SUBCHAPTER D—WATER PROGRAMS (CONTINUED)

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

Sec.

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APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95–217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251, *et seq.*) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

§ 136.1 Applicability.

(a) The procedures prescribed herein shall, except as noted in §136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(1) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40, and,

(2) Reports required to be submitted by dischargers under the NPDES established by parts 124 and 125 of this chapter, and,

(3) Certifications issued by States pursuant to section 401 of the CWA, as amended.

(b) The procedure prescribed herein and in part 503 of title 40 shall be used to perform the measurements required for an application submitted to the Administrator or to a State for a sewage sludge permit under section 405(f) of the Clean Water Act and for recordkeeping and reporting requirements under part 503 of title 40.

[72 FR 14224, Mar. 26, 2007]

§ 136.2 Definitions.

As used in this part, the term:

(a) *Act* means the Clean Water Act of 1977, Pub. L. 95–217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251 *et seq.*) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *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 set forth at appendix B of this part.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 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 IH. In the event

of a conflict between the reporting requirements of 40 CFR Parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of 40 CFR 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 IH. The incorporation by reference of these documents, as specified in paragraph (b) of this section, was approved by 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 in paragraph (b) of 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*. 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 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, ID, IE, IF, IG, and IH or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances paragraph (c) of this section, § 136.5(a) through (d) or 40 CFR 401.13, 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[1]	EPA	Standard methods 18th, 19th, 20th ed.	Standard methods on-line	AOAC, ASTM, USGS	Other
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[3] ..... 1680[12,14] ..... 1681[12,19]	9221 C E	9221 C E–99.		
	Membrane filter (MF)[2], single step.	p. 124[3]	9222 D	9222 D–97	B–0050–85[5].	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132[3]	9221 C E	9221 C E–99.		
	MF[2], single step	p. 124[3]	9222 D	9222 D–97.		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B	9221 B–99.		
	MF[2], single step or two step	p. 108[3]	9222 B	9222 B–97	B–0025–8[5].	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B	9221 B–99.		
	MF[2] with enrichment	p. 111[3]	9222 (B+B.5c)	9222 (B+B.5c)−97.		
5. *E. coli*, number per 100 mL[20].	MPN[7,9,15] multiple tube/multiple well.	.....	9223 B[13]	9223 B–97[13]	991.15[11]	Colilert®[13,17] Colilert-18®[13,16,17]
	MF[2,6,7,8,9] single step	1603[21]	.....	.....	.....	mColiBlue-24®[18]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube 3 dilution, ...	p. 139[3]	9230 B	9230 B–93.		
	MF[2], or	p. 136[3]	9230 C	9230 C–93	B–0055–85[5].	
	Plate count	p. 143[3].				
7. Enterococci, number per 100 mL[20].	MPN[7,9], multiple tube/multiple well.	.....	.....	.....	D6503–99[10]	Enterolert®[13,23]
	MF[2,6,7,8,9] single step	1600[24].				
8. Salmonella, number per gram dry weight[12].	MPN multiple tube	1682[22].				
Aquatic Toxicity:						
9. Toxicity, acute, fresh water organisms, LC_{50}, percent effluent.	*Ceriodaphnia dubia* acute	2002.0[25].				
	Daphnia puplex and *Daphnia magna* acute.	2021.0[25].				
	Fathead Minnow, *Pimephales promelas*, and Bannerfin *shiner*, *Cyprinella leedsi*, acute.	2000.0[25].				
	Rainbow Trout, *Oncorhynchus mykiss*, and brook trout, *Salvelinus fontinalis*, acute.	2019.0[25].				

TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE—Continued

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th ed.	Standard methods on-line	AOAC, ASTM, USGS	Other
10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC_{50}, percent effluent.	Mysid, *Mysidopsis bahia*, acute	2007.0 [25].				
	Sheepshead Minnow, *Cyprinodon* variegatus, acute.	2004.0 [25].				
	Silverside, *Menidia beryllina*, *Menidia menidia*, and *Menidia peninsulae*, acute.	2006.0 [25].				
11. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fathead minnow, *Pimephales promelas*, larval survival and growth.	1000.0 [26].				
	Fathead minnow, *Pimephales promelas*, embryo-larval survival and teratogenicity.	1001.0 [26].				
	Daphnia, *Ceriodaphnia dubia*, survival and reproduction.	1002.0 [26].				
	Green alga, *Selenastrum capricornutum*, growth.	1003.0 [26].				
12. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC_{25}, percent effluent.	Sheepshead minnow, *Cyprinodon variegatus*, larval survival and growth.	1004.0 [27].				
	Sheepshed minnow, *Cyprinodon variegatus*, embryo-larval survival and teratogenicity.	1005.0 [27].				
	Inland silverside, *Menidia beryllina*, larval survival and growth.	1006.0 [27].				
	Mysid, *Mysidopsis bahia*, survival, growth, and fecundity.	1007.0 [27].				
	Sea urchin, *Arbacia punctulata*, fertilization.	1008.0 [27].				

[1] The method must be specified when results are reported.
[2] A 0.45 μm 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, OH, EPA/600/8–78/017.
[4] [Reserved]

[5] 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 the Interior, Reston, VA.

[6] 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.

[7] 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.

[8] When the MF method has been used previously to test waters with high turbidity, large numbers 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.

[9] 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.

[10] ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[11] AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877–2417.

[12] Recommended for enumeration of target organism in sewage sludge.

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

[14] USEPA. July 2006. 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–06–012.

[15] 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® Quanti-Tray® 2000, and the MPN calculated from the table provided by the manufacturer.

[16] 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.

[17] Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

[18] A description of the mColiBlue24® test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

[19] USEPA. July 2006. 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.

[20] Recommended for enumeration of target organism in wastewater effluent.

[21] USEPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–011.

[22] USEPA. July 2006. 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.

[23] A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

[24] USEPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–009.

[25] 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.

[26] 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.

[27] 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.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
1. Acidity, as $CaCO_3$, mg/L.	Electrometric endpoint or phenolphthalein endpoint.	.....	2310 B(4a)	2310 B(4a)	2310 B(4a)–97 ...	D1067–92, 02	I–1020–85 [2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
2. Alkalinity, as $CaCO_3$, mg/L.	Electrometric or Colorimetric titration to pH 4.5, manual, or	.....	2320 B	2320 B	2320 B–97	D1067–92, 02	973.43 [3], I–1030–85 [2]
	automatic	310.2 (Rev. 1974) [1].	.....	.....	.....	.....	I–2030–85 [2]
3. Aluminum—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 D	.....	3111 D–99	.....	I–3051–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–9750
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14[3]
	Direct Current Plasma (DCP) [36].	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (Eriochrome cyanine R).	.....	3500–Al D	3500–Al B	3500–Al B–01.		
4. Ammonia (as N), mg/L	Manual, distillation (at pH 9.5) [6] followed by:	350.1, Rev. 2.0 (1993).	4500–NH B_3	4500–NH_3 B	4500–NH_3 B–97	.....	973.49 [3]
	Nesslerization	.....	4500–NH_3 C (18th only).	.....	.....	D1426–98, 03 (A).	973.49 [3], I–3520–85 [2]
	Titration	.....	4500–NH_3 C (19th) and 4500–NH_3 E (18th).	4500–NH_3 C	4500–NH_3 C–97.		
	Electrode	.....	4500–NH_3 D or E (19th) and 4500–NH_3 F or G (18th).	4500–NH_3 D or E.	4500–NH_3 D or E–97.	D1426–98, 03 (B).	

	Automated phenate, or.	350.1 [60], Rev. 2.0 (1993).	4500–NH_3 G (19th) and 4500–NH_3 H (18th).	4500–NH_3 G ...	4500–NH_3 G–97	.....	I–4523–85 [2]
	Automated electrode	.....	.....	.....	.....	.....	See footnote 7
	Ion Chromatography	.....	.....	.....	.....	D6919–03.	
5. Antimony—Total, [4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 B	.....	3111 B–99.		
	AA furnace	.....	3113 B	.....	3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
6. Arsenic—Total, [4] mg/L	Digestion [4] followed by.						
	AA gaseous hydride	.....	3114 B 4.d	.....	3114 B 4.d–97 ...	D2972–97, 03 (B).	I–3062–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D2972–97, 03 (C).	I–4063–98 [49]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	Colorimetric (SDDC)	.....	3500–As C	3500–As B	3500–As B–97 ...	D2972–97, 03 (A).	I–3060–85
7. Barium—Total, [4] mg/L ..	Digestion [4] followed by;						
	AA direct aspiration [36].	.....	3111 D	.....	3111 D–99	.....	I–3084–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D4382–95, 02.	
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP [36]	.....	.....	.....	.....	.....	See footnote [34]
8. Beryllium—Total, [4] mg/L	Digestion [4] followed by:						

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
	AA direct aspiration	.....	3111 D	.....	3111 D–99	D3645–93 (88), 03 (A).	I–3095–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D3645–93 (88), 03 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP, or	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (aluminon).	.....	3500–Be D.				
9. Biochemical oxygen demand (BOD_5), mg/L.	Dissolved Oxygen Depletion.	.....	5210 B	5210 B	5210 B–01	.....	973.44,[3] p. 17.[9], I–1578–78 [8]
10. Boron—Total,[37] mg/L	Colorimetric (curcumin).	.....	4500–B B	4500–B B	4500–B B–00	.....	I–3112–85 [2]
	ICP/AES, or	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B99	.....	I–4471–97 [50]
	DCP	.....	.....	.....	.....	D4190–94, 99	See footnote 34
11. Bromide, mg/L	Titrimetric	.....	.....	.....	.....	D1246–95, 99 (C).	p. S44.[10]
	.....	.....	.....	.....	.....	.....	I–1125–85 [2]
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30 [3]
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev. 2 [54]
12. Cadmium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 B or C	.....	3111 B or C–99	D3557–95, 02 (A or B).	974.27,[3] p. 37.[9], I–3135–85 [2] or I–3136–85 [2]

	AA furnace	.....	3113 B	.....	3113 B–99	D3557–95, 02 (D).	I–4138–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–1472–85[2] or I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14[3]
	DCP[36]	.....	.....	.....	.....	D4190–94, 99	See footnote[34]
	Voltametry[11], or	.....	.....	.....	.....	D3557–95, 02 (C).	
13. Calcium—Total,[4] mg/L	Colorimetric (Dithizone).	.....	3500–Cd D.				
	Digestion[4] followed by;						
	AA direct aspiration	.....	3111 B	.....	3111 B–99	D511–93, 03(B)	I–3152–85[2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97[50]
	DCP, or	.....	.....	.....	.....	.....	See footnote[34]
	Titrimetric (EDTA)	.....	3500–Ca D	3500–Ca B	3500–Ca B–97 ...	D511–93, 03(A).	
	Ion Chromatography	.....	.....	.....	.....	D6919–03.	
14. Carbonaceous biochemical oxygen demand ($CBOD_5$), mg/L[12].	Dissolved Oxygen Depletion with nitrification inhibitor.	.....	5210 B	5210 B	5210 B–01.		
15. Chemical oxygen demand (COD), mg/L.	Titrimetric	410.3 (Rev. 1978)[1].	5220 C	5220 C	5220 C–97	D1252–95, 00 (A).	973.46[3], p. 17[9] I–3560–85[2]
	Spectrophotometric, manual or automatic.	410.4, Rev. 2.0 (1993).	5220 D	5220 D	5220 D–97	D1252–95, 00 (B).	See footnotes[13,14], I–3561–85[2]
16. Chloride, mg/L	Titrimetric: (silver nitrate) or.	.....	4500–Cl–B	4500–Cl–B	4500–Cl–B–97 ...	D512–89(99) (B).	I–1183–85[2]
	(Mercuric nitrate)	.....	4500–Cl–C	4500–Cl–C	4500–Cl–C–97 ...	D512–89 (99) (A).	973.51[3], I–1184–85[2]
	Colorimetric: manual or.	.....	.....	.....	.....	.....	I–1187–85[2]
	Automated (Ferricyanide).	.....	4500–Cl–E	4500–Cl–E	4500–Cl–E–97 ...	.....	I–2187–85[2]
	Potentiometric Titration.	.....	4500–Cl–D	4500–Cl–D	4500–Cl–D–97.		
	Ion Selective Electrode.	.....	.....	.....	.....	D512–89(99)(C).	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30 [3]
	CIE/UV						D6508, Rev. 2 [54]
17. Chlorine—Total residual, mg/L; Titrimetric.	Amperometric direct, or.		4500–Cl D	4500–Cl D	4500–Cl D–00	D1253–86 (96), 03.	
	Amperometric direct (low level).		4500–Cl E	4500–Cl E	4500–Cl E–00.		
	Iodometric direct		4500–Cl B	4500–Cl B	4500–Cl B–00.		
	Back titration ether end-point [15] or.		4500–Cl C	4500–Cl C	4500–Cl C–00.		
	DPD–FAS		4500–Cl F	4500–Cl F	4500–Cl F–00.		
	Spectrophotometric, DPD or.		4500–Cl G	4500–Cl G	4500–Cl G–00.		
	Electrode						See footnote [16]
18. Chromium VI dissolved, mg/L.	0.45–micron Filtration followed by:						
	AA chelation-extraction or.		3111 C		3111 C–99		I–1232–85
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr E	3500–Cr C	3500–Cr C–01	D5257–97	993.23
	Colorimetric (Diphenyl-carbazide).		3500–Cr D	3500–Cr B	3500–Cr B–01	D1687–92, 02 (A).	I–1230–85
19. Chromium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration [36].		3111 B		3111 B–99	D1687–92, 02 (B).	974.27 [3], I–3236–85 [2]
	AA chelation-extraction.		3111 C		3111 C–99.		
	AA furnace		3113 B		3113 B–99	D1687–92, 02 (C).	I–3233–93 [46]
	STGFAA	200.9, Rev. 2.2 (1994).					

20. Cobalt—Total,[4] mg/L	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.			
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]	
	DCP,[36] or					D4190–94, 99	See footnote[34]	
	Colorimetric (Diphenyl-carbazide).		3500–Cr D	3500–Cr B	3500–Cr B–01.			
	Digestion[4] followed by:							
	AA direct aspiration		3111 B or C		3111 B or C–99	D3558–94, 03 (A or B).	p. 37[9], I–3239–85[2]	
	AA furnace		3113 B		3113 B–99	D3558–94, 03 (C).	I–4243–89[51]	
	STGFAA	200.9, Rev. 2.2 (1994).						
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]	
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]	
	DCP					D4190–94, 99	See footnote[34]	
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity.	Colorimetric (ADMI), or.		2120 E	2120 E			See footnote[18]	
22. Copper—Total,[4] mg/L	(Platinum cobalt), or Spectrophotometric		2120 B	2120 B	2120 B–01		I–1250–85[2]	
	Digestion[4] followed by:		2120 C	2120 C.				
	AA direct aspiration[36];		3111 B or C		3111 B or C–99	D1688–95, 02 (A or B).	974.27[3] p. 37[9] I–3270–85[2] or I–3271–85[2]	
	AA furnace		3113 B		3113 B–99	D1688–95, 02 (C).	I–4274–89[51]	
	STGFAA	200.9, Rev. 2.2 (1994).						
	ICP/AES[36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97[50]	
	ICP/MS	200.8, Rev. 5.4 (1994).				D5673–03	993.14[3]	
	DCP[36] or					D4190–94, 99	See footnote[34]	
	Colorimetric (Neocuproine) or.		3500–Cu D	3500–Cu B	3500–Cu B–99.			
	(Bicinchoninate)		3500–Cu E	3500–Cu C	3500–Cu C–99		See footnote[19]	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
23. Cyanide—Total, mg/L	Automated Distillation and Colorimetry, or.	.....	.....	.....	.....	.....	Kelada–01 [55]
	Manual distillation with $MgCl_2$ followed by:	335.4, Rev. 1.0 (1993) [57].	4500–CN–C	4500–CN–C	.....	D2036–98(A) ...	10–204–00–1–X [56]
	Titrimetric or	.....	4500–CN–D	4500–CN–D	4500–CN–D–99 ..	.....	p. 22 [9]
	Spectrophotometric, manual or.	.....	4500–CN–E	4500–CN–E	4500–CN–E–99 ..	D2036–98(A) ...	I–3300–85
	Automated [20] or	335.4, Rev. 1.0 (1993) [57].	.....	.....	.....	.....	10–204–00–1–X [56], I–4302–85 [2]
	Ion Selective Electrode.	.....	4500–CN–F	4500–CN–F	4500–CN–F–99 ..	D2036–98(A).	
24. Available Cyanide, mg/L.	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$ followed by Titrimetric or Spectrophotometric.	.....	4500–CN–G	4500–CN–G	4500–CN–G–99	D2036–98(B).	
	Flow injection and ligand exchange, followed by amperometry [61].	.....	.....	.....	.....	D6888–04	OIA–1677 [44]
	Automated Distillation and Colorimetry.	.....	.....	.....	.....	.....	Kelada–01 [55]
25. Fluoride—Total, mg/L	Manual distillation [6] followed by:	.....	4500–F–B	4500–F–B	4500–F–B–97.		
	Electrode, manual or	.....	4500–F–B	4500–F–B	4500–F–C–97	D1179–93, 99 (B).	
	Automated	.....	.....	.....	.....	.....	I–4327–85 [2]
	Colorimetric, (SPADNS) or.	.....	4500–F–D	4500–F–D	4500–F–D–97	D1179–93, 99 (A).	

	Automated complexone.	.....	4500–F–E	4500–F–E	4500–F–E–97.			
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97,03 ...	993.30[3]	
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev. 2[54]	
26. Gold—Total,[4] mg/L	Digestion[4] followed by;	.....	3111 B	.....	3111 B–99.	.....	See footnote[34]	
	AA direct aspiration, or,	.....						
	AA furnace, or	231.2 (Rev. 1978)[1].						
	DCP	.....	.....	.....	.....	.....		
27. Hardness—Total, as $CaCO_3$, mg/L.	Automated colorimetric,.	130.1 (Issued 1971)[1].						
	Titrimetric (EDTA) or	.....	2340 B or C	2340 B or C	2340 B or C–97	D1126–86(92), 02.	973.52B[3], I–1338–85[2]	
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33)..							
28. Hydrogen ion (pH), pH units.	Electrometric measurement or.	.....	4500–H^+ B	4500–H^+ B	4500–H^+ B–00 ..	D1293–84 (90), 99 (A or B).	973.41.[3], I–1586–85[2]	
	Automated electrode	150.2 (Dec. 1982)[1].	.....	.....	.....	.....	See footnote[21], I–2587–85[2]	
29. Iridium—Total,[4] mg/L	Digestion[4] followed by;							
	AA direct aspiration or,	.....	3111 B	.....	3111 B–99.			
	AA furnace	235.2 (Issued 1978)[1].						
30. Iron—Total,[4] mg/L	Digestion[4] followed by;							
	AA direct aspiration[36].	.....	3111 B or C	.....	3111 B or C–99	D1068–96, 03 (A or B).	974.27[3], I–3381–85[2]	
	AA furnace	.....	3113 B	.....	3113 B–99	D1068–96, 03 (C).		
	STGFAA	200.9, Rev. 2.2 (1994).						

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99		I–4471–97 [50]
	DCP [36] or					D4190–94, 99	See footnote [34]
	Colorimetric (Phenanthroline).		3500–Fe D	3500–Fe B	3500–Fe B–97	D1068–96, 03 (D).	See footnote [22]
31. Kjeldahl Nitrogen [5]—Total, (as N), mg/L.	Digestion and distillation followed by: [20]		4500–N_{org} B or C and 4500–NH_3 B.	4500–N_{org} B or C and 4500–NH_3 B.	4500–N_{org} B or C–97 and 4500–NH_3 B–97.	D3590–89, 02 (A).	
	Titration or		4500–NH_3 C (19th) and 4500–NH_3 E (18th).	4500–NH_3 C	4500–NH_3 C–97	D3590–89, 02 (A).	973.48 [3]
	Nesslerization or		4500–NH_3 C (18th Only).			D3590–89, 02 (A).	
	Electrode		4500–NH_3 F or G (18th) and 4500–NH_3 D or E (19th).	4500–NH_3 D or E.	4500–NH_3 D or E–97.		
	Automated phenate colorimetric.	351.1 (Rev. 1978) [1].					I–4551–78 [8]
	Semi-automated block digestor colorimetric.	351.2, Rev. 2.0 (1993).				D3590–89, 02 (B).	I–4515–91 [45]
	Manual or block digestor potentiometric.					D3590–89, 02 (A).	
	Block digester, followed by Auto distillation and Titration, or.						See footnote [39]
	Nesslerization, or						See footnote [40]
	Flow injection gas diffusion.						See footnote [41]
32. Lead—Total,[4] mg/L	Digestion [4] followed by:						

33. Magnesium—Total,[4] mg/L.	AA direct aspiration [36].	.....	3111 B or C	.....	3111 B or C–99	D3559–96, 03 (A or B).	974.27 [3], I–3399–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D3559–96, 03 (D).	I–4403–89 [51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP [36]	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Voltametry [11] or	.....	.....	.....	.....	D3559–96, 03 (C).	
	Colorimetric (Dithizone).	.....	3500–Pb D	3500–Pb B	3500–Pb B–97.		
	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 B	.....	3111 B–99	D511–93, 03(B)	974.27 [3], I–3447–85 [2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	DCP or	.....	.....	.....	.....	.....	See footnote [34]
	Gravimetric	.....	3500–Mg D.				
	Ion Chromatography	.....	.....	.....	.....	D6919–03.	
34. Manganese—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 B	.....	3111 B–99	D858–95, 02 (A or B).	974.27 [3], I–3454–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D858–95, 02 (C).	
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP36, or	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (Persulfate), or. (Periodate)	.....	3500—Mn D ..	3500–Mn B	3500–Mn B–99 ..	.....	920.203 [3]
35. Mercury—Total [4], mg/L.	Cold vapor, manual or.	245.1, Rev. 3.0 (1994).	3112 B	.....	3112 B–99	.....	See footnote [23]
	Automated	245.2 (Issued 1974).		.....		D3223–97, 02	977.22 [3], I–3462–85 [2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
	Cold vapor atomic fluorescence spectrometry (CVAFS).	245.7 Rev. 2.0 (2005) [59].					
	Purge and Trap CVAFS.	1631E [43].					
36. Molybdenum—Total [4], mg/L.	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 D	.....	3111 D–99	.....	I–3490–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	.....	I–3492–96 [47]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
.....	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP	.....	.....	.....	.....	.....	See footnote [34]
37. Nickel—Total,[4] mg/L ..	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 B or C	.....	3111 B or C–99	D1886–90, 94 (98) (A or B).	I–3499–85 [2]
	AA furnace	.....	3113 B	.....	3113 B–99	D1886–90, 94 (98) (C).	I–4503–89 [51]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP [36], or	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (heptoxime).	.....	3500–Ni D (17th Edition).				
38. Nitrate (as N), mg/L ...	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30 [3]
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev. 2 [54]

	Ion Selective Electrode.	.....	4500–NO_3^-D ..	4500–NO_3^-D ..	4500–NO_3^-D–00.		
	Colorimetric (Brucine sulfate), or.	352.1 [1]	.....	.....	.....	.....	973.50 [3], 419D [1, 7], p. 28 [9]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)..	.....	4500–NO_3^-E ...	4500–NO_3^-E ...	4500–$NO3^-$E–00	D3867–99(B).	
39. Nitrate-nitrite (as N), mg/L.	Cadmium reduction, manual or.	.....	4500–NO_3^-E ...	4500–NO_3^-E ...	4500–NO_3^-F–00	D3867–99(A) ...	I–4545–85 [2]
	Automated, or	353.2, Rev. 2.0 (1993).	4500–NO_3^-F	4500–NO_3^-F	4500–NO_3^-F–00	D3867–99(A) ...	I–4545–85 [2]
	Automated hydrazine	.....	4500–NO_3^-H ...	4500–NO_3^-H ...	4500–NO_3^-H–00.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97	993.30 [3]
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev. 2 [54]
40. Nitrite (as N), mg/L	Spectrophotometric: Manual or.	.....	4500–NO_2^-B ...	4500–NO_2^-B ...	4500–NO_2^-B–00	.....	See footnote [25]
	Automated (Diazotization).	.....	.....	.....	.....	.....	I–4540–85 [2]
	Automated (*bypass cadmium reduction).	353.2, Rev. 2.0 (1993).	4500–NO_3^-F	4500–NO_3^-F	4500–NO_3^-F–00	D3867–99(A) ...	I–4545–85 [2]
	Manual (*bypass cadmium reduction).	.....	4500–NO_3^-E ...	4500–NO_3^-E ...	4500–NO_3^-E–00	D3867–99(B).	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30 [3]
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev.2 [54]
41. Oil and grease—Total recoverable, mg/L.	Hexane extractable material (HEM); n–Hexane extraction and gravimetry.	1664A [42]	.....	5520 B [38]	5520 B–01 [38].	.....	
	Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry..	1664A [42].					

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
42. Organic carbon—Total (TOC), mg/L.	Combustion or oxidation.	.....	5310 B, C, or D	5310 B, C, or D	5310 B, C, or D–00.	D2579–93 (A or B).	973.47,[3] p. 14 [24]
43. Organic nitrogen (as N), mg/L.	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4).						
44. Orthophosphate (as P), mg/L.	Ascorbic acid method:.						
	Automated, or	365.1, Rev. 2.0 (1993).	4500–P F	4500–P F	.....	.....	973.56 [3], I–4601–85 [2]
	Manual single reagent.	.....	4500–P E	4500–P E	.....	D515–88(A)	973.55 [3]
	Manual two reagent	365.3 (Issued 1978)[1].					
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30 [3]
	CIE/UV	.....	.....	.....	.....	.....	D6508, Rev. 2 [54]
45. Osmium—Total [4], mg/L.	Digestion [4] followed by:						
	AA direct aspiration, or.	.....	3111 D	.....	3111 D–99.		
	AA furnace	252.2 (Issued 1978) [1].					
46. Oxygen, dissolved, mg/L.	Winkler (Azide modification), or.	.....	4500–O C	4500–O C	4500–O C–01	D888–92, 03 (A).	973.4 5B [3], I–1575–78 [8]
	Electrode	.....	4500–O G	4500–O G	4500–O G–01	D888–92, 03 (B).	I–1576–78 [8]
47. Palladium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration, or.	.....	3111 B	.....	3111 B–99	.....	p. S27 [10]
	AA furnace	253.2 [1] (Issued 1978).	.....	.....	.....	.....	p. S28 [10]

48. Phenols, mg/L	DCP	.....	.....	.....	.....	.....	See footnote [34]
	Manual distillation [26] Followed by:	420.1 [11] (Rev. 1978).	.....	.....	.....	.....	See footnote [27]
	Colorimetric (4AAP) manual, or.	420.1 [11] (Rev. 1978).	.....	.....	.....	.....	See footnote [27]
	Automated	420.4 Rev. 1.0 (1993).	.....	.....	.....	.....	See footnote [28]
49. Phosphorus (elemental), mg/L.	Gas-liquid chromatography.	.....	.....	.....	.....	.....	973.55 [3]
50. Phosphorus—Total, mg/L.	Persulfate digestion followed by: [20]	.....	4500–P B.5	4500–P B.5	.....	.....	D515–88(A).
	Manual or	365.3 [11] (Issued 1978).	4500–P E	4500–P E	.....	.....	973.56 [3], I–4600–85 [2]
	Automated ascorbic acid reduction.	365.1 Rev. 2.0 (1993).	4500–P F	4500–P F	.....	.....	I–4610–91 [48]
	Semi-automated block digestor.	365.4 [11] (Issued 1974).	.....	.....	.....	D515–88(B)	
51. Platinum—Total, [4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 B	.....	3111 B–99.	.....	See footnote [34]
	AA furnace	255.2 [1].	.....	.....	.....	.....	
	DCP	.....	.....	.....	.....	.....	
52. Potassium—Total, [4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 B	.....	3111 B–99	.....	973.53 [3], I–3630–85 [2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.	.....	
	Flame photometric, or.	.....	3500–K D	3500–K B	3500–K B–97.	.....	
	Colorimetric	.....	.....	.....	.....	.....	317 B [17]
53. Residue—Total, mg/L	Ion Chromatography	.....	.....	.....	.....	.....	D6919–03.
	Gravimetric, 103–105°.	.....	2540 B	2540 B	2540 B–97	.....	I–3750–85 [2]
54. Residue—filterable, mg/L.	Gravimetric, 180°	.....	2540 C	2540 C	2540 C–97	.....	I–1750–85 [2]
55. Residue—non-filterable (TSS), mg/L.	Gravimetric, 103–105 °C post washing of residue.	.....	2540 D	2540 D	2540 D–97	.....	I–3765–85 [2]
56. Residue—settleable, mg/L.	Volumetric, (Imhoff cone), or gravimetric.	.....	2540 F	2540 F	2540 F–97.	.....	
57. Residue—Volatile, mg/L.	Gravimetric, 550 °C	160.4 [1]	.....	.....	.....	.....	I–3753–85 [2]

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
58. Rhodium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration, or.	.....	3111 B	.....	3111 B–99.		
	AA furnace	265.2 [1].					
59. Ruthenium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration, or.	.....	3111 B	.....	3111 B–99.		
	AA furnace	267.2 [1].					
60. Selenium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA furnace	.....	3113 B	.....	3113 B–99	D3859–98, 03 (B).	I–4668–98 [49]
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	AA gaseous hydride	.....	3114 B	.....	3114 B–97	D3859–98, 03 (A).	I–3667–85 [2]
61. Silica—Dissolved,[37] mg/L.	0.45 micron filtration followed by:						
	Colorimetric, Manual or.	.....	4500–Si D	4500–SiO_2 C ...	4500–SiO_2C–97	D859–94, 00 ...	I–1700–85 [2]
	Automated (Molybdosilicate), or.	.....	.....	.....	.....	.....	I–2700–85 [2]
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
62. Silver—Total,[4, 31] mg/L.	Digestion [4, 29] followed by:						
	AA direct aspiration	.....	3111 B or C	.....	3111 B or C–99	.....	974.27 [3], p. 37 [9], I–3720–85 [2]

	AA furnace	.....	3113 B	.....	3113 B–99	.....	.....	I–4724–89[51]
	STGFAA	200.9, Rev. 2.2 (1994).						
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	.....	I–4471–97[50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14[3]	
	DCP	.....	.....	.....	.....	.....	.....	See footnote[34]
63. Sodium—Total,[4] mg/L	Digestion[4] followed by:	.....	.....	.....	.....	.....	.....	
	AA direct aspiration	.....	3111 B	.....	3111 B–99	.....	973.54[3], I–3735–85[2]	
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97[50]	
	DCP, or	.....	.....	.....	.....	.....	.....	See footnote[34]
	Flame photometric ...	.....	3500–Na D	3500–Na B	3500–Na B–97.			
	Ion Chromatography	.....	.....	.....	.....	D 6919–03.		
64. Specific conductance, micromhos/cm at 25 °C.	Wheatstone bridge ..	120.1[1] (Rev. 1982).	2510 B	2510 B	2510 B–97	D1125–95 (99) (A).	973.40[3], I–2781–85[2]	
65. Sulfate (as SO_4), mg/L	Automated colorimetric.	375.2, Rev. 2.0 (1993).	.....	.....	.....	.....	.....	
	Gravimetric	.....	4500–SO_4^{2-}C or D.	4500–SO_4^{2-}C or D.	.....	.....	925.54[3]	
	Turbidimetric	.....	.....	.....	.....	D516–90, 02 ...	426C[30]	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]	
	CIE/UV	.....	.....	.....	.....	.....	.....	D6508, Rev. 2[54]
66. Sulfide (as S), mg/L ...	Titrimetric (iodine), or	.....	4500–S^{2-}F (19th) 4500–S^{2-}E (18th).	4500–S^{2-}F	4500–S^{2-}F–00 ...	.....	I–3840–85[2]	
	Colorimetric (methylene blue).	.....	4500–S^{2-}D	4500–S^{2-}D	4500–S^{2-}D–00.			
	Ion Selective Electrode.	.....	4500–S^{2-}G	4500–S^{2-}G	4500–S^{2-}G–00 ..	D4658–03.		
67. Sulfite (as SO_3), mg/L	Titrimetric (iodine-iodate).	.....	4500–SO_3^{2-}B ..	4500–SO_3^{2-}B ..	4500–SO_3^{2-}B–00.			
68. Surfactants, mg/L	Colorimetric (methylene blue).	.....	5540 C	5540 C	5540 C–00	D2330–88, 02.		
69. Temperature, °C	Thermometric	.....	2550 B	2550 B	2550 B–00	.....	See footnote[32]	
70. Thallium—Total,[4] mg/L.	Digestion[4] followed by:							

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology [58]	Reference (method number or page)					
		EPA [35, 52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/ other
	AA direct aspiration	.....	3111 B	.....	3111 B–99.		
	AA furnace	279.2 [1] (Issued 1978).					
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99.		
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
71. Tin—Total,[4] mg/L	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 B	.....	3111 B–99	.....	I–3850–78 [8]
	AA furnace, or	.....	3113 B	.....	3113 B–99.		
	STGFAA	200.9, Rev. 2.2 (1994).					
	ICP/AES	200.7, Rev. 4.4 (1994).					
72. Titanium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 D	.....	3111 D–99.		
	AA furnace	283.2 [1] (Issued 1978).					
	DCP	.....	.....	.....	.....	.....	See footnote [34]
73. Turbidity, NTU [53]	Nephelometric	180.1, Rev. 2.0 (1993).	2130 B	2130 B	2130 B–01	D1889–94, 00	I–3860–85 [2]
74. Vanadium—Total,[4] mg/L.	Digestion [4] followed by:						
	AA direct aspiration	.....	3111 D	.....	3111 D–99.		
	AA furnace	.....	.....	.....	.....	D3373–93, 03.	
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP, or	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (Gallic Acid).	.....	3500–V D	3500–V B	3500–V B–97.		

75. Zinc –Total [4], mg/L	Digestion [4] followed by:						
	AA direct aspiration [36].	.....	3111 B or C	.....	3111 B or C–99	D1691–95, 02 (A or B).	974.27 [3], p. 37 [9], I–3900–85 [2]
	AA furnace	289.2 [1] (Issued 1978).					
	ICP/AES [36]	200.7, Rev. 4.4 (1994).	3120 B	3120 B	3120 B–99 [59]	.....	I–4471–97 [50]
	ICP/MS	200.8, Rev. 5.4 (1994).	.....	.....	.....	D5673–03	993.14 [3]
	DCP,[36] or	.....	.....	.....	.....	D4190–94, 99	See footnote [34]
	Colorimetric (Dithizone) or.	.....	3500–Zn E.				
	(Zincon)	.....	3500–Zn F	3500–Zn B	3500–Zn B–97 ...	.....	See footnote [33]

Table 1B Notes:

[1] "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI), EPA–600/4–79–020 (NTIS PB 84–128677), Revised March 1983 and 1979 where applicable.

[2] Fishman, M. J., *et al.* "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1989, unless otherwise stated.

[3] "Official Methods of Analysis of the Association of Official Analytical Chemists," Methods Manual, Sixteenth Edition, 4th Revision, 1998.

[4] For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R–94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non–EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP–AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP–AES, and ICP–MS) use EPA Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.

[5] Copper sulfate may be used in place of mercuric sulfate.

[6] Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary: however, manual distillation will be required to resolve any controversies.

[7] Ammonia, Automated Electrode Method, Industrial Method Number 379–75 WE, dated February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[8] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).

[9] American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd st., New York, NY 10036.

[10] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[11] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[12] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data using a nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[14] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[15] The back titration method will be used to resolve controversy.

[16] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.

[17] The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater,* 14th Edition, 1976.

[18] National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

[19] Copper, Biocinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[20] When using a method with block digestion, this treatment is not required.

[21] Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[22] Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[23] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2–113 and 2–117, Hach Chemical Company, Loveland, CO 80537.

[24] Wershaw, R. L.,*et al.*, "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14.

[25] Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[26] Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[27] The approved method is cited in *Standard Methods for the Examination of Water and Wastewater,* 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrometric procedure.

[28] R.F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas–Liquid Chromatography," *Journal of Chromatography,* Vol. 47, No.3, pp. 421–426, 1970.

[29] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[30] The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater,* 15th Edition.

[31] For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH_4OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I_2 to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH>7 with NH_4OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.

[32] Stevens, H.H., Ficke, J. F., and Smoot, G. F., "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.

[33] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2–231 and 2–333, Hach Chemical Company, Loveland, CO 80537.

[34] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991, Thermo Jarrell Ash Corporation, 27 Forge Parkway, Franklin, MA 02038

[35] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals."

[36] Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.

[37] When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.

[38] Only use n-hexane extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). Use of other extraction solvents (e.g., those in the 18th and 19th editions) is prohibited.

[39] Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[40] Nitrogen, Total Kjeldahl, Method PAI–DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[41] Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

[42] Method 1664, Revision A "*n*-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT-HEM; Nonpolar Material) by Extraction and Gravimetry" EPA–821–R–98–002, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.

[43] USEPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–024). The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA–821–R–96–011) are recommended to preclude contamination at low-level, trace metal determinations.

[44] Available Cyanide, Method OIA–1677, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842–9010.

[45] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method," Open File Report (OFR) 00–170.

[46] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 93–449.

[47] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 97–198.

[48] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis" Open File Report (OFR) 92–146.

[49] "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," Open File Report (OFR) 98–639.

[50] "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," Open File Report (OFR) 98-165.

[51] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment," Open File Report (OFR) 93–125.

[52] All EPA methods, excluding EPA Method 300.1, are published in "Methods for the Determination of Metals in Environmental Samples," Supplement I, National Exposure Risk Laboratory-Cincinnati (NERL–CI), EPA/600/R–94/111, May 1994; and "Methods for the Determination of Inorganic Substances in Environmental Samples," NERL–CI, EPA/600/R–93/100, August, 1993. EPA Method 300.1 is available from *http://www.epa.gov/safewater/methods/pdfs/met300.pdf.*

[53] Styrene divinyl benzene beads (e.g., AMCO–AEPA–1 or equivalent) and stabilized formazin (e.g., Hach StablCal™ or equivalent) are acceptable substitutes for formazin.

[54] Method D6508, Rev. 2, "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte," available from Waters Corp, 34 Maple St., Milford, MA, 01757, Telephone: 508/482–2131, Fax: 508/482–3625.

[55] Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821–B–01–009, Revision 1.2, August 2001, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. The toll free telephone number is: 800–553–6847. Note: A 450–W UV lamp may be used in this method instead of the 550–W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.

[56] 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" is available from Lachat Instruments 6645 W. Mill Road, Milwaukee, WI 53218, Telephone: 414–358–4200.

[57] When using sulfide removal test procedures described in Method 335.4, reconstitute particulate that is filtered with the sample prior to distillation.

[58] Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.

[59] Method 245.7, Rev. 2.0, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry," February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, Telephone: 703–461–2100, Fax: 703–461–8056.

[60] The use of EDTA may decrease method sensitivity in some samples. Analysts may omit EDTA provided that all method specified quality control acceptance criteria are met.

[61] Samples analyzed for available cyanide using Methods OIA–1677 or D6888–04 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample analysis to no more than 30 minutes to preclude settling of materials in samples.

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter [1]	EPA method number [2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
1. Acenaphthene	610	625, 1625B	610	6440 B [18th, 19th, 20th].		D4657–92 (99)	See footnote [9], p. 27
2. Acenaphthylene	610	625, 1625B	610	6410 B, 6440 B, [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
3. Acrolein	603	624 [4], 1624B.					
4. Acrylonitrile	603	624 [4], 1624B.					
5. Anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
6. Benzene	602	624, 1624B		6200 B [20th] and 6210 B [18th,19th], 6200 C [20th] and 6220 B [18th,19th].	6200 B and C–97.		
7. Benzidine		625 [5], 1625B	605				See footnote [3], p.1
8. Benzo(a)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
9. Benzo(a)pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
10. Benzo(b)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
11. Benzo(g,h,i) perylene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
12. Benzo(k) fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
13. Benzyl chloride							See footnote [3], p. 130: See footnote [6], p. S102
14. Benzyl butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27

15. Bis(2-chloroethoxy) methane.	611	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
16. Bis(2-chloroethyl) ether.	611	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
17. Bis(2-ethylhexyl) phthalate.	606	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
18. Bromodichloro-methane.	601	624, 1624B	.....	6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
19. Bromoform	601	624, 1624B	.....	6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
20. Bromomethane	601	624, 1624B	.....	6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].	6200 B and C–97.		
21. 4-Bromophenyl phenyl ether.	611	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
22. Carbon tetrachloride	601	624, 1624B	.....	6200 C [20th] and 6230 B [18th, 19th].	6200 C–97	.....	See footnote [3], p. 130
23. 4-Chloro-3-methyl phenol.	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
24. Chlorobenzene	601, 602	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	.....	See footnote [3], p. 130
25. Chloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number[2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
26. 2-Chloroethylvinyl ether.	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
27. Chloroform	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97	.....	See footnote[3], p. 130
28. Chloromethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
29. 2-Chloronaph-thalene	612	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote[9], p. 27
30. 2-Chlorophenol	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B(00, 6420 B–00.	.....	See footnote[9], p. 27
31. 4-Chlorophenyl phenyl ether.	611	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote[9], p. 27
32. Chrysene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
33. Dibenzo(a,h)an-thracene.	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote[9], p. 27
34. Dibromochloro-methane.	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
35. 1,2-Dichloro-benzene	601, 602	624, 1625B	.....	6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97	.....	See footnote[9], p. 27

36. 1,3-Dichloro-benzene	601, 602	624, 1625B	.....	6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97	.....	See footnote [9], p. 27
37. 1,4-Dichloro-benzene	601, 602	624, 1625B	.....	6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 C–97	.....	See footnote [9], p. 27
38. 3,3-Dichloro-benzidine.	.....	625, 1625B	605	6410 B [18th, 19th, 20th].	6410 B–00.		
39. Dichlorodifluoro-methane.	601	.....	.....	6200 C [20th] and 6230 B [18th, 19th].	6200 C–97.		
40. 1,1-Dichloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
41. 1,2-Dichloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
42. 1,1-Dichloroethene	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
43. trans-1,2-Dichloro-ethene.	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
44. 2,4-Dichlorophenol	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
45. 1,2-Dichloro-propane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
46. cis-1,3-Dichloropropene.	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
47. trans-1,3-Dichloropropene.	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
48. Diethyl phthalate	606	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
49. 2,4-Dimethylphenol ...	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
50. Dimethyl phthalate	606	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
51. Di-n-butyl phthalate ...	606	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
52. Di-n-octyl phthalate ...	606	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
53. 2,3-Dinitrophenol	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	
54. 2,4-Dinitrotoluene	609	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
55. 2,6-Dinitrotoluene	609	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
56. Epichlorohydrin	.....	.....	.....	.....	.....	.....	See footnote [3], p. 130; See footnote [6], p. S102
57. Ethylbenzene	602	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97	.....	
58. Fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27

59. Fluorene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
60. 1,2,3,4,6,7,8-Heptachlorodibenzofuran.		1613B [10].					
61. 1,2,3,4,7,8,9-Heptachlorodibenzofuran.		1613B [10].					
62. 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin.		1613B [10].					
63. Hexachlorobenzene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
64. Hexachloro-butadiene	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
65. Hexachlorocyclopentadiene.	612	625 [5], 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
66. 1,2,3,4,7,8-Hexachlorodibenzofuran.		1613B [10].					
67. 1,2,3,6,7,8-Hexachlorodibenzofuran.		1613B [10].					
68. 1,2,3,7,8,9-Hexachlorodibenzofuran.		1613B [10].					
69. 2,3,4,6,7,8-Hexachlorodibenzofuran.		1613B [10].					
70. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin.		1613B [10].					
71. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin.		1613B [10].					
72. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin 1613B [10].		1613B [10].					
73. Hexachloroethane	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27
74. Ideno(1,2,3-cd) pyrene.	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
75. Isophorone	609	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [9], p. 27

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
76. Methylene chloride	601	624, 1624B	.....	6200 C [20th] and 6230 B [18th, 19th].	6200 C–97	.....	See footnote [3], p. 130
77. 2-Methyl-4,6-dinitrophenol.	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
78. Naphthalene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
79. Nitrobenzene	609	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
80. 2-Nitrophenol	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
81. 4-Nitrophenol	604	625, 1625B	.....	6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.	.....	See footnote [9], p. 27
82. N-Nitrosodimethylamine.	607	6255, 1625B ...	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
83. N-Nitrosodi-n-propylamine.	607	6255, 1625B ...	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
84. N-Nitrosodiphenylamine.	607	6255, 1625B ...	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [9], p. 27
85. Octachlorodibenzofuran.	.....	1613B [10]*.					
86. Octachlorodibenzo-p-dioxin.	.....	1613B [10].					
87. 2,2'-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether].	611	625, 1625B	.....	6410 B [18th, 19th, 20th].	6410 B–00.		
88. PCB–1016	608	625	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [3], p. 43; See footnote [8]
89. PCB–1221	608	625	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [3], p. 43; See footnote [8]
90. PCB–1232	608	625	.....	6410 B [18th, 19th, 20th].	6410 B–00	.....	See footnote [3], p. 43; See footnote [8]

91. PCB–1242	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [3], p. 43; See footnote [8]
92. PCB–1248	608	625.					
93. PCB–1254	608	625		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [3], p. 43; See footnote [8]
94. PCB–1260	608	625		6410 B, 6630 B [18th, 19th, 20th].	6410 B–00		See footnote 3, p. 43; See footnote 8
95. 1,2,3,7,8-Pentachlorodibenzofuran		1613B[10].					
96. 2,3,4,7,8-Pentachlorodibenzofuran		1613B[10].					
97. 1,2,3,7,8,-Pentachlorodibenzo-*p*-dioxin.		1613B[10].					
98. Pentachlorophenol	604	625, 1625B		6410 B, 6630 B [18th, 19th, 20th].	6410 B–00		See footnote [3], p. 140; See footnote [9], p. 27
99. Phenanthrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
100. Phenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote [9], p. 27
101. Pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	6410 B–00	D4657–92 (99)	See footnote [9], p. 27
102. 2,3,7,8-Tetrachlorodibenzofuran.		1613B10.					
103. 2,3,7,8-Tetrachlorodibenzo-p-dioxin.		613, 625 [5a], 1613B [10].					
104. 1,1,2,2-Tetra-chloro ethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote [3], p. 130
105. Tetrachloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97		See footnote [3], p. 130

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter[1]	EPA method number[2,7]			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
106. Toluene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	6200 B and C–97.		
107. 1,2,4-Trichloro-benzene.	612	625, 1625B		6410 B [18th, 19th, 20th].	6410 B–00		See footnote [3], p. 130; See footnote [9], p. 27
108. 1,1,1-Trichloro-ethane.	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
109. 1,1,2-Trichloro-ethane.	601	624, 1624B	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C–97		See footnote [3], p. 130.	
110. Trichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
111. Trichlorofluoro-methane.	601	624		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		
112. 2,4,6-Trichlorophenol.	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].	6410 B–00, 6420 B–00.		See footnote [9], p. 27

113. Vinyl chloride	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], ≤6200 C [20th] and 6230 B [18th, 19th].	6200 B and C–97.		

[1] All parameters are expressed in micrograms per liter (μg/L) except for Method 1613B 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, ''Test Procedures for Analysis of Organic Pollutants,'' of this part 136. The full text of Method 1613B is incorporated by reference into this part 136 and is available from the National Technical Information Services as stock number PB95–104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, ''Definition and Procedure for the Determination of the Method Detection Limit,'' of this part 136.

[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 extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624B.

[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.

[5a] 625, screening only.

[6] ''Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency,'' Supplement to the *Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater* (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, 1624B, and 1625B (See appendix A of this part 136) in accordance with procedures each 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 1624B and 1625B) 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. The results should be reported, but cannot be used 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 meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

Parameter	Method	EPA [2, 7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
1. Aldrin	GC	608	6630 B & C	.....	D3086–90, ... D5812-96 (2002) ..	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.		
2. Ametryn	GC		.....	.....	.....	See footnote [3], p. 83; See footnote [6], p S68
3. Aminocarb	TLC		.....	.....	.....	See footnote [3], p. 94; See footnote [6], p. S16
4. Atraton	GC		.....	.....	.....	See footnote [3], p. 83; See footnote [6], p. S68

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1]—Continued

Parameter	Method	EPA[2,7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
5. Atrazine	GC					See footnote[3], p. 83; See footnote[6], p. S68; See footnote[9]
6. Azinphos methyl	GC					See footnote[3], p. 25; See footnote[6], p. S51
7. Barban	TLC					See footnote[3], p. 104; See footnote[6], p. S64
8. α–BHC	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
9. β–BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
10. δ–BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote[8]
	GC/MS	625[5]	6410 B	6410 B–00.		
11. γ-BHC (Lindane)	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
12. Captan	GC		6630 B		D3086–90, D5812–96(02)	See footnote[3], p. 7
13. Carbaryl	TLC					See footnote[3], p. 94, See footnote[6], p. S60
14. Carbo-phenothion	GC					See footnote[4], p. 27; See footnote[6], p. S73
15. Chlordane	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
16. Chloro-propham	TLC					See footnote[3], p. 104; See footnote[6], p. S64.
17. 2,4-D	GC		6640 B			See footnote[3], p. 115; See footnote[4], p. 40
18. 4,4′-DDD	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		
19. 4,4′-DDE	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote[3], p. 7; See footnote[4], p. 27; See footnote[8]
	GC/MS	625	6410 B	6410 B–00.		

20. 4,4′-DDT	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
					D5812–96(02)	
	GC/MS	625	6410 B	6410 B–00.		
21. Demeton-O	GC					See footnote [3], p. 25; See footnote [6], p. S51
22. Demeton-S	GC					See footnote [3], p. 25; See footnote [6], p. S51
23. Diazinon	GC					See footnote [3], p. 25; See footnote [4], p. 27; See footnote [6], p. S51
24. Dicamba	GC					See footnote [3], p. 115
25. Dichlofen-thion	GC					See footnote [4], p. 27; See footnote [6], p. S73
26. Dichloran	GC		6630 B & C			See footnote [3], p. 7
27. Dicofol	GC				D3086–90,	
					D5812–96(02).	
28. Dieldrin	GC	608	6630 B & C			See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.		
29. Dioxathion	GC					See footnote [4], p. 27; See footnote [6], p. S73
30. Disulfoton	GC					See footnote [3], p. 25; See footnote [6], p. S51
31. Diuron	TLC					See footnote [3], p. 104; See footnote [6], p. S64
32. Endosulfan I	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
					D5812–96(02)	
	GC/MS	625[5]	6410 B	6410 B–00.		
33. Endosulfan II	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [8]
					D5812–96(02)	
	GC/MS	625[5]	6410 B	6410 B–00.		
34. Endosulfan Sulfate	GC	608	6630 C			See footnote [8]
	GC/MS	625	6410 B	6410 B–00.		
35. Endrin	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
					D5812–96(02)	
	GC/MS	625[5]	6410 B	6410 B–00.		
36. Endrin aldehyde	GC	608				See footnote [8]
	GC/MS	625				
37. Ethion	GC					See footnote [4], p. 27; See footnote [6], p. S73
38. Fenuron	TLC					See footnote [3], p. 104; See footnote [6], p. S64
39. Fenuron-TCA	TLC					See footnote [3], p. 104; See footnote [6], p. S64

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]—Continued

Parameter	Method	EPA [2, 7]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
40. Heptachlor	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.	D5812–96(02)	
41. Heptachlor epoxide	GC	608	6630 B & C		D3086–90,	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [6], p. S73; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.	D5812– 96(02)	
42. Isodrin	GC					See footnote [4], p. 27; See footnote [6], p. S73
43. Linuron	GC					See footnote [3], p. 104; See footnote [6], p. S64
44. Malathion	GC		6630 C			See footnote [3], p. 25; See footnote [4], p. 27; See footnote [6], p. S51
45. Methiocarb	TLC					See footnote [3], p. 94; See footnote [6], p. S60
46. Methoxy-chlor	GC		6630 B & C		D3086–90, D5812–96(02).	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
47. Mexacar-bate	TLC					See footnote [3], p. 94; See footnote [6], p. S60
48. Mirex	GC		6630 B & C			See footnote [3], p. 7; See footnote [4], p. 27
49. Monuron	TLC					See footnote [3], p. 104; See footnote [6], p. S64
50. Monuron-TCA	TLC					See footnote [3], p. 104; See footnote [6], p. S64
51. Nuburon	TLC					See footnote [3], p. 104; See footnote [6], p. S64
52. Parathion methyl	GC		6630 C			See footnote [3], p. 25; See footnote [4], p. 27
53. Parathion ethyl	GC		6630 C			See footnote [3], p. 25; See footnote [4], p. 27
54. PCNB	GC		6630 B & C			See footnote [3], p. 7
55. Perthane	GC				D3086–90, D5812–96(02).	See footnote [4], p. 27
56. Prometon	GC					See footnote [3], p. 83; See footnote [6], p. S68; See footnote [9]
57. Prometryn	GC					See footnote [3], p. 83; See footnote [6], p. S68; See footnote [9]
58. Propazine	GC					See footnote [3], p. 83; See footnote [6], p. S68; See footnote [9]

59. Propham	TLC					See footnote [3], p. 104; See footnote [6], p. S64
60. Propoxur	TLC					See footnote [3], p. 94; See footnote [6], p. S60
61. Secbumeton	TLC					See footnote [3], p. 83; See footnote [6], p. S68
62. Siduron	TLC					See footnote [3], p. 104; See footnote [6], p. S64
63. Simazine	GC					See footnote [3], p. 83; See footnote [6], p. S68; See footnote [9]
64. Strobane	GC		6630 B & C			See footnote [3], p. 7
65. Swep	TLC					See footnote [3], p. 104; See footnote [6], p. S64
66. 2,4,5–T	GC		6640 B			See footnote [3], p. 115; See footnote [4], p. 40
67. 2,4,5–TP (Silvex)	GC		6640 B			See footnote [3], p. 115; See footnote [4], p. 40
68. Terbuthylazine	GC					See footnote [3], p. 83; See footnote [6], p. S68
69. Toxaphene	GC	608	6630 B & C		D3086–90, D5812–96(02).	See footnote [3], p. 7; See footnote [4], p. 27; See footnote [8]
	GC/MS	625	6410 B	6410 B–00.		
70. Trifluralin	GC		6630 B			See footnote [3], p. 7; See footnote [9]

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

[2] The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this part 136.

[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 α-BHC, γ-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 for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See appendix A of this part 136) 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. The results should be reported, but cannot be used 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 0–3106–93 from "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" U.S. Geological Survey Open File Report 94–37.

TABLE IE—LIST OF APPROVED RADIOLOGIC TEST TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)				
		EPA[1]	Standard Methods 18th, 19th, 20th Ed.	Standard Methods On-line	ASTM	USGS[2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter.	900.0	7110 B	7110 B–00	D1943–90, 96	pp. 75 and 78[3]
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	pp. 75 and 78[3]
4. Beta-Counting error, pCi	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	7500-Ra B	7500-Ra B–01	D2460–90, 97	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500-Ra C	7500-Ra C–01	D3454–91, 97	p. 81

[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

Pharmaceuticals pollutants	CAS registry No.	Analytical method number
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 Δ	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.
triethlyamine	121–44–8	1666/1671.
xylenes	(Note 1)	1624C/1666.

TABLE 1F NOTE:
1. 1624C: 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.); 1666: m,p-xylene 136777–61–2, o-xylene 95–47–6.

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS

EPA Survey Code	Pesticide name	CAS No.	EPA Analytical Method No.(s)
8	Triadimefon	43121–43–3	507/633/525.1/1656
12	Dichlorvos	62–73–7	1657/507/622/525.1
16	2,4–D; 2,4–D Salts and Esters [2,4–Dichloro-phenoxyacetic 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
25	Cyanazine	21725–46–2	629/507
26	Propachlor	1918–16–7	1656/508/608.1/525.1
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) benzo-thiazole].	21564–17–0	637
39	Pronamide	23950–58–5	525.1/507/633.1
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

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued

EPA Survey Code	Pesticide name	CAS No.	EPA Analytical Method No.(s)
53	Acifluorfen	50594–66–6	515.1/515.2/555
54	Alachlor	15972–60–8	505/507/645/525.1/1656
55	Aldicarb	116–06–3	531.1
58	Ametryn	834–12–8	507/619/525.1
60	Atrazine	1912–24–9	505/507/619/525.1/1656
62	Benomyl	17804–35–2	631
68	Bromacil; Bromacil Salts and Esters.	314–40–9	507/633/525.1/1656
69	Bromoxynil	1689–84–5	1625/1661
69	Bromoxynil octanoate	1689–99–2	1656
70	Butachlor	23184–66–9	507/645/525.1/1656
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
82	Chlorothalonil	1897–45–6	508/608.2/525.1/1656
84	Stirofos	961–11–5	1657/507/622/525.1
86	Chlorpyrifos	2921–88–2	1657/508/622
90	Fenvalerate	51630–58–1	1660
103	Diazinon	333–41–5	1657/507/614/622/525.1
107	Parathion methyl	298–00–0	1657/614/622
110	DCPA [Dimethyl 2,3,5,6-tetra-chloro-terephthalate].	1861–32–1	508/608.2/525.1/515.1/515.2/1656
112	Dinoseb	88–85–7	1658/515.1/615/515.2/555
113	Dioxathion	78–34–2	1657/614.1
118	Nabonate [Disodium cyanodithio-imidocarbonate].	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
125	Ethalfluralin	55283–68–6	1656/627 See footnote 1
126	Ethion	563–12–2	1657/614/614.1
127	Ethoprop	13194–48–4	1657/507/622/525.1
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
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
172	Nabam	142–59–6	630/630.1
173	Naled	300–76–5	1657/622
175	Norflurazon	27314–13–2	507/645/525.1/1656
178	Benfluralin	1861–40–1	11656/1627
182	Fensulfothion	115–90–2	1657/622
183	Disulfoton	298–04–4	1657/507/614/622/525.1
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
208	Permethrin	52645–53–1	608.2/508/525.1/1656/1660
212	Phorate	298–02–2	1657/622
218	Busan 85 [Potassium dimethyldithiocarbamate].	128–03–0	630/630.1

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued

EPA Survey Code	Pesticide name	CAS No.	EPA Analytical Method No.(s)
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate].	51026–28–9	630/630.1
220	KN Methyl [Potassium N-methyl-dithiocarbamate].	137–41–7	630/630.1
223	Prometon	1610–18–0	507/619/525.1
224	Prometryn	7287–19–6	507/619/525.1
226	Propazine	139–40–2	507/619/525.1/1656
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	505/507/619/525.1/1656
241	Carbam-S [Sodium dimethyldithiocarbanate].	128–04–1	630/630.1
243	Vapam [Sodium methyldithiocarbamate].	137–42–8	630/630.1
252	Tebuthiuron	34014–18–1	507/525.1
254	Terbacil	5902–51–2	507/633/525.1/1656
255	Terbufos	13071–79–9	1657/507/614.1/525.1
256	Terbuthylazine	5915–41–3	619/1656
257	Terbutryn	886–50–0	507/619/525.1
259	Dazomet	533–74–4	630/630.1/1659
262	Toxaphene	8001–35–2	1656/505/508/608/617/525.1
263	Merphos [Tributyl phosphorotrithioate].	150–50–5	1657/507/525.1/622
264	Trifluralin	1582–09–8	1656/508/617/627/525.1
268	Ziram [Zinc dimethyldithiocarbamate].	137–30–4	630/630.1

[1] Monitor and report as total Trifluralin.

TABLE IH—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th Ed.	Standard methods online	AOAC, ASTM, USGS	Other
Bacteria:						
1. E. coli, number per 100 mL.	MPN [6,8,14] multiple tube,		9221 B.1/9221 F [11,13]	9221 B.1–99/9221 F [11,13].		
	Multiple tube/multiple well,		9223 B [12]	9223 B–97 [12]	991.15 [10]	Colilert® [12,16] Colilert-18® [12,15,16].
	MF [2,5,6,7,8] two step, or	1103.1 [19]	9222 B/9222 G [18], 9213 D.	9222 B–97/9222 G [18]	D5392–93 [9].	
	Single step	1603 [20], 1604 [21]				mColiBlue-24® [17].
2. Enterococci, number per 100 mL.	MPN [6,8] multiple tube,		9230 B	9230 B–93.		
	Multiple tube/multiple well				D6503–99 [9]	Enterolert® [12,22].
	MF [2,5,6,7,8] two step	1106.1 [23]	9230 C	9230 C–93	D5259–92 [9].	
	Single step, or	1600 [24].				
	Plate count	p. 143 [3].				
Protozoa:						
3. *Cryptosporidium*	Filtration/IMS/FA	1622 [25],1623 [26].				
4. *Giardia*	Filtration/IMS/FA	1623 [26].				

[1] The method must be specified when results are reported.

[2] A 0.45 μm 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, OH. EPA/600/8–78/017.

[4] [Reserved]

[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] ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[10] AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877–2417.

[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 β-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 μg/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., 1 IDEXX Drive, Westbrook, ME 04092.

[17] A description of the mColiBlue24® test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

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

[19] USEPA. July 2006. Method 1103.1: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–010.

[20] USEPA. July 2006. Method 1603: *Escherichia coli (E. coli)* in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–011.

[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 USEPA. September 2002.: Method 1604: Total Coliforms and *Escherichia coli (E. coli)* in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA 821–R–02–024.

[22] A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

[23] USEPA. July 2006. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–008.

[24] USEPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–009.

[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.* USEPA. 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–026.

[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. USEPA. 2001. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–025.

(b) 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. The full texts of all the test procedures cited are available for inspection 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*.

REFERENCES, SOURCES, COSTS, AND TABLE CITATIONS:

(1) The full texts of Methods 601–613, 624, 625, 1613, 1624, and 1625 are printed in appendix A of this part 136. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part 136. The full text of Method 200.7 is printed in appendix C of this part 136. Cited in: Table IB, Note 5; Table IC, Note 2; and Table ID, Note 2.

(2) 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. Available at *http://www.epa.gov/clariton/srch.htm* or from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB–290329/A.S. Table IA, Note 3; Table IH, Note 3.

(3) ''Methods for Chemical Analysis of Water and Wastes,'' U.S. Environmental Protection Agency, EPA–600/4–79–020, March 1979, or ''Methods for Chemical Analysis of Water and Wastes,'' U.S. Environmental Protection Agency, EPA–600/4–79–020, Revised March 1983. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IB, Note 1.

(4) ''Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,'' U.S. Environmental Protection Agency, 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IC, Note 3; Table D, Note 3.

(5) ''Prescribed Procedures for Measurement of Radioactivity in Drinking Water,'' U.S. Environmental Protection Agency, EPA–600/4–80–032, 1980. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IE, Note 1.

(6) American Public Health Association. 1992, 1995, and 1998. Standard Methods for the Examination of Water and Wastewater. 18th, 19th, and 20th Edition (respectively). Available from: American Public Health Association, 1015 15th Street, NW., Washington, DC 20005. Standard Methods Online is available through the Standard Methods Web site (*http://www.standardmethods.org*). Tables IA, IB, IC, ID, IE, and IH.

(7) Ibid, 15th Edition, 1980. Table IB, Note 30; Table ID.

(8) Ibid, 14th Edition, 1975. Table IB, Notes 17 and 27.

(9) ''Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency,'' Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater, 1981. Available from: American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20036. Cost available from publisher. Table IB, Note 10; Table IC, Note 6; Table ID, Note 6.

(10) ASTM International. Annual Book of ASTM Standards, Water, and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994, 1996, 1999, Volume 11.02, 2000, and individual standards published after 2000. Available from: ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959, or *http://www.astm.org*. Tables IA, IB, IC, ID, IE, and IH.

(11) USGS. 1989. 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, U.S. Geological Survey, U.S. Department of the Interior, Reston, Virginia. Available

from USGS Books and Open-File Reports Section, Federal Center, Box 25425, Denver, Colorado 80225. Table IA, Note 5; Table IH.

(12) ''Methods for Determination of Inorganic Substances in Water and Fluvial Sediments,'' by M.J. Fishman and Linda C. Friedman, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5 Chapter A1 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $108.75 (subject to change). Table IB, Note 2.

(13) ''Methods for Determination of Inorganic Substances in Water and Fluvial Sediments,'' N.W. Skougstad and others, editors. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1 (1979). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $10.00 (subject to change), Table IB, Note 8.

(14) ''Methods for the Determination of Organic Substances in Water and Fluvial Sediments,'' Wershaw, R.L., et al, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $0.90 (subject to change). Table IB, Note 24; Table ID, Note 4.

(15) ''Water Temperature—Influential Factors, Field Measurement and Data Presentation,'' by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $1.60 (subject to change). Table IB, Note 32.

(16) ''Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters,'' by M.J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76–77 (1976). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: $13.50 (subject to change). Table IE, Note 2.

(17) AOAC-International. Official Methods of Analysis of AOAC-International, 16th Edition, (1995). Available from: AOAC-International, 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877. Table IB, See footnote 3.

(18) ''American National Standard on Photographic Processing Effluents,'' April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018. Table IB, Note 9.

(19) ''An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color,'' NCASI Technical Bulletin No. 253, December 1971. Available from: National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016. Cost available from publisher. Table IB, Note 18.

(20) Ammonia, Automated Electrode Method, Industrial Method Number 379–75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 7.

(21) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 14.

(22) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 13.

(23) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 16.

(24) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979, Method and price available from Hach Chemical Company, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 19.

(25) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA. October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 21.

(26) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water,

Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 389 Loveland, Colorado 80537. Table IB, Note 22.

(27) Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 23.

(28) Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 25.

(29) Zincon Method for Zinc, Method 8009. Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 33.

(30) ''Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography,'' by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421–426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. Table IB, Note 28.

(31) ''Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes'', Method AES 0029, 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table B, Note 34.

(32) ''Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106–0200, April 16, 1992. Available from the CEM Corporation. Table IB, Note 36.

(33) ''Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk'' Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220–9E–10, St. Paul, MN 55144–1000. Method available from 3M Corporation. Table IC, Note 8 and Table ID, Note 8.

(34) 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. Available at *http://www.epa.gov/epahome/index/sources.htm* or from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108488. Table IA, Note 25.

(35) ''Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 39.

(36) ''Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 40.

(37) ''Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 41.

(38) USEPA. October 2002. Short-Term Methods for Measuring 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. Available at *http://www.epa.gov/epahome/index/sources.htm* or from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108489. Table IA, Note 26.

(39) USEPA. October 2002. Short-Term Methods for Measuring 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. Available at *http://www.epa.gov/epahome/index/sources.htm* or from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108490. Table IA, Note 27.

(40) EPA Methods 1666, 1667, and 1671 listed in the table above are published in the compendium titled Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters (EPA 821–B–98–016). EPA Methods 502.2 and 524.2 have been incorporated by reference into 40 CFR 141.24 and are in Methods for the Determination of Organic Compounds in Drinking Water, EPA–600/4–88–039, December 1988, Revised, July 1991, and Methods for the Determination of Organic Compounds in Drinking Water-Supplement II, EPA–600/R–92–129, August 1992, respectively. These EPA test method compendia are available from the National Technical Information Service, NTIS PB91–231480 and PB92–207703, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll-free number is 800–553–6847. ASTM test methods D3371, D3695, and D4763 are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.

(41) USEPA. 2002. Method 1631, Revision E, ''Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry.'' September 2002. Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–019). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161. Publication No. PB2002–108220. Cost: $25.50 (subject to change).

(42) [Reserved]

(43) Method OIA–1677, Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry. August 1999. ALPKEM, OI Analytical, Box 648, Wilsonville, Oregon 97070 (EPA–821–R–99–013). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161. Publication No. PB99–132011. Cost: $22.50. Table IB, Note 44.

(44) ''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'', Open File Report (OFR) 00–170. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 45.

(45) ''Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry'', Open File Report (OFR) 93–449. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 46.

(46) ''Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum in Water by Graphite Furnace Atomic Absorption Spectrophotometry'', Open File Report (OFR) 97–198. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 47.

(47) ''Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis'' Open File Report (OFR) 92–146. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 48.

(48) ''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'' Open File Report (OFR) 98–639. Table IB, Note 49.

(49) ''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'' , Open File Report (OFR) 98–165. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 50.

(50) ''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'' U.S.Geological Survey Open File Report 94–37. Available from: U.S.

Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table ID, Note 9.

(51) ''Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments'', Open File Report (OFR) 93–125. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Table IB, Note 51; Table IC, Note 9.

(52) IDEXX Laboratories, Inc. 2002. Description of Colilert®, Colilert-18®, Quanti-Tray®, Quanti-Tray®/2000, Enterolert® methods are available from IDEXX Laboratories, Inc., One Idexx Drive, Westbrook, Maine 04092. Table IA, Notes 17 and 23; Table IH, Notes 16 and 22.

(53) Hach Company, Inc. Revision 2, 1999. Description of m-ColiBlue24® Method, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave, Ames IA 50010. Table IA, Note 18; Table IH, Note 17.

(54) USEPA. July 2006. Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–621–R–06–010. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 19.

(55) USEPA. July 2006. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–621–R–06–008. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 23

(56) USEPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–821–R–06–011. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 19; Table IH, Note 20.

(57) Brenner *et al*. 1993. New Medium for the Simultaneous Detection of Total Coliforms and *Escherichia coli* in Water. Appl. Environ. Microbiol. 59:3534–3544. Available from the American Society for Microbiology, 1752 N Street NW., Washington DC 20036. Table IH, Note 21.

(58) USEPA. September 2002. Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–821–R–02–024. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 20.

(59) USEPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–821–R–06–009. Available at *http://www.epa.gov/waterscience/methods/*. Table IA, Note 24; Table IH, Note 24.

(60) USEPA. April 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC EPA–821–R–01–026. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 25.

(61) USEPA. April 2001. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA–821–R–01–025. Available at *http://www.epa.gov/waterscience/methods/*. Table IH, Note 26.

(62) AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. AOAC International, 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877–2417. Table IA, Note 11; Table IH.

(63) Waters Corporation. Method D6508, Rev. 2, ''Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte,'' available from Waters Corp, 34 Maple Street, Milford, MA 01757, Telephone: 508/482–2131, Fax: 508/482–3625, Table IB, See footnote 54.

(64) Kelada-01, ''Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate,'' EPA 821–B–01–009 Revision 1.2, August 2001 is available from

National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. Telephone: 800–553–6847. Table IB, See footnote 55.

(65) 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 is available from Lachat Instruments 6645 W. Mill Road, Milwaukee, WI 53218, Telephone: 414–358–4200. Table IB, See footnote 56.

(66) ''Methods for the Determination of Metals in Environmental Samples,'' Supplement I, National Exposure Risk Laboratory-Cincinnati (NERL–CI), EPA/600/R–94/111, May 1994; and ''Methods for the Determination of Inorganic Substances in Environmental Samples,'' NERL–CI, EPA/600/R–93/100, August, 1993 are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800–553–6847. Table IB.

(67) ''Determination of Inorganic Ions in Drinking Water by Ion Chromatography,'' Rev. 1.0, 1997 is available from from *http://www.epa.gov/safetwater/methods/met300.pdf*. Table IB.

(68) Table IG Methods are available in ''Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I,'' EPA 821–R–93–010A, August 1993 Revision I, and ''Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II,'' EPA 821–R–93–010B (August 1993) are available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161. Telephone: 800–553–6847.

(69) Method 245.7, Rev. 2.0, ''Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry,'' February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, Telephone: 703–461–8056. Table IB, See footnote 59.

(70) USEPA. July 2006. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using Lauryl Tryptose Broth (LTB) and EC Medium. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA 821–R–06–012. Available at *http://www.epa.gov/waterscience/methods/*.

(71) USEPA. July 2006. 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. Available at *http://www.epa.gov/waterscience/methods/*.

(72) USEPA. July 2006. 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. Available at *http://www.epa.gov/waterscience/methods/*.

(c) 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 recommendation of the Alternate Test Procedure Program Coordinator, Washington, DC.

(d) Under certain circumstances, the Administrator may approve additional alternate test procedures for nationwide use, upon recommendation by the Alternate Test Procedure Program Coordinator, Washington, DC.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, IG and IH 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 variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by

the Regional Administrator, to the Alternate Test Procedure Program Coordinator, Washington, DC, for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Alternate Test Procedure Program Coordinator, the Regional Administrator may grant a variance applicable to the specific discharge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

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

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*.	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22,23]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22]
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22]
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22]
Table IA—Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C [16]	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C [18]	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C [18]	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C [18]	48 hours.
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P, FP G	Cool, ≤6 °C [18]	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C [18]	48 hours.
23–24. Cyanide, total or available (or CATC).	P, FP, G	Cool, ≤6 °C [18], NaOH to pH>12 [6], reducing agent [5].	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
Table IB—Metals: [7]			
18. Chromium VI	P, FP, G	Cool, ≤6 °C [18], pH = 9.3–9.7 [20]	28 days.
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap [17].	5 mL/L 12N HCl or 5 mL/L BrCl [17].	90 days.[17]
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH<2, or at least 24 hours prior to analysis [19].	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C [18]	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C [18]	48 hours.
41. Oil and grease	G	Cool to ≤6 °C [18], HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C [18], HCl, H_2SO_4, or H_3PO_4 to pH<2.	28 days.
44. Orthophosphate	P, FP, G	Cool, ≤6 °C [18]	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
49. Phosphorous (elemental)	G	Cool, ≤6 °C [18]	48 hours.
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
53. Residue, total	P, FP, G	Cool, ≤6 °C [18]	7 days.
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C [18]	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C [18]	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C [18]	48 hours.

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

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C [18]	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C [18]	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C [18]	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C [18]	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C [18], add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, ≤6 °C [18]	48 hours.
69. Temperature	P, FP, G	None required	Analyze.
73. Turbidity	P, FP, G	Cool, ≤6 °C [18]	48 hours.
Table IC—Organic Tests [8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5].	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons.	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2 [9].	14 days.[9]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], pH to 4–5 [10].	14 days.[10]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11].	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
7, 38. Benzidines [11,12]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5].	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters [11].	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines [11,14]	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
88–94. PCBs [11]	G, FP-lined cap	Cool, ≤6 °C [18]	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone [11].	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11].	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers [11]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
29, 35–37, 63–65, 107. Chlorinated hydrocarbons [11].	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs [11].			
Aqueous Samples: Field and Lab Preservation.	G	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], pH<9.	1 year.
Solids and Mixed-Phase Samples: Field Preservation.	G	Cool, ≤6 °C [18]	7 days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C [18]	24 hours.
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation.	G	Freeze, ≤ −10 °C	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides [11]	G, FP-lined cap	Cool, ≤6 °C [18], pH 5–9 [15]	7 days until extraction, 40 days after extraction.
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH<2	6 months.
Table IH—Bacterial Tests:			
1. *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22]
2. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5].	6 hours.[22]
Table IH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours.[21]

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

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
9. Giardia	LDPE; field filtration ..	0–8 °C	96 hours.[21]

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

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

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

[5] Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If $NaBH_4$ or $NaAsO_2$ is used, 25 mg/L $NaBH_4$ or 100 mg/L $NaAsO_2$ will reduce more than 50 mg/L of chlorine (see method "Kelada-01" and/or Standard Method 4500–CN^- for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

[6] Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) Sulfur: To remove elemental sulfur (S_8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH < 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) Sulfite, thiosulfate, or thiocyanate: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA–1677) to preclude cyanide loss or positive interference.

(4) Aldehyde: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) Carbonate: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500–CN.B.3.d).

(6) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

[7] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

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

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

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (*i.e.*, use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

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

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[16] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[17] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[18] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[19] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

[20] To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

[21] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

[22] Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

[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.

[38 FR 28758, Oct. 16, 1973]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting §136.3, see the List of CFR Sections Affected, which appears in the Finding Aids section of the printed volume and on GPO Access.

§ 136.4 Application for alternate test procedures.

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

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

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

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

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

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

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

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

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

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

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

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

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976; 62 FR 30763, June 5, 1997; 72 FR 11239, Mar. 12, 2007]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will