-7	1666/D3695
n-amyl alcohol	71-41-0	1666/D3695
benzene	71-43-2	D4763/D3695/502.2/524.2
n-butyl-acetate	123-86-4	1666/D3695
tert-butyl alcohol	75-65-0	1666
chlorobenzene	108-90-7	502.2/524.2
chloroform	67-66-3	502.2/524.2/551
o-dichlorobenzene	95-50-1	1625C/502.2/524.2
1,2-dichloroethane	107-06-2	D3695/502.2/524.2
diethylamine	109-89-7	1666/1671
dimethyl sulfoxide	67-68-5	1666/1671
ethanol	64-17-5	1666/1671/D3695
ethyl acetate	141-78-6	1666/D3695
n-heptane	142-82-5	1666/D3695
n-hexane	110-54-3	1666/D3695
isobutyraldehyde	78-84-2	1666/1667
isopropanol	67-63-0	1666/D3695
isopropyl acetate	108-21-4	1666/D3695.
isopropyl ether	108-20-3	1666/D3695
methanol	67-56-1	1666/1671/D3695
Methyl Cellosolve	109-86-4	1666/1671
methylene chloride	75-09-2	502.2/524.2
methyl formate	107-31-3	1666
4-methyl-2-pentanone (MIBK)	108-10-1	1624C/1666/D3695/D4763/524.2
phenol	108-95-2	D4763
n-propanol	71-23-8	1666/1671/D3695.
2-propanone (acetone)	67-64-1	D3695/D4763/524.2
tetrahydrofuran	109-99-9	1666/524.2
toluene	108-88-3	D3695/D4763/502.2/524.2
triethylamine	121-44-8	1666/1671
xylenes	(Note 1)	1624C/1666
1. m-xylene 108-38-3, o,p-xylene E-14095 (Not a CAS number; this is the number provided in the Environmental Monitoring Methods Index (EMMI) database.); m,p-xylene 136777-61-2, o-xylene 95-47-6.		

[Editor's Note: This table had previously existed in 40 CFR Part 455, and while it is new to this location, the information is not new.]

**Table IG.—Test Methods for Pesticide Active Ingredients**

<b>EPA Survey Code / Pesticide name</b>	<b>CAS No.</b>	<b>EPA Analytical Method No.(s)</b>
8 Triadimefon	43121-43-3	507/633/525.1/1656
12 Dichlorvos	62-73-7 1	657/507/622/525.1/525.2
16 2,4-D; 2,4-D Salts and Esters [2,4-Dichlorophenoxyacetic acid]	94-75-7	1658/515.1/615/515.2/555
17 2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid]	94-82-6	1658/515.1/615/515.2/555
22 Mevinphos	7786-34-7	1657/507/622/525.1/525.2
25 Cyanazine	21725-46-2	629/507
26 Propachlor	1918-16-7	1656/508/608.1/525.1/525.2

27 MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid]	94-74-6	1658/615/555
30 Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid]	120-36-5	1658/515.1/615/515.2/555
31 MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid]	93-65-2	1658/615/555
35 TCMTB [2-(Thiocyanomethylthio) benzothiazole]	21564-17-0	637
39 Pronamide	23950-58-5	525.1/525.2/507/633.1
41 Propanil	709-98-8	632.1/1656
45 Metribuzin	21087-64-9	507/633/525.1/1656
52 Acephate	30560-19-1	1656/1657
53 Acifluorfen	50594-66-6	515.1/515.2/555
54 Alachlor	15972-60-8	505/507/645/525.1/525.2/1656
55 Aldicarb	116-06-3	531.1
58 Ametryn	834-12-8	507/619/525.2
60 Atrazine	1912-24-9	505/507/619/525.1/525.2/1656
62 Benomyl	17804-35-2	631
68 Bromacil; Bromacil Salts and Esters	314-40-9	507/633/525.1/525.2/1656
69 Bromoxynil	1689-84-5	1625/1661
69 Bromoxynil octanoate	1689-99-2	1656
70 Butachlor	23184-66-9	507/645/525.1/525.2/1656
73 Captafol	2425-06-1	1656
75 Carbaryl [Sevin]	63-25-2	531.1/632/553
76 Carbofuran	1563-66-2	531.1/632
80 Chloroneb	2675-77-6	1656/508/608.1/525.1/525.2
82 Chlorothalonil	1897-45-6	508/608.2/525.1/1656
84 Stirofos	961-11-5	1657/507/622/525.1/525.2
86 Chlorpyrifos	2921-88-2	1657/508/622
90 Fenvalerate	51630-58-1	1660
103 Diazinon	333-41-5	1657/507/614/622/525.2
107 Parathion methyl	298-00-0	1657/614/622
110 DCPA [Dimethyl 2,3,5,6-tetrachloroterephthalate]	1861-32-1	508/608.2/525.1/525.2/515.1 <sup>2</sup> /515.2 <sup>2</sup> /1656
112 Dinoseb	88-85-7	1658/515.1/615/515.2/555
113 Dioxathion	78-34-2	1657/614.1
118 Nabonate [Disodium cyanodithioimidocarbonate]	138-93-2	630.1
119 Diuron	330-54-1	632/553
123 Endothall	145-73-3	548/548.1
124 Endrin	72-20-8	1656/505/508/608/617/525.1/525.2
125 Ethalfluralin	55283-68-6	1656/627 <sup>1</sup>
126 Ethion	563-12-2	1657/614/614.1
127 Ethoprop	13194-48-4	1657/507/622/525.1/525.2
132 Fenarimol	60168-88-9	507/633.1/525.1/1656
133 Fenthion	55-38-9	1657/622
138 Glyphosate [N-(Phosphonomethyl) glycine]	1071-83-6	547
140 Heptachlor	76-44-8	1656/505/508/608/617/525.1/525.2
144 Isopropalin	33820-53-0	1656/627
148 Linuron	330-55-2	553/632
150 Malathion	121-75-5	1657/614
154 Methamidophos	10265-92-6	1657
156 Methomyl	16752-77-5	531.1/632
158 Methoxychlor	72-43-5	1656/505/508/608.2/617/525.1/525.2
172 Nabam	142-59-6	630/630.1
173 Naled	300-76-5	1657/622
175 Norflurazon	27314-13-2	507/645/525.1/525.2/1656

178 Benfluralin	1861-40-1 1	1656/1627
182 Fensulfothion	15-90-2	1657/622
183 Disulfoton	298-04-4	1657/507/614/622/525.2
185 Phosmet	732-11-6	1657/622.1
186 Azinphos Methyl	86-50-0	1657/614/622
192 Organo-tin pesticides	12379-54-3	Ind-01/200.7/200.9
197 Bolstar	35400-43-2	1657/622
203 Parathion	56-38-2	1657/614
204 Pendimethalin	40487-42-1	1656
205 Pentachloronitrobenzene	82-68-8	1656/608.1/617
206 Pentachlorophenol	87-86-5	625/1625/515.2/555/515.1/ 525.1/525.2
208 Permethrin	52645-53-1	608.2/508/525.1/525.2/1656/1660
212 Phorate	298-02-2	1657/622
218 Busan 85 [Potassium dimethyldithiocarbamate]	128-03-0	630/630.1
219 Busan 40 [Potassium N-hydroxymethyl-Nmethylthiocarbamate]	51026-28-9	630/630.1
220 KN Methyl [Potassium N-methyldithiocarbamate]	137-41-7	630/630.1
223 Prometon	1610-18-0	507/619/525.2
224 Prometryn	7287-19-6	507/619/525.1/525.2
226 Propazine	139-40-2	507/619/525.1/525.2/1656
230 Pyrethrin I	121-21-1	1660
232 Pyrethrin II	121-29-9	1660
236 DEF [S,S,S-Tributyl phosphorotrithioate]	78-48-8 1657	
239 Simazine	122-34-9	505/507/619/525.1/525.2/1656
241 Carbam-S [Sodium dimethyldithiocarbamate]	128-04-1	630/630.1
243 Vapam [Sodium methyldithiocarbamate]	137-42-8	630/630.1
252 Tebuthiuron	34014-18-1	507/525.1/525.2
254 Terbacil	5902-51-2	507/633/525.1/1656
255 Terbufos	13071-79-9	1657/507/614.1/525.1/525.2
256 Terbutylazine	5915-41-3	619/1656
257 Terbutryn	886-50-0	507/619/525.1/525.2
259 Dazomet	533-74-4	630/630.1/1659
262 Toxaphene	8001-35-2	1656/505/508/608/617/525.1/525.2
263 Merphos [Tributyl phosphorotrithioate]	150-50-5	1657/507/525.1/525.2/622
264 Trifluralin	1582-09-8	1656/508/617/627/525.1/525.2
268 Ziram [Zinc dimethyldithiocarbamate]	137-30-4	630/630.1

1 Monitor and report as total Trifluralin.

2 Applicable to the analysis of DCPA degradates.

3 EPA Methods 608.1 through 645, 1645 through 1661, and Ind-01 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, Revision I, August 1993, US EPA. EPA Methods 200.9 and 505 through 555 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, August 1993, US EPA.. The full text of Methods 608, 625 and 1625 are provided at Appendix A of this Part 136. The full text of Method 200.7 is provided at Appendix C of this Part 136.

**Table IH.--List of Approved Biological Methods for Ambient Water**

Parameter and units	Method <sup>1</sup>	EPA	Standard Methods Online	ASTM <sup>10</sup> AOAC <sup>11</sup> USGS <sup>5</sup>	Other
<b>Bacteria:</b>					
Coliform (fecal), number per 100 mL or number per gram dry weight	Most Probable Number (MPN), 5 tube, 3 dilution, or Membrane filter (MF) <sup>2</sup> , single step	p. 132 <sup>3</sup>	9221 C E-06		
		p. 124 <sup>3</sup>	9222 D-97	B-0050-85 <sup>4</sup>	



2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 132 <sup>3</sup>	9221 C E-06		
	MF2, single step <sup>5</sup>	p. 124 <sup>3</sup>	9222 D-97		
3. Coliform (total), number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114 <sup>3</sup>	9221 B-06		
	MF2, single step or two step	p. 108 <sup>3</sup>	9222 B-97	B-0025-85 <sup>4</sup>	
4. Coliform (total), in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114 <sup>3</sup>	9221 B-06		
	MF2 with enrichment	p. 111 <sup>3</sup>	9222 (B+B.5c)-1997		
5. E. coli, number per 100 mL	MPN <sup>6,8,14</sup> multiple tube		9221 B.1-06/ 9221 F-06 <sup>11,13</sup>		
	multiple tube/ multiple well		9223 B-04 <sup>12</sup>	991.15 <sup>10</sup>	Colilert <sup>12,16</sup> Colilert-18 <sup>12,15,16</sup>
	MF <sup>2,5,6,7,8</sup> two step, or	1103.1 <sup>19</sup>	9222 B-97/ 9222 G-97 <sup>18</sup> 9213 D-97	D5392-93 <sup>9</sup>	
	single step	1603 <sup>20</sup> 1604 <sup>21</sup>			mColiBue 24 <sup>17</sup>
6. Fecal streptococci, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 139 <sup>3</sup>	9230 B-07		
	MF <sup>2</sup> , or Plate count	p. 136 <sup>3</sup> p. 143 <sup>3</sup>	9230 C-07	B-0055-85 <sup>4</sup>	
7. Enterococci, number per 100 mL.	MPN <sup>6,8</sup> multiple tube			D6503-99 <sup>9</sup>	Enterolert <sup>12,22</sup>
	multiple tube/ multiple well		9230 C-07	D5259-92 <sup>9</sup>	
	MF <sup>2,5,6,7,8</sup> two step	1106.1 <sup>23</sup>	9230 C-07		
	single step	1600 <sup>24</sup>			
	Plate count	p. 143 <sup>3</sup>			
Protozoa:					
9. <i>Cryptosporidium</i> <sup>28</sup>	Filtration/IMS/FA	1622 <sup>25</sup> 1623 <sup>26</sup>			
10. <i>Giardia</i> <sup>28</sup>	Filtration/IMS/FA	1623 <sup>26</sup>			

1 The method must be specified when results are reported.

2 A 0.45 um membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

3 USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017.

4 U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS

5 Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

6 Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

7 When the MF method has not been used previously to test waters with high turbidity, large number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

8 To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

9 Annual Book of ASTM Standards--Water and Environmental Technology. Section 11.02. 2000, 1999, 1996. ASTM.

10 Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 1995. AOAC.

11 The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and



this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

12 These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme beta-glucuronidase produced by *E. coli*.

13 After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 ug/mL of MUG may be used.

14 Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® or Quanti-Tray® 2000, and the MPN calculated from the table provided by the manufacturer.

15 Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 C rather than the 24 h required for the Colilert® test and is recommended for marine water samples.

16 Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories, Inc.

17 A description of the mColiBlue24® test, Total Coliforms and *E. coli*, is available from Hach Company.

18 Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.

19 Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). EPA-821-R-10-002. March 2010. USEPA.

20 Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). EPA-821-R-09-007. December 2009. USEPA.

21 Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. *New Medium for the Simultaneous Detection of Total Coliform and Escherichia coli in Water*. Appl. Environ. Microbiol. 59:3534-3544 and in: Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). DC EPA 821-R-02-024. September 2002. USEPA.

22 A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc.

23 Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA). EPA-821-R-09-015. December 2009. USEPA.

24 Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-beta-D-Glucoside Agar (mEI). EPA-821-R-09-016. December 2009. USEPA.

25 Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. EPA-821-R-05-001. December 2005. USEPA.

26 Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts.. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. EPA-821-R-05-002. December 2005 USEPA.

[Editor's Note: The section which follows was completely reformatted and is not shown as a change.]

#### References, Sources, Costs, and Table Citations:

The documents required in this section are incorporated by reference into this section with approval of the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the documents may be obtained from the sources listed this section. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue, NW., Room B102, Washington, DC (Telephone: 202-566-2426); or at the National Archives and Records

Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: [http://www.archives.gov/federal\\_register/code\\_of\\_federal\\_regulations/ibr\\_locations.html](http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html). These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the Federal Register. The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, IE, IF, IG and IH are incorporated by reference into this regulation and may be obtained from the source identified. All costs cited are subject to change and must be verified from the indicated source.

(1) Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161

(i) Microbiological Methods for Monitoring the Environment, Water, and Wastes. 1978. EPA/600/8-78/017, Pub. No. PB-290329/A.S.

(A) Part III Analytical Methodology, Section B Total Coliform Methods, page 108. Table Page 106 of 293 IA, Note 3; Table IH, Note 3.

(B) Part III Analytical Methodology, Section B Total Coliform Methods, 2.6.2 Two-Step Enrichment Procedure, page 111. Table IA, Note 3; Table IH, Note 3.

(C) Part III Analytical Methodology, Section B Total Coliform Methods, 4 Most Probable Number (MPN) Method, page 114. Table IA, Note 3; Table IH, Note 3.

(D) Part III Analytical Methodology, Section C Fecal Coliform Methods, 2 Direct Membrane Filter (MF) Method, page 124. Table IA, Note 3; Table IH, Note 3.

(E) Part III, Analytical Methodology, Section C Fecal Coliform Methods, 5 Most Probable Number (MPN) Method, page 132. Table IA, Note 3; Table IH, Note 3.

(F) Part III Analytical Methodology, Section D Fecal Streptococci, 2 Membrane Filter (MF) Method, page 136. Table IA, Note 3; Table IH, Note 3.

(G) Part III Analytical Methodology, Section D Fecal Streptococci, 4 Most Probable Number Method, page 139. Table IA, Note 3; Table IH, Note 3.

(H) Part III Analytical Methodology, Section D Fecal Streptococci, 5 Pour Plate Method, page 143. Table IA, Note 3; Table IH, Note 3.

(ii) [Reserved]

(2) Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.

(i) Method 300.1 (including Errata Cover Sheet, April 27, 1999), Determination of Inorganic Ions in Drinking Water by Ion Chromatography, Revision 1.0, 1997. Table IB, Note 52.

(ii) Method 551, Determination of Chlorination Disinfection Byproducts and Chlorinated Solvents in Drinking Water by Liquid-Liquid Extraction and Gas Chromatography With Electron-Capture Detection. 1990. Table IF.

(3) National Exposure Risk Laboratory-Cincinnati, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available from <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800-553-6847.

(i) Methods for the Determination of Inorganic Substances in Environmental Samples. August 1993. EPA/600/R-93/100, Pub. No. PB 94120821. Table IB, Note 52.

(A) Method 180.1, Determination of Turbidity by Nephelometry. Revision 2.0. Table IB, Note 52.

(B) Method 300.0, Determination of Inorganic Anions by Ion Chromatography. Revision 2.1. Table IB, Note 52.

(C) Method 335.4, Determination of Total Cyanide by Semi-Automated Colorimetry. Revision 1.0. Table IB, Notes 52 and 57.

(D) Method 350.1, Determination of Ammonium Nitrogen by Semi-Automated Colorimetry. Revision 2.0. Table IB, Notes 30 and 52.

(E) Method 351.2, Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(F) Method 353.2, Determination of Nitrate-Nitrite Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(G) Method 365.1, Determination of Phosphorus by Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(H) Method 375.2, Determination of Sulfate by Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(I) Method 410.4, Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry. Revision 2.0. Table IB, Note 52.

(ii) Methods for the Determination of Metals in Environmental Samples, Supplement I. May 1994. EPA/600/R-94/111, Pub. No. PB 95125472. Table IB, Note 52.

(A) Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry. Revision 4.4. Table IB, Note 52.

(B) Method 200.8, Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma Mass Spectrometry. Revision 5.3. Table IB, Note 52.

(C) Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry. Revision 2.2. Table IB, Note 52.

(D) Method 218.6, Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by Ion Chromatography. Revision 3.3. Table IB, Note 52.

(E) Method 245.1, Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry. Revision 3.0. Table IB, Note 52.

(4) National Exposure Risk Laboratory-Cincinnati, U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>.

(i) EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry. Revision 4.2, October 2003. EPA/600/R-06/115. Table IB, Note 68.

(ii) EPA Method 525.2, Determination of Organic Compounds in Drinking Water by Liquid- Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry. Revision 2.0, 1995. Table ID, Note 10.

(5) Office of Research and Development, Cincinnati OH. U.S. Environmental Protection Agency, Cincinnati OH (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati OH 45268.

(i) Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater. 1978. Table IC, Note 3; Table ID, Note 3.

(ii) Methods for Chemical Analysis of Water and Wastes. March 1979. EPA-600/4-79-020. Table IB, Note 1.

(iii) Methods for Chemical Analysis of Water and Wastes. Revised March 1983. EPA-600/4-79-020. Table IB, Note 1

(A) Method 120.1, Conductance, Specific Conductance,  $\mu\text{mhos}$  at  $25^{\circ}\text{C}$ . Revision 1982. Table IB, Note 1.

(B) Method 130.1, Hardness, Total (mg/L as  $\text{CaCO}_3$ ), Colorimetric, Automated EDTA. Issued 1971. Table IB, Note 1.

(C) Method 150.2, pH, Continuous Monitoring (Electrometric). December 1982. Table IB, Note 1.

(D) Method 160.4, Residue, Volatile, Gravimetric, Ignition at  $550^{\circ}\text{C}$ . Issued 1971. Table IB, Note 1.

(E) Method 206.5, Arsenic, Sample Digestion Prior to Total Arsenic Analysis by Silver Diethyldithiocarbamate or Hydride Procedures. Issued 1978. Table IB, Note 1.

(F) Method 231.2, Gold, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(G) Method 245.2, Mercury, Automated Cold Vapor Technique. Issued 1974. Table IB, Note 1.

(H) Method 252.2, Osmium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(I) Method 253.2, Palladium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(J) Method 255.2, Platinum, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(K) Method 265.2, Rhodium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(L) Method 279.2, Thallium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(M) Method 283.2, Titanium, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(N) Method 289.2, Zinc, Atomic Absorption, Furnace Technique. Issued 1978. Table IB, Note 1.

(O) Method 310.2, Alkalinity, Colorimetric, Automated, Methyl Orange. Revision 1974. Table IB, Note 1.

(P) Method 351.1, Nitrogen, Kjeldahl, Total, Colorimetric, Automated Phenate. Revision 1978. Table IB, Note 1.

(Q) Method 352.1, Nitrogen, Nitrate, Colorimetric, Brucine. Issued 1971. Table IB, Note 1.

(R) Method 365.3, Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Two Reagent. Issued 1978. Table IB, Note 1.

(S) Method 365.4, Phosphorus, Total, Colorimetric, Automated, Block Digestor AA II. Issued 1974. Table IB, Note 1.

(T) Method 410.3, Chemical Oxygen Demand, Titrimetric, High Level for Saline Waters. Revision 1978. Table IB, Note 1.

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(B) Method 903.0, Alpha-Emitting iRadio Isotopes. Table IE.

(C) Method 903.1, Radium-226, Radon Emanation Technique. Table IE.

(D) Appendix B, Error and Statistical Calculations. Table IE.

(6) Office of Science and Technology, U.S. Environmental Protection Agency, Washington DC (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm>

(i) Method 1625C, Semivolatile Organic Compounds by Isotope Dilution GCMS. 1989. Table IF.

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(7) Office of Water, U.S. Environmental Protection Agency, Washington DC (US EPA). Available at <http://water.epa.gov/scitech/methods/cwa/index.cfm> or from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161

(i) Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. Revision E, August 2002. EPA-821-R-02-019, Pub. No. PB2002-108220. Table IB, Note 43.

(ii) Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate. Revision 1.2, August 2001. EPA 821-B-01-009, Pub. No. PB 2001-108275. Table IB, Note 55.

(iii) In the compendium Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters. July 1998. EPA 821-B-98-016, Pub. No. PB95201679. Table IF, Note 1.

(A) EPA Method 1666, Volatile Organic Compounds Specific to the Pharmaceutical Industry by Isotope Dilution GC/MS. Table IF, Note 1.

(B) EPA Method 1667, Formaldehyde, Isobutyraldehyde, and Furfural by Derivatization Followed by High Performance Liquid Chromatography. Table IF

(C) Method 1671, Volatile Organic Compounds Specific to the Pharmaceutical Manufacturing Industry by GC/FID. Table IF.

(iv) Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I. Revision I, August 1993. EPA 821-R-93-010A, Pub. No. PB 94121654. Tables ID, IG

(A) Method 608.1, Organochlorine Pesticides. Table ID, Note 10; Table IG, Note 3

(B) Method 608.2, Certain Organochlorine Pesticides. Table ID, Note 10; Table IG, Note 3

- (C) Method 614, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3
- (D) Method 614.1, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3
- (E) Method 615, Chlorinated Herbicides. Table ID, Note 10; Table IG, Note 3
- (F) Method 617, Organohalide Pesticides and PCBs. Table ID, Note 10; Table IG, Note 3
- (G) Method 619, Triazine Pesticides. Table ID, Note 10; Table IG, Note 3
- (H) Method 622, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3
- (I) Method 622.1, Thiophosphate Pesticides. Table ID, Note 10; Table IG, Note 3
- (J) Method 627, Dinitroaniline Pesticides. Table ID, Note 10; Table IG, Notes 1 and 3
- (K) Method 629, Cyanazine. Table IG, Note 3
- (L) Method 630, Dithiocarbamate Pesticides. Table IG, Note 3
- (M) Method 630.1, Dithiocarbamate Pesticides. Table IG, Note 3
- (N) Method 631, Benomyl and Carbendazim. Table IG, Note 3
- (O) Method 632, Carbamate and Urea Pesticides. Table ID, Note 10; Table IG, Note 3
- (P) Method 632.1, Carbamate and Amide Pesticides. Table IG, Note 3
- (Q) Method 633, Organonitrogen Pesticides. Table IG, Note 3
- (R) Method 633.1, Neutral Nitrogen-Containing Pesticides. Table IG, Note 3
- (S) Method 637, MBTS and TCMTB. Table IG, Note 3
- (T) Method 644, Picloram. Table IG, Note 3
- (U) Method 645, Certain Amine Pesticides and Lethane. Table IG, Note 3
- (V) Method 1656, Organohalide Pesticides. Table ID, Note 10; Table IG, Notes 1 and 3
- (W) Method 1657, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3
- (X) Method 1658, Phenoxy-Acid Herbicides. Table IG, Note 3
- (Y) Method 1659, Dazomet. Table IG, Note 3
- (Z) Method 1660, Pyrethrins and Pyrethroids. Table IG, Note 3
- (AA) Method 1661, Bromoxynil. Table IG, Note 3
- (BB) Ind-01. Methods EV-024 and EV-025, Analytical Procedures for Determining Total Tin and Triorganotin in Wastewater. Table IG, Note 3

(v) Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II. August 1993. EPA 821-R-93-010B, Pub. No. PB 94166311. Table IG.

- (A) Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry. Table IG, Note 3
- (B) Method 505, Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography. Table ID, Note 10; Table IG, Note 3
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(viii) m-ColiBlue24® Method, for total Coliforms and E. coli. Revision 2, 1999. Table IA, Note 18; Table IH, Note 17.

(20) IDEXX Laboratories Inc., One Idexx Drive, Westbrook ME 04092.

(i) Colilert® Method. 2002. Table IA, Notes 17 and 18; Table IH, Notes 14, 15 and 16.

(ii) Colilert-18® Method. 2002. Table IA, Notes 17 and 18; Table IH, Notes 14, 15 and 16.

(iii) Enterolert® Method. 2002. Table IA, Note 24; Table IH, Note 12.

(iv) Quanti-Tray® Method. 2002. Table IA, Note 18; Table IH, Notes 14 and 16.

(v) Quanti-Tray®/2000 Method. 2002. Table IA, Note 18; Table IH, Notes 14 and 16.

(21) In-Situ Incorporated, 221 E. Lincoln Ave., Ft. Collins CO 80524. Telephone: 970-498-1500.

(i) In-Situ Inc. Method 1002-8-2009, Dissolved Oxygen Measurement by Optical Probe. 2009. Table IB, Note 64.

(ii) In-Situ Inc. Method 1003-8-2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 2009. Table IB, Note 10.

(iii) In-Situ Inc. Method 1004-8-2009, Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe. 2009. Table IB, Note 35.

(22) Journal of Chromatography, Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York NY 10164. (Also available from most public libraries.)

(i) Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography. Addison, R.F. and R.G. Ackman. 47(3): 421–426, 1970. Table IB, Note 28

(ii) [Reserved]

(23) Lachat Instruments, 6645 W. Mill Road, Milwaukee WI 53218, Telephone: 414–358–4200.

(i) QuikChem Method 10–204–00–1–X, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis. Revision 2.2, March 2005. Table IB, Note 56.

(ii) [Reserved]

(24) Leck Mitchell, Ph.D., P.E., 656 Independence Valley Dr., Grand Junction CO 81507. Telephone: 970-244-8661.

(i) Mitchell Method M5271, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Table IB, Note 66.

(ii) Mitchell Method M5331, Determination of Turbidity by Nephelometry. Revision 1.0, July 31, 2008. Table IB, Note 65.

(25) National Council of the Paper Industry for Air and Stream Improvements, Inc. (NCASI), 260 Madison Avenue, New York NY 10016.

(i) NCASI Technical Bulletin No. 253, An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color. December 1971. Table IB, Note 18.

(ii) [Reserved]

(26) Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station TX 77840.

(i) OIC Chemical Oxygen Demand Method. 1978. Table IB, Note 13.

(ii) [Reserved]

(27) OI Analytical, Box 9010, College Station TX 77820-9010.

(i) Method OIA–1677-09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA). Copyright 2010. Table IB, Note 59.

(ii) Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection. Revised December 22, 1994. Table IB, Note 39.

(iii) Method PAI-DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection. Revised December 22, 1994. Table IB, Note 40.

(iv) Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. Table IB, Note 41.

(28) ORION Research Corporation, 840 Memorial Drive, Cambridge, Massachusetts 02138.

(i) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97–70. 1977. Table IB, Note 16.

(ii) [Reserved]

(29) Technicon Industrial Systems, Tarrytown NY 10591.

(i) Industrial Method Number 379–75WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II. February 19, 1976. Table IB, Note 7.

(ii) [Reserved]

(30) Thermo Jarrell Ash Corporation, 27 Forge Parkway, Franklin MA 02038.

(i) Method AES0029. Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes. 1986, Revised 1991. Table IB, Note 34.

(ii) [Reserved]

(31) Thermo Scientific, 166 Cummings Center, Beverly MA 01915. Telephone: 1-800-225- 1480.  
www.thermoscientific.com.

(i) Thermo Scientific Orion Method AQ4500, Determination of Turbidity by Nephelometry. Revision 5, March 12, 2009. Table IB, Note 67.

(ii) [Reserved]

(32) 3M Corporation, 3M Center Building 220–9E–10, St. Paul MN 55144–1000.

(i) Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk™ Test Method 3M 0222. Revised October 28, 1994. Table IC, Note 8; Table ID, Note 8.

(ii) [Reserved]

(33) U.S. Geological Survey (USGS), U.S. Department of the Interior, Reston, Virginia. Available from USGS Books and Open-File Reports (OFR) Section, Federal Center, Box 25425, Denver, CO 80225.

(i) OFR 76–177, Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters. 1976. Table IE, Note 2.

(ii) OFR 91-519, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Organonitrogen Herbicides in Water by Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry With Selected-Ion Monitoring. 1992. Table ID, Note 14.

- (iii) OFR 92–146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by a Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. Table IB, Note 48.
- (iv) OFR 93–125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. Table IB, Note 51; Table IC, Note 9.
- (v) OFR 93–449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1993. Table IB, Note 46.
- (vi) OFR 94–37, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors. 1994. Table ID, Note 9.
- (vii) OFR 95-181, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Pesticides in Water by C-18 Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry With Selected-Ion Monitoring. 1995. Table ID, Note 11.
- (viii) OFR 97–198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. Table IB, Note 47.
- (ix) OFR 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-Water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. Table IB, Note 50.
- (x) OFR 98–639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace—Atomic Absorption Spectrometry. 1999. Table IB, Note 49.
- (xi) OFR 00–170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. Table IB, Note 45.
- (xii) Water-Resources Investigation Report 01-4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Moderate-Use Pesticides and Selected Degradates in Water by C-18 Solid-Phase Extraction and Gas Chromatography/Mass Spectrometry. 2001. Table ID, Note 13.
- (xiii) Water-Resources Investigations Report 01-4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Organic Plus Inorganic Mercury in

Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry. 2001. Table IB, Note 71.

(xiv) Water-Resources Investigation Report 01-4134, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry. 2001. Table ID, Note 12.

(xv) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, editors, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. Table IB, Note 8.

(xvi) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1989. Table IB, Note 2.

(xvii) Methods for the Determination of Organic Substances in Water and Fluvial Sediments. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3. 1987. Table IB, Note 24; Table ID, Note 4.

(xviii) Techniques and Methods Book 5-B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry. Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis. 2006. Table IB, Note 70.

(xix) U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. Table IA, Note 4; Table IH, Note 4.

(x) Water Temperature—Influential Factors, Field Measurement and Data Presentation, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1. 1975. Table IB, Note 32.

(35) Waters Corporation, 34 Maple Street, Milford MA 01757, Telephone: 508/482–2131.

(i) Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. Revision 2, December 2000. Table IB, Note 54.

(ii) [Reserved]

Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon the recommendation of the Director of EMSL

Under certain circumstances, the Administrator may approve, upon recommendation by the Director, EMSL, additional alternate test procedures for nationwide use.

e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, IE, 1F, IG and 1H are prescribed in Table II. **Information in the table takes precedence over information in specific methods or elsewhere.** Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for **such limited use changes** may be made by letters to the Regional **Alternative Test Procedure (ATP) Program Coordinator or the permitting authority** in the Region in which the discharge will occur. Sufficient data should be provided to assure such **changes in sample preservation, containers or holding times** do not adversely affect the integrity of the sample. **The Regional ATP Coordinator or permitting authority will review the application and then notify the applicant and the appropriate State agency of approval or rejection of the use of the alternate test procedure.** A decision to approve or deny **any request on deviations from the prescribed Table II requirements** will be made within 90 days of receipt of the application by the Regional Administrator. **An analyst may not modify any sample preservation and/or holding time requirements of an approved method unless the requirements of this section are met.**

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

<b>Parameter No./name</b>	<b>Container<sup>1</sup></b>	<b>Preservation<sup>2,3</sup></b>	<b>Maximum holding time<sup>4</sup></b>
<b>Table IA-Bacteria Tests:</b>			
1-5 Coliform, total, fecal and E. coli	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22,23</sup>
6 Fecal streptococci	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22</sup>
7 Enterocci	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22</sup>
8 Salmonella	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22</sup>
<b>Table IA-Aquatic Toxicity Tests:</b>			
9-11 Toxicity, acute and chronic	P, FP, G	Cool, 4°C <sup>16</sup>	36 hours.
<b>Table IB-Inorganic Tests:</b>			
1. Acidity	P, FP, G	Cool, < 6°C <sup>18</sup>	14 days
2. Alkalinity	P, FP, G	Cool, < 6°C <sup>18</sup>	14 days
4. Ammonia	P, FP, G	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
10. Boron	P, FP, or Quartz	HNO <sub>3</sub> 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 <sup>18</sup>	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, FP, G	None required	Analyze immediately
21. Color	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours.
23-24. Cyanide, total or available (or CATC) <b>and free</b>	P, FP, G	Cool, < 6°C <sup>18</sup> , NaOH to pH>10 <sup>5,6</sup> , reducing agent <b>if oxidizer present</b>	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P	HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze immediately within 15 minutes
31, 43. Kjeldahl and organic nitrogen	P, FP, G	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
<b>Table IB-Metals<sup>7</sup></b>			
18. Chromium VI	P, FP, G	Cool, < 6°C <sup>18</sup> , pH = 9.3-9.7 <sup>20</sup>	24 hours 28 days
35. Mercury (CVAA)	P, FP, G	HNO <sub>3</sub> to pH<2	28 days
35. Mercury (CVAFS)	FP, G; and FP-lined cap <sup>17</sup>	5 mL/L 12N HCl or 5mL/L BrCl <sup>17</sup>	90 days <sup>17</sup>
3,5-8,12,13,19,20,22,26,29,30,32-34,36,37,45,47,51,52,58-60,62,63,70-	P, FP, G	HNO <sub>3</sub> to pH<2, or at least 24 hours prior to analysis <sup>19</sup>	6 months



72,74,75. Metals, except boron, chromium VI and mercury

38. Nitrate	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
40. Nitrite	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
41. Oil and grease	G	Cool to < 6°C <sup>18</sup> , HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool to < 6°C <sup>18</sup> , HCl or H <sub>2</sub> SO <sub>4</sub> or H <sub>3</sub> PO <sub>4</sub> , to pH<2	28 days
44. Orthophosphate	P, FP, G	Filter immediately, Cool, < 6°C	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately within 15 minutes
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G only	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
49. Phosphorus (elemental)	G	Cool, < 6°C <sup>18</sup>	48 hours
50. Phosphorus, total	P, FP, G	Cool, < 6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
53. Residue, total	P, FP, G	Cool, < 6°C <sup>18</sup>	7 days
54. Residue, Filterable	P, FP, G	Cool, < 6°C <sup>18</sup>	7 days
55. Residue, Nonfilterable(TSS)	P, FP, G	Cool, < 6°C <sup>18</sup>	7 days
56. Residue, Settleable	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
61. Silica	P or Quartz	Cool, < 6°C <sup>18</sup>	28 days
64. Specific conductance	P, FP, G	Cool, < 6°C <sup>18</sup>	28 days
65. Sulfate	P, FP, G	Cool, < 6°C <sup>18</sup>	28 days
66. Sulfide	P, FP, G	Cool, < 6°C <sup>18</sup> add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, FP, G	None required	Analyze immediately within 15 minutes
68. Surfactants	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, < 6°C <sup>18</sup>	48 hours
<b>Table IC-Organic Tests.<sup>8</sup></b>			
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 <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP -lined septum	Cool, < 6°C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH2 <sup>9</sup>	14 days <sup>9</sup>
3, 4, Acrolein and acrylonitrile	G, FP -lined septum	Cool, < 6°C <sup>18</sup> , 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>	14 days <sup>10</sup>
23,30,44,49,53,77,80,81,98,100,112. Phenols <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , 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>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction <sup>13</sup>
14, 17, 48, 50-52. Phthalate esters <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup>	7 days until extraction; 40 days after extraction
82-84. Nitrosamines <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , store in dark, 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
88-94. PCBs <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup>	7 days until extraction; 40 days after extraction
54, 55, 75, 79. Nitroaromatics and isophorone <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> store in dark	7 days until extraction; 40 days after extraction
1,2,5,8-12,32,33, 58,59,74,78,99,101. Polynuclear aromatic hydrocarbons <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , 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
15, 16, 21, 31, 87. Haloethers <sup>11</sup>	G, FP -lined cap	Cool, < 6°C <sup>18</sup> , 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
29, 35-37, 63-65, 73,107. Chlorinated	G, FP-lined cap	Cool, < 6°C <sup>18</sup>	7 days until extraction; 40

hydrocarbons <sup>11</sup> 60-62,66-72,85,86,95-97,102,103. CDDs/CDFs <sup>11</sup>			days after extraction
Aqueous, field and lab preservation	G, FP-lined cap	Cool, < 6°C <sup>18</sup> , 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	G, FP-lined cap	Cool, < 6°C <sup>18</sup>	7 days
Tissue: field preservation	G, FP-lined cap	Cool, < 6°C <sup>18</sup>	24 hours
Solids, mixed phase and tissue: lab preservation	G, FP-lined cap	Freeze, < -10°C	1 year
114 -118. Alkylated phenols	G	Cool, < 6 °C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days until extraction, 40 days after extraction
119. Adsorbable Organic Halides (AOX)	G	Cool, < 6 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> HNO <sub>3</sub> to pH < 2	Hold at least 3 days, but not more than 6 months
120. Chlorinated Phenolics		Cool, < 6 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> H <sub>2</sub> SO <sub>4</sub> to pH < 2	30 days until acetylation, 30 days after acetylation
<b>Table ID-Pesticides Tests:</b>			
1-70. Pesticides <sup>11</sup>	G, FP-lined cap	Cool, < 6°C <sup>18</sup> , pH 5-9 <sup>15</sup>	7 days until extraction; 40 days after extraction.
<b>Table IE-Radiological Tests:</b>			
1-5. Alpha, beta and radium	P, FP, G	HNO <sub>3</sub> to pH<2	6 months.
<b>Table IH-Bacteria Tests:</b>			
1 E. coli	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22,23</sup>
7 Enterocci	PA, G	Cool, <10°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours <sup>22</sup>
<b>Table IH--Protozoa Tests:</b>			
8 Cryptosporidium	LDPE; field filtration	1-10 C	96 hours <sup>21</sup>
9 Giardia	LDPE; field filtration	1-10 C	96 hours <sup>21</sup>

#### Table II Notes

1 P is polyethylene; FP is fluoropolymer (polytetrafluoroethylene (PTFE), 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), 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 with in 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 Method 1664A (and the procedures at 141.34.

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, 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 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 of 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 analyses 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. 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), 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 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). Also, Section 9060A of *Standard Methods for the Examination of Water and Wastewater* (20<sup>th</sup> and 21<sup>st</sup> 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 Samples should be filtered immediately on site before adding preservative for dissolved metals, except for samples collected for trace-level mercury (see footnote 17). 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), 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 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.

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% 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.

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% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

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, 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 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 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. 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  $<6^{\circ}\text{C}$ , 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 " $<^{\circ}\text{C}$ " is used in place of the " $4^{\circ}\text{C}$ " and " $< 4^{\circ}\text{C}$ " 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  $6^{\circ}\text{C}$  may not be used to meet the  $<6^{\circ}\text{C}$  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 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).**

[Editor's Note: Sections 136.4 and 136.5 were completely rewritten and are not shown as change.]

### **136.4 Application for and Approval of Alternate Test Procedures for Nationwide Use**

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure (ATP) Program Coordinator (National Coordinator), Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW, Washington, DC 20460. Any application for an alternate test procedure (ATP) under this paragraph 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 test procedure is being requested.
- (3) Provide a detailed description of the proposed alternate test procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure for the analysis of the pollutant(s) or parameter(s) in wastewater discharges from representative and specified industrial or other categories.
- (4) Provide comparability data for the performance of the proposed alternative test procedure compared to the performance of the reference method.

(b) The National Coordinator may request additional information and analyses from the applicant in order to determine whether the alternate test procedure satisfies the applicable requirements of this Part.

(c) Approval for nationwide use

- (1) After a review of the application and any additional analyses requested from the applicant, the National Coordinator will notify the applicant, in writing, of acceptance or rejection of the alternate test procedure for

nationwide use in CWA programs. If the application is not approved, the National Coordinator will specify what additional information might lead to a reconsideration of the application, and notify the Regional Alternate Test Procedure Coordinators of such rejection. Based on the National Coordinator's rejection of a proposed alternate test procedure and an assessment of any approvals for limited uses for the unapproved method, the Regional ATP Coordinator or permitting authority may decide to withdraw approval of the method for limited use in the Region.

(2) Where the National Coordinator approved an applicant's request for nationwide use of an alternate test procedure, the National Coordinator will notify the applicant that the National Coordinator will recommend rulemaking to approve the alternate test procedure. The National Coordinator will notify the Regional ATP Coordinator or permitting authorities that they may consider approval of this alternate test procedure for limited use in their Regions based on the information and data provided in the applicant's application. The Regional ATP Coordinator or permitting authority will grant approval on a case-by-case basis prior to use of the alternate test procedure for compliance analyses until the alternate test procedure is approved by publication in a final rule in the Federal Register.

(3) EPA will propose to amend Part 136 to include the alternate test procedure in 136.3. EPA shall make available for review all the factual bases for its proposal, including any performance data submitted by the applicant and any available EPA analysis of those data.

(4) Following public comment, EPA shall publish in the Federal Register a final decision on whether to amend Part 136 to include the alternate test procedure as an approved analytical method.

(5) Whenever the National Coordinator has approved an applicant's request for nationwide use of an alternate test procedure, any person may request an approval of the method for limited use under 136.5 from the EPA Region.

### **136.5 Approval of Alternate Test Procedures for Limited Use**

(a) Any person may request the Regional Alternate Test Procedure (ATP) Coordinator or permitting authority to approve the use of an alternate test procedure in the Region.

(b) When the request for the use of an alternate test procedure concerns use in a State with an NPDES permit program approved pursuant to section 402 of the Act, the requestor shall first submit an application for limited use to the Director of the State agency having responsibility for issuance of NPDES permits within such State (*i.e.*, permitting authority). The Director will forward the application to the Regional ATP Coordinator or permitting authority with a recommendation for or against approval.

(c) Any application for approval of an alternate test procedure for limited use may be made by letter, email or by hard copy. The application shall include the following:

(1) Provide the name and address of the applicant and the applicable ID number of the existing or pending permit and issuing agency for which use of the alternate test procedure is requested, and the discharge serial number.

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

(3) Provide justification for using testing procedures other than those specified in Tables IA through IH, or in the NPDES permit.

(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.

(5) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the reference method.

(d) Approval for limited use

(1) After a review of the application by the Alternate Test Procedure Regional ATP Coordinator or permitting authority, the Regional ATP Coordinator or permitting authority notifies the applicant and the appropriate State agency of approval or rejection of the use of the alternate test procedure. The approval may be restricted to use only with respect to a specific discharge or facility (and its laboratory) or, at the discretion of the Regional ATP Coordinator or permitting authority, to all discharger or facilities (and their associated laboratories) specified in the approval for the Region. If the application is not approved, the Regional ATP Coordinator or permitting

authority shall specify what additional information might lead to a reconsideration of the application.

(2) The Regional ATP Coordinator or permitting authority will forward a copy of every approval and rejection notification to the National Alternate Test Procedure Coordinator.

### **136.6 Method Modifications and Analytical Requirements.**

(a) *Definitions of terms used in this Section.*

(1) Analyst means the person or laboratory using a test procedure (analytical method).

(2) Chemistry of the Method means the reagents and reactions used in a test procedure that allow determination of the analyte(s) of interest in an environmental sample.

(3) Determinative Technique means the way in which an analyte is identified and quantified (e.g., colorimetry, mass spectrometry).

(4) Equivalent Performance means that the modified method produces results that meet the QC acceptance criteria of the approved method at this part.

(5) Method-defined Analyte means an analyte defined solely by the method used to determine the analyte. Such an analyte may be a physical parameter, a parameter that is not a specific chemical, or a parameter that may be comprised of a number of substances. Examples of such analytes include temperature, oil and grease, total suspended solids, total phenolics, turbidity, chemical oxygen demand, and biochemical oxygen demand.

(6) QC means "quality control."

(b) *Method Modifications.*

(1) If the underlying chemistry and determinative technique in a modified method are essentially the same as an approved Part 136 method, then the modified method is an equivalent and acceptable alternative to the approved method provided the requirements of this section are met. However, those who develop or use a modification to an approved (Part 136) method must document that the performance of the modified method, in the matrix to which the modified method will be applied, is equivalent to the performance of the approved method. If such a demonstration cannot be made and documented, then the modified method is not an acceptable alternative to the approved method. Supporting documentation must, if applicable, include the routine initial demonstration of capability and ongoing QC including determination of precision and accuracy, detection limits, and matrix spike recoveries. Initial demonstration of capability typically includes analysis of four replicates of a mid-level standard and a method detection limit study. Ongoing quality control typically includes method blanks, mid-level laboratory control samples, and matrix spikes (QC is as specified in the method). The method is considered equivalent if the quality control requirements in the reference method are achieved. The method user's Standard Operating Procedure (SOP) must clearly document the modifications made to the reference method. Examples of allowed method modifications are listed below. The user must notify their permitting authority of the intent to use a modified method. Such notification should be of the form "Method xxx has been modified within the flexibility allowed in 40 CFR Part 136.6." The user may indicate the specific section of 136.6 allowing the method modification. However, specific details of the modification need not be provided, but must be documented in the Standard Operating Procedure (SOP). If the method user is uncertain whether a method modification is allowed, the Regional ATP Coordinator or permitting authority should be contacted for approval prior to implementing the modification. The method user should also complete necessary performance checks to verify that acceptable performance is achieved with the method modification prior to analyses of compliance samples.

(2) Requirements. The modified method must be sufficiently sensitive and meet or exceed performance of the approved method(s) for the analyte(s) of interest, as documented by meeting the initial and ongoing quality control requirements in



the method.

(i) Requirements for establishing equivalent performance. If the approved method contains QC tests and QC acceptance criteria, the modified method must use these QC tests and the modified method must meet the QC acceptance criteria with the following conditions:

- (A) The analyst may only rely on QC tests and QC acceptance criteria in a method if it includes wastewater matrix QC tests and QC acceptance criteria (e.g., matrix spikes) and both initial (start-up) and ongoing QC tests and QC acceptance criteria.
- (B) If the approved method does not contain QC tests and QC acceptance criteria or if the QC tests and QC acceptance criteria in the method do not meet the requirements of this section, then the analyst must employ QC tests published in the “equivalent” of a Part 136 method that has such QC, or the essential QC requirements specified at 136.7, as applicable. If the approved method is from a compendium or VCSB and the QA/QC requirements are published in other parts of that organization’s compendium rather than within the Part 136 method then that part of the organization’s compendium must be used for the QC tests.
- (C) In addition, the analyst must perform ongoing QC tests, including assessment of performance of the modified method on the sample matrix (e.g., analysis of a matrix spike/matrix spike duplicate pair for every twenty samples), and analysis of an ongoing precision and recovery sample (e.g., laboratory fortified blank or blank spike) and a blank with each batch of 20 or fewer samples.
- (D) If the performance of the modified method in the wastewater matrix or reagent water does not meet or exceed the QC acceptance criteria, the method modification may not be used.

(ii) Requirements for documentation. The modified method must be documented in a method write-up or an addendum that describes the modification(s) to the approved method prior to the use of the method for compliance purposes. The write-up or addendum must include a reference number (e.g., method number), revision number, and revision date so that it may be referenced accurately. In addition, the organization that uses the modified method must document the results of QC tests and keep these records, along with a copy of the method write-up or addendum, for review by an auditor.

(3) Restrictions. An analyst may not modify an approved Clean Water Act analytical method for a method-defined analyte. In addition, an analyst may not modify an approved method if the modification would result in measurement of a different form or species of an analyte. Changes in method procedures are not allowed if such changes would alter the defined chemistry (i.e., method principle) of the unmodified method. For example, phenol method 420.1 or 420.4 defines phenolics as ferric iron oxidized compounds that react with 4-aminoantipyrine (4-AAP) at pH 10 after being distilled from acid solution. Because total phenolics represents a group of compounds that all react at different efficiencies with 4-AAP, changing test conditions likely would change the behavior of these different phenolic compounds. An analyst may not modify any sample collection, preservation, or holding time requirements of an approved method. Such modifications to sample collection, preservation, and holding time requirements do not fall within the scope of the flexibility allowed at Part 136.6. Method flexibility refers to modifications of the analytical procedures used for identification and measurement of the analyte only and does not apply to sample collection, preservation, or holding time procedures, which may only be modified as specified in 136.3 (e).

(4) *Allowable Changes*. Except as under *Restrictions* of this section, an analyst may modify an approved test procedure (analytical method) provided that the chemistry of the method or the determinative technique is not changed, and provided that the requirements of this section are met. If equal or better performance can be obtained with an alternative reagent, then it is allowed. A laboratory wishing to use these modifications must demonstrate acceptable method performance by performing and documenting all applicable initial demonstration of capability and ongoing QC tests and meeting all applicable QC acceptance criteria as described in Part 136.7. Some examples of the allowed types of changes, provided the requirements of this section are met include:

- (A) Changes between manual method, flow analyzer, and discrete instrumentation.
- (B) Changes in chromatographic columns or temperature programs.
- (C) Changes between automated and manual sample preparation, such as digestions, distillations, and extractions;

in-line sample preparation is an acceptable form of automated sample preparation for CWA methods.

- (D) In general, ICP-MS is a sensitive and selective detector for metal analysis; however isobaric interference can cause problems for quantitative determination, as well as identification based on the isotope pattern. Interference reduction technologies, such as collision cells or reaction cells, are designed to reduce the effect of spectroscopic interferences that may bias results for the element of interest. The use of interference reduction technologies is allowed, provided the method performance specifications relevant to ICP-MS measurements are met.
- (E) The use of EPA Method 200.2 or the sample preparation steps from EPA Method 1638, including the use of closed-vessel digestion, is allowed for EPA Method 200.8, provided the method performance specifications relevant to the ICP-MS are met.
- (F) Changes in pH adjustment reagents. Changes in compounds used to adjust pH are acceptable as long as they do not produce interference. For example, using a different acid to adjust pH in colorimetric methods.
- (G) Changes in buffer reagents are acceptable provided that the changes do not produce interferences.
- (H) Changes in the order of reagent addition are acceptable provided that the change does not alter the chemistry and does not produce an interference. For example, using the same reagents, but adding them in different order, or preparing them in combined or separate solutions (so they can be added separately), is allowed, provided reagent stability or method performance is equivalent or improved.
- Changes in calibration range (provided that the modified range covers any relevant regulatory limit and the method performance specifications for calibration are met).
- (J) Changes in calibration model. Linear calibration models do not adequately fit calibration data with one or two inflection points. For example, vendor-supplied data acquisition and processing software on some instruments may provide quadratic fitting functions to handle such situations. If the calibration data for a particular analytical method routinely display quadratic character, using quadratic fitting functions may be acceptable. In such cases, the minimum number of calibrators for second order fits should be six, and in no case should concentrations be extrapolated for instrument responses that exceed that of the most concentrated calibrator. Examples of methods with nonlinear calibration functions include chloride by SM4500-Cl-E-1997, hardness by EPA Method 130.1, cyanide by ASTM D6888 or OIA1677, Kjeldahl nitrogen by PAI-DK03, and anions by EPA Method 300.0. As an alternative to using the average response factor, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\% \text{ RSE} = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[ \frac{x'_i - x_i}{x_i} \right]^2}{(n-p)}}$$

where:

$x'_i$  = Calculated concentration at level  $i$

$x_i$  = Actual concentration of the calibration level  $i$

$n$  = Number of calibration points

$p$  = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)

- Using the RSE as a metric has the added advantage of allowing the same numerical standard to be applied to the calibration model, regardless of the form of the model. Thus, if a method states that the RSD should be  $\leq 20\%$  for the traditional linear model through the origin, then the RSE acceptance limit can remain  $\leq 20\%$  as well. Similarly, if a method provides an RSD acceptance limit of  $\leq 15\%$ , then that same figure can be used as the acceptance limit for the RSE. The RSE may be used as an alternative to correlation coefficients and coefficients of determination for evaluating calibration curves for any of the methods at Part 136. If the method includes a numerical criterion for the RSD, then the same numerical value is used for the RSE. Some older methods do not include any criterion for the calibration curve – for these methods, if RSE is used the value should be  $\leq 20\%$ . Note that the use of the RSE is included as an alternative to the use of the correlation coefficient as a measure of the



suitability of a calibration curve. It is not necessary to evaluate both the RSE and the correlation coefficient.

- (K) Changes in equipment such as equipment from a vendor different from the one specified in the method.
- (L) The use of micro or midi distillation apparatus in place of macro distillation apparatus.
- (M) The use of prepackaged reagents.
- (N) The use of digital titrators and methods where the underlying chemistry used for the determination is similar to that used in the approved method.
- (O) Use of selected ion monitoring (SIM) mode for analytes that cannot be effectively analyzed in full-scan mode and reach the required sensitivity. False positives are more of a concern when using SIM analysis, so at a minimum, one quantitation and two qualifying ions must be monitored for each analyte (unless fewer than three ions with intensity greater than 15% of the base peak are available). The ratio of each of the two qualifying ions to the quantitation ion must be evaluated and should agree with the ratio observed in an authentic standard within  $\pm 20$  percent. Analyst judgment must be applied to the evaluation of ion ratios because the ratios can be affected by co-eluting compounds present in the sample matrix. The signal-to-noise ratio of the least sensitive ion should be at least 3:1. Retention time in the sample should match within 0.05 minute of an authentic standard analyzed under identical conditions. Matrix interferences can cause minor shifts in retention time and may be evident as shifts in the retention times of the internal standards. The total scan time should be such that a minimum of eight scans are obtained per chromatographic peak.
- (P) Changes are allowed in purge-and-trap sample volumes or operating conditions. Some examples are:
  - Changes in purge time and purge-gas flow rate. A change in purge time and purge-gas flow rate is allowed provided that sufficient total purge volume is used to achieve the required minimum detectable concentration and calibration range for all compounds. In general, a purge rate in the range 20 - 200 mL/min and a total purge volume in the range 240 - 880 mL are recommended.
  - Use of nitrogen or helium as a purge gas, provided that the required sensitivities for all compounds are met.
  - Sample temperature during the purge state. Gentle heating of the sample during purging (e.g., 40 °C) increases purging efficiency of hydrophilic compounds and may improve sample-to-sample repeatability because all samples are purged under precisely the same conditions.
  - Trap sorbent. Any trap design is acceptable, provided that the data acquired meet all QC criteria.
  - Changes to the desorb time. Shortening the desorb time (e.g., from 4 minutes to 1 minute) may not affect compound recoveries, and can shorten overall cycle time and significantly reduce the amount of water introduced to the analytical system, thus improving the precision of analysis, especially for water-soluble analytes. A desorb time of four minutes is recommended, however a shorter desorb time may be used, provided that all QC specifications in the method are met.
  - Use of water management techniques is allowed. Water is always collected on the trap along with the analytes and is a significant interference for analytical systems (GC and GC/MS). Modern water management techniques (e.g., dry purge or condensation points) can remove moisture from the sample stream and improve analytical performance.
- (Q) The following modifications are allowable when performing EPA Method 625: The base/neutral and acid fractions may be added together and analyzed as one extract, provided that the analytes can be reliably identified and quantified in the combined extracts; the pH extraction sequence may be reversed to better separate acid and neutral components; neutral components may be extracted with either acid or base components; a smaller sample volume may be used to minimize matrix interferences provided matrix interferences are demonstrated and documented; alternative surrogate and internal standard concentrations other than those specified in the method are acceptable, provided that method performance is not degraded; an alternative concentration range may be used for the calibration other than the range specified in the method; the solvent for the calibration standards may be changed to match the solvent of the final sample extract.
- (R) If the characteristics of a wastewater matrix prevent efficient recovery of organic pollutants and prevent the method from meeting QC requirements, the analyst may attempt to resolve the issue by adding salts to the sample, provided that such salts do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such salts), and that

all requirements of paragraph (b)(2) of this section are met. Samples having residual chlorine or other halogen must be dechlorinated prior to the addition of such salts.

- (S) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevent the analyst from meeting QC requirements, the analyst may attempt to resolve the issue by adding an inert surfactant that does not affect the chemistry of the method, such as Brij-35 or sodium dodecyl sulfate (SDS), provided that such surfactant does not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such surfactant) and that all requirements of paragraph (b)(1) and (b)(2) of this section are met. Samples having residual chlorine or other halogen must be dechlorinated prior to the addition of such surfactant.
- (T) The use of gas diffusion (using pH change to convert the analyte to gaseous form and/or heat to separate an analyte contained in steam from the sample matrix) across a hydrophobic semi-permeable membrane to separate the analyte of interest from the sample matrix may be used in place of manual or automated distillation in methods for analysis such as ammonia, total cyanide, total Kjeldahl nitrogen, and total phenols. These procedures do not replace the digestion procedures specified in the approved methods and must be used in conjunction with those procedures.
- (U) Changes in equipment operating parameters such as the monitoring wavelength of a colorimeter or the reaction time and temperature as needed to achieve the chemical reactions defined in the unmodified CWA method. For example, molybdenum blue phosphate methods have two absorbance maxima, one at about 660 nm and another at about 880 nm. The former is about 2.5 times less sensitive than the latter. Wavelength choice provides a cost-effective, dilution-free means to increase sensitivity of molybdenum blue phosphate methods.
- (V) Interchange of oxidants, such as the use of titanium oxide in UV-assisted automated digestion of TOC and total phosphorus, as long as complete oxidation can be demonstrated.
- (W) Use of an axially viewed torch with Method 200.7.

[Editor's Note: Sections 136.7 is a completely new section and is not shown as a change.]

### 136.7 Quality Assurance and Quality Control

The permittee/laboratory shall use suitable QA/QC procedures when conducting compliance analyses with any Part 136 chemical method or an alternative method specified by the permitting authority. These QA/QC procedures are generally included in the analytical method or may be part of the methods compendium for approved Part 136 methods from a consensus organization. For example, Standard Methods contains QA/QC procedures in the Part 1000 section of the Standard Methods Compendium. The permittee/laboratory shall follow these QA/QC procedures, as described in the method or methods compendium.

If the method lacks QA/QC procedures, the permittee/laboratory has the following options to comply with the QA/QC requirements:

- a. Refer to and follow the QA/QC published in the "equivalent" EPA method for that parameter that has such QA/QC procedures;
- b. Refer to the appropriate QA/QC section(s) of an approved Part 136 method from a consensus organization compendium;
- c. Incorporate the following twelve quality control elements, where applicable, into the laboratory's documented standard operating procedure (SOP) for performing compliance analyses when using an approved Part 136 method when the method lacks such QA/QC procedures. One or more of the twelve QC elements may not apply to a given method and may be omitted if a written rationale is provided indicating why the element(s) is/are inappropriate for a specific method.
  - (1) Demonstration of Capability (DOC);
  - (2) Method Detection Limit (MDL);
  - (3) Laboratory reagent blank (LRB), also referred to as method blank (MB);

- (4) Laboratory fortified blank (LFB), also referred to as a spiked blank, or laboratory control sample (LCS);
- (5) Matrix spike (MS) and matrix spike duplicate (MSD), or laboratory fortified matrix (LFM) and LFM duplicate, may be used for suspected matrix interference problems to assess precision;
- (6) Internal standards (for GC/MS analyses), surrogate standards (for organic analysis) or tracers (for radiochemistry);
- (7) Calibration (initial and continuing), also referred to as initial calibration verification (ICV) and continuing calibration verification (CCV);
- (8) Control charts (or other trend analyses of quality control results);
- (9) Corrective action (root cause analysis);
- (10) QC acceptance criteria;
- (11) Definitions of preparation and analytical batches that may drive QC frequencies; and
- (12) Minimum frequency for conducting all QC elements.

These twelve quality control elements must be clearly documented in the written standard operating procedure for each analytical method not containing QA/QC procedures, where applicable.

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