SUBCHAPTER D—WATER PROGRAMS (CONTINUED)

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

Sec.

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.

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:

(a) 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,

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

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

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

§ 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, and IF. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, and IF. 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. Information regarding obtaining these documents can be obtained from the EPA Office of Water Statistics and Analytical Support Branch at 202–566–1000. Documents may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue, NW., Room B135, 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 anys 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, IE, and IF, 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 (b) or (c) of this section or 40 CFR 401.13) other test procedures may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th Ed.	ASTM	AOAC	USGS	Other
Bacteria:							
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube 3 dilution, or	p. 132 [3]	9221C E [4]				
	Membrane filter (MF) [2], single step.	p. 124 [3]	9222D [4]			B–0050–85 [5]	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132 [3]	9221C E [4]				
	MF, single step [6]	p. 124 [3]	9222D [4]				
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]				
	MF [2], single step or two step	p. 108 [3]	9222B [4]			B–0025–85 [5]	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]				
	MF [2] with enrichment	p. 111 [3]	9222(B+B.5c) [4]				
5. *E. coli,* number per 100 mL [28].	MPN [7,9,15], multiple tube,		9221B.1/9221F [4,12,14]				
	multiple tube/multiple well,		9223B [4,13]		991.15 [11]		Colilert ® [13,17] Colilert-18 ® [13,16,17]
	MF [2,6,7,8,9] two step, or	1103.1 [20]	9222B/9222G [4,19] 9213D [4]	D5392–93 [10]			
	single step	1603 [21] 1604 [22]					mColiBue 24 [18]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution,	p. 139 [3]	9230B [4], 9230C [4]				
	MF [2], or	p. 136 [3]			B–0055–85 [5]		
	Plate count	p. 143 [4]					
7. Enterococci, number per 100 mL.	MPN [7, 9] multiple tube		9230B [4]				
	multiple tube/multiple well			D6503–99 [10]			Enterolert ® [13,23]
	MF [2,6,7,8,9] two step	1106.1 [24]	9230C [4]	D5259–92 [10]			
	single step, or	1600 [25]					
	Plate count	p. 143 [3]					
Protozoa:							
8. *Cryptosporidium* [28]	Filtration/IMS/FA	1622 [26] 1623 [27]					
9. *Giardia* [28]	Filtration/IMS/FA	1623 [27]					
Aquatic Toxicity:							
10. Toxicity, acute, fresh water organisms, LC50, percent effluent.	*Ceriodaphnia dubia* acute	2002.0 [29]					

TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS—Continued

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th Ed.	ASTM	AOAC	USGS	Other
	Daphnia pulex and *Daphnia magna* acute.	2021.0 [29]					
	Fathead Minnow, *Pimephales promelas,* and Bannerfin shiner, *Cyprinella leedsi,* acute.	2000.0 [29]					
	Rainbow Trout, *Oncorhynchus mykiss,* and brook trout, *Salvelinus fontinalis,* acute.	2019.0 [29]					
11. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC50, percent effluent.	Mysid, *Mysidopsis bahia,* acute ..	2007.0 [29]					
	Sheepshead Minnow, *Cyprinodon variegatus,* acute.	2004.0 [29]					
	Silverside, *Menidia beryllina, Menidia menidia,* and *Menidia peninsulae,* acute.	2006.0 [29]					
12. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent.	Fathead minnow, *Pimephales promelas,* larvalsurvival and growth.	1000.0 [30]					
	Fathead minnow, *Pimephales promelas,* embryo-larval survival and teratogenicity.	1001.0 [30]					
	Daphnia, *Ceriodaphnia dubia,* survival and reproduction.	1002.0 [30]					
	Green alga, *Selenastrum capricornutum,* growth.	1003.0 [30]					
13. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC25, percent effluent.	Sheepshead minnow, *Cyprinodon variegatus,*larval survival and growth.	1004.0 [31]					
	Sheepshead minnow, *Cyprinodon variegatus,*embryo-larval survival and teratogenicity.	1005.0 [31]					
	Inland silverside, *Menidia beryllina,* larval survival and growth.	1006.0 [31]					
	Mysid, *Mysidopsis bahia,* survival, growth,and fecundity.	1007.0 [31]					

	Sea urchin, *Arbacia punctulata,* fertilization.	1008.0 [31]					

Notes to Table IA:

[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, Ohio. EPA/600/8–78/017.

[4] APHA. 1998, 1995, 1992. Standard Methods for the Examination of Water and Wastewater. American Public Health Association. 20th, 19th, and 18th Editions. Amer. Publ. Hlth. Assoc., Washington, D.C.

[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 Interior, Reston, Virginia.

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

[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. American Society for Testing and Materials. 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, Maryland 20877–2417.

[12] 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.

[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] 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.

[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® or 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., One IDEXX Drive, Westbrook, Maine 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] Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.

[20] USEPA. 2002. 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 D.C. EPA–821–R–02–020.

[21] USEPA. 2002. 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 D.C. EPA–821–R–02–023.

[22] 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. 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.

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

[24] USEPA. 2002. 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–02–021.

[25] USEPA. 2002. 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–02–022.

[26] 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.

[27] 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.

[28] Recommended for enumeration of target organism in ambient water only.

[29] 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.
[30] 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.
[31] 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, units and method	Reference (method number or page)				
	EPA [1, 35]	*Standard Methods* [Edition(s)]	ASTM	USGS [2]	Other
1. Acidity, as $CaCO_3$, mg/L:					
Electrometric endpoint or phenolphthalein endpoint.	305.1	2310 B(4a) [18th, 19th, 20th].	D1067–92	I–1020–85 I–2030–85	
2. Alkalinity, as $CaCO_3$, mg/L:					
Electrometric or Colorimetric titration to pH 4.5, manual or automatic.	310.1 310.2.	2320 B [18th, 19th, 20th] ...	D1067–92	I–1030–85 I–2030–85	973.43 [3]
3. Aluminium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	202.1	3111 D [18th, 19th]		I–3051–85	
AA furnace	202.2	3113 B [18th, 19th].			
Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES) [36].	200.7 [5]	3120 B [18th, 19th, 20th] ...		I–4471–97 [50]	
Direct Current Plasma (DCP) [36].			D4190–94		Note 34.
Colorimetric (Eriochrome cyanine R).		3500–Al B [20th] and 3500–Al D [18th, 19th].			
4. Ammonia (as N), mg/L:					
Manual, distillation (at pH 9.5) [6] followed by.	350.2	4500–NH_3 B [18th, 19th, 20th].			973.49 [3]
Nesslerization	350.2	4500–NH_3 C [18th]	D1426–98(A)	I–3520–85	973.49 [3]
Titration	350.2	4500–NH_3 C [19th, 20th] and 4500–NH_3 E [18th].			
Electrode	350.3	4500–NH_3 D or E [19th, 20th] and 4500–NH_3 F or G [18th].	D1426–98(B).		
Automated phenate, or	350.1	4500–NH_3 G [19th, 20th] and 4500–NH_3 H [18th].		I–4523–85	
Automated electrode					Note 7.
5. Antimony-Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	204.1	3111 B [18th, 19th]			
AA furnace	204.2	3113 B [18th, 19th]			
ICP/AES [36]	200.7 [5]	3120 B [18th, 19th, 20th] ...			
6. Arsenic-Total[4] mg/L:					

Digestion[4] followed by	206.5				
AA gaseous hydride	206.3	3114 B 4.d [18th, 19th]	D2972–97(B)	I–3062–85	
AA furnace	206.2	3113 B [18th, 19th]	D2972–97(C)	I–4063–98[49]	
ICP/AES[36] or	200.7[5]	3120 B [18th, 19th, 20th].			
Colorimetric (SDDC)	206.4	3500–As B [20th] and 3500–As C [18th, 19th].	D2972–97(A)	I–3060–85	
7. Barium-Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[14]	208.1	3111 D [18th, 19th]		I–3084–85	
AA furnace	208.2	3113 B [18th, 19th]	D4382–95		
ICP/AES[14]	200.7[5]	3120 B [18th, 19th, 20th]			
DCP[14]					Note 34.
8. Beryllium-Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	210.1	3111 D [18th, 19th]	D3645–93(88)(A)	I–3095–85	
AA furnace	210.2	3113 B [18th, 19th]	D3645–93(88)(B)		
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP, or			D4190–94		Note 34.
Colorimetric (aluminon		3500–Be D [18th, 19th]			
9. Biochemical oxygen demand (BOD_5), mg/L: Dissolved Oxygen Depletion	405.1	5210 B [18th, 19th, 20th]		I–1578–78[8]	973.44,[3] p. 17[9]
10. Boron[37]–Total, mg/L: Colorimetric (curcumin)	212.3	4500–B B [18th, 19th, 20th]		I–3112–85	
ICP/AES, or	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP			D4190–94		Note 34.
11. Bromide, mg/L: Titrimetric	320.1		D1246–95(C)	I–1125–85	p. S44[10]
12. Cadmium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	213.1	3111 B or C [18th, 19th]	D3557–95 (A or B)	I–3135–85 or I–3136–85	974.27,[3] p. 37[9]
AA furnace	213.2	3113 B [18th, 19th]	D3557–95(D)	I–4138–89[51]	
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I–1472–85 or I–4471–97[50]	
DCP[36]			D4190–94		Note 34.
Voltametry[11], or			D3557–95(C).		
Colorimetric (Dithizone)		3500–Cd D [18th, 19th].			
13. Calcium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	215.1	3111 B [18th, 19th]	D511–93(B)	I–3152–85	
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP, or					Note 34.
Titrimetric (EDTA)	215.2	3500–Ca B [20th] and 3500–Ca D [18th, 19th].	D511–93(A).		
14. Carbonaceous biochemical oxygen demand (CBOD 3), mg/L[12]: Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B [18th, 19th, 20th].			
15. Chemical oxygen demand (COD), mg/L: Titrimetric	410.1	5220 C [18th, 19th, 20th]	D1252–95(A)	I–3560–85	973.46,[3] p. 17[9]
or	410.2			I–3562–85	

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1, 35]	*Standard Methods* [Edition(s)]	ASTM	USGS [2]	Other
	410.3.				
Spectrophotometric, manual or automatic.	410.4.	5220 D [18th, 19th, 20th]	D1252–95(B)	I–3561–85	Notes 13, 14.
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or		4500–Cl^- B [18th, 19th, 20th].	D512–89(B)	I–1183–85	
(Mercuric nitrate)	325.3	4500–Cl^- C [18th, 19th, 20th].	D512–89(A)	I–1184–85	973.51 [3]
Colorimetric, manual or				I–1187–85	
Automated (Ferricyanide)	325.1 or 325.2	4500–Cl^- E [18th, 19th, 20th].		I–2187–85	
17. Chlorine—Total residual, mg/L; Titrimetric:					
Amperometric direct	330.1	4500–Cl D [18th, 19th, 20th].	D1253–86(92).		
Iodometric direct	330.3	4500–Cl B [18th, 19th, 20th].			
Back titration ether end-point [15] or.	330.2	4500–Cl C [18th, 19th, 20th].			
DPD-FAS	330.4	4500–Cl F [18th, 19th, 20th].			
Spectrophotometric, DPD	330.5	4500–Cl G [18th, 19th, 20th].			
Or Electrode					Note 16.
18. Chromium VI dissolved, mg/L; 0.45 micron filtration followed by:					
AA chelation-extraction or	218.4	3111 C [18th, 19th]		I–1232–85	
Colorimetric (Diphenylcarbazide).		3500–Cr B [20th] and 3500–Cr D [18th, 19th].	D1687–92(A)	I–1230–85	
19. Chromium-Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	218.1	3111 B [18th, 19th]	D1687–92(B)	I–3236–85	974.27 [3]
AA chelation-extraction	218.3	3111 C [18th, 19th].			
AA furnace	218.2	3113 B [18th, 19th]	D1687–92(C)	I–3233–93 [46].	
ICP/AES [36]	200.7 [5]	3120 B [18th, 19th, 20th].			
DCP [36] or			D4190–94		Note 34.
Colorimetric (Diphenylcarbazide).		3500–Cr B [20th] and 3500–Cr D [18th, 19th].			
20. Cobalt—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	219.1	3111 B or C [18th, 19th]	D3558–94(A or B)	I–3239–85	p. 37 [9]
AA furnace	219.2	3113 B [18th, 19th]	D3558–94(C)	I–4243–89 [51].	
ICP/AES	200.7 [5]	3120 B [18th, 19th, 20th]		I–4471–97 [50].	

DCP			D4190–94		Note 34.
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:					
Colorimetric (ADMI), or.		2120 E [18th, 19th, 20th]			
(Platinum cobalt), or	110.1	2120 B [18th, 19th, 20th]		I–1250–85	Note 18.
Spectrophotometric	110.2	2120 C [18th, 19th, 20th].			
	110.3				
22. Copper—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	220.1	3111 B or C [18th, 19th]	D1688–95(A or B)	I–3270–85 or I–3271–85	974.27[3] p. 37[9]
AA furnace	220.2	3113 B [18th, 19th]	D1688–95(C)	I–4274–89[51]	
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I—4471—97[50]	Note 34.
DCP[36] or			D4190–94		
Colorimetric (Neocuproine) or.		3500–Cu B [20th] and 3500–Cu D [18th, 19th].			
(Bicinchoninate)		3500–Cu C [20th] and 3500–As B [18th, 19th].			Note 19.
23. Cyanide—Total, mg/L:					
Manual distillation with $MgCl_2$ followed by..		4500–CN C [18th, 19th, 20th].	D2036–98(A)		
Titrimetric, or		4500–CN D [18th, 19th, 20th].			p. 22[9]
Spectrophotometric, manual or.	335.2[31]	4500–CN E [18th, 19th, 20th].	D2036–98(A)	I–3300–85	
Automated[20]	335.3[31]			I–4302–85	
24. Available Cyanide, mg/L:					
Manual distillation with $MgCl_2$ followed by titrimetric or Spectrophotometric.	335.1	4500–CN G [18th, 19th, 20th].	D2036–98(B)		
Flow injection and ligand exchange, followed by amperometry.					OIA–1677[44]
25. Fluoride—Total, mg/L:					
Manual distillation[6] followed by.		4500–F B [18th, 19th, 20th]			
Electrode, manual or	340.2	4500–F C [18th, 19th, 20th]	D1179–93(B)	I–4327–85	
Automated					
Colorimetric (SPADNS)	340.1	4500–F D [18th, 19th, 20th]	D1179–93(A)		
Or Automated complexone	340.3	4500–F E [18th, 19th, 20th]			
26. Gold—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	231.1	3111 B [18th, 19th]			
AA furnace, or	231.2				
DCP					Note 34.
27. Hardness—Total, as $CaCO_3$, mg/L:					
Automated colorimetric,	130.1				

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1, 35]	*Standard Methods* [Edition(s)]	ASTM	USGS [2]	Other
Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration (See Parameters 13 and 33).	130.2	2340 B or C [18th, 19th, 20th].	D1126–86(92)	I–1338–85	973.52B [3]
28. Hydrogen ion (pH), pH units:					
Electrometric measurement, or.	150.1	4500–H^+ B [18th, 19th, 20th].	D1293–84 (90)(A or B)	I–1586–85	973.41 [3]
Automated electrode				I–2587–85	Note 21.
29. Iridium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration or	235.1	3111 B [18th, 19th]			
AA furnace	235.2				
30. Iron—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	236.1	3111 B or C [18th, 19th]	D1068–96(A or B)	I–3381–85	974.27 [3]
AA furnace	236.2	3113 B [18th, 19th]	D1068–96(C)		
ICP/AES [36]	200.7 [5]	3120 B [18th, 19th, 20th]		I–4471–97 [50]	
DCP [36] or			D4190–94		Note 34.
Colorimetric (Phenanthroline).		3500–Fe B [20th] and 3500–Fe D [18th, 19th].	D1068–96(D)		Note 22.
31. Kjeldahl Nitrogen—Total, (as N), mg/L:					
Digestion and distillation followed by.	351.3	4500–N_{org} B or C and 4500–NH_3 B [18th, 19th, 20th].	D3590–89(A)		
Titration	351.3		D3590–89(A)		973.48 [3]
Nesslerization	351.3	4500–NH_3 C [18th]	D3590–89(A)		
Electrode	351.3	4500–NH_3 C [19th, 20th] and 4500–NH_3 E [18th].			
Automated phenate colorimetric	351.1			I–4551–78 [8]	
Semi-automated block digestor colorimetric.	351.2		D3590–89(B)	I–4515–91 [45].	
Manual or block digestor potentiometric.	351.4		D3590–89(A)		
Block digester, followed by Auto distillation and Titration, or.					Note 39.
Nesslerization, or					Note 40.
Flow injection gas diffusion					Note 41.
32. Lead—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	239.1	3111 B or C [18th, 19th]	D3559–96(A or B)	I–3399–85	974.27 [3]

AA furnace	239.2	3113 B [18th, 19th]	D3559–96(D)	I–4403–89[51]	
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP[36]			D4190–94		Note 34.
Voltametry[11] or			D3559–96(C)		
Colorimetric (Dithizone)		3500–Pb B [20th] and 3500–Pb D [18th, 19th].			
33. Magnesium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	242.1	3111 B [18th, 19th]	D511–93(B)	I–3447–85	974.27[3]
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP or					Note 34.
Gravimetric		3500–Mg D [18th, 19th]			
34. Manganese-Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	243.1	3111 B [18th, 19th]	D858–95(A or B)	I–3454–85	974.27[3]
AA furnace	243.2	3113 B [18th, 19th]	D858–95(C)		
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP[36], or			D4190–94		Note 34
Colorimetric (Persulfate), or		3500–Mn B [20th] and 3500–Mn D [18th, 19th].			920.203[3]
(Periodate)					Note 23.
35. Mercury—Total,[4] mg/L:					
Cold vapor, manual or	245.1	3112 B [18th, 19th]	D3223–91	I–3462–85	977.22[3]
Automated	245.2				
Oxidation, purge and trap, and cold vapor atomic fluorescence spectrometry (ng/L).	1631E[43]				
36. Molybdenum—Total[4], mg/L; Digestion[4] followed by:					
AA direct aspiration	246.1	3111 D [18th, 19th]		I–3490–85	
AA furnace	246.2	3113 B [18th, 19th]		I–3492–96[47]	
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP					Note 34.
37. Nickel—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	249.1	3111 B or C [18th, 19th]	D1886–90(A or B)	I–3499–85.	
AA furnace	249.2	3113 B [18th, 19th]	D1886–90(C)	I–4503–89[51].	
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50].	
DCP[36], or			D4190–94		Note 34.
Colorimetric (heptoxime)		3500–Ni D [17th].			
38. Nitrate (as N), mg/L:					
Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).	352.1				973.50,[3] 419D,[17] p. 28[9]
39. Nitrate-nitrite (as N), mg/L:					
Cadmium reduction, Manual or.	353.3	4500–NO_3^- E [18th, 19th, 20th].	D3867–99(B).		

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1, 35]	*Standard Methods* [Edition(s)]	ASTM	USGS [2]	Other
Automated, or	353.2	4500–NO_3^- F [18th, 19th, 20th].	D3867–99(A)	I–4545–85.	
Automated hydrazine	353.1	4500–NO_3^- H [18th, 19th, 20th].			
40. Nitrite (as N), mg/L; Spectrophotometric:					
Manual or	354.1	4500–NO_2^- B [18th, 19th, 20th].			Note 25.
Automated (Diazotization)				I–4540–85.	
41. Oil and grease—Total recoverable, mg/L:					
Gravimetric (extraction)	413.1	5520B [18th, 19th, 20th] [38].			
Oil and grease and nonpolar material, mg/L: Hexane extractable material (HEM): n-Hexane extraction and gravimetry.	1664A [42]	5520B [18th, 19th, 20th] [38].			
Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry.	1664A [42].				
42. Organic carbon—Total (TOC), mg/L:					
Combustion or oxidation	415.1	5310 B, C, or D [18th, 19th, 20th].	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	4500–P F [18th, 19th, 20th]		I–4601–85	973.56 [3]
Manual single reagent	365.2	4500–P E [18th, 19th, 20th]	D515–88(A)		973.55 [3]
Manual two reagent	365.3.				
45. Osmium—Total [4], mg/L; Digestion [4] followed by:					
AA direct aspiration, or	252.1	3111 D [18th, 19th].			
AA furnace	252.2.				
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification), or.	360.2	4500–O C [18th, 19th, 20th]	D888–92(A)	I–1575–78 [8]	973.45B [3]
Electrode	360.1	4500–O G [18th, 19th, 20th].	D888–92(B)	I–1576–78 [8].	

47. Palladium—Total,[4] mg/L; Digestion[4] followed by:						
AA direct aspiration, or	253.1	3111 B [18th, 19th]				p. S27 [10]
AA furnace	253.2					p. S28 [10]
DCP						Note 34.
48. Phenols, mg/L:						
Manual distillation [26]	420.1					Note 27.
Followed by:.						
Colorimetric (4AAP) manual, or.	420.1					Note 27.
Automated [19]	420.2.					
49. Phosphorus (elemental), mg/L: Gas-liquid chromatography						Note 28.
50. Phosphorus—Total, mg/L:						
Persulfate digestion followed by.	365.2	4500–P B, 5 [18th, 19th, 20th].				973.55 [3]
Manual or	365.2 or 365.3	4500–P E [18th, 19th, 20th]	D515–88(A)			
Automated ascorbic acid reduction.	365.1	4500–P F [18th, 19th, 20th]		I–4600–85		973.56 [3]
Semi-automated block digestor.	365.4		D515–88(B)	I–4610–91 [48].		
51. Platinum—Total,[4] mg/L: Digestion[4] followed by:						
AA direct aspiration	255.1	3111 B [18th, 19th].				
AA furnace	255.2.					
DCP						Note 34
52. Potassium—Total,[4] mg/L: Digestion[4] followed by:						
AA direct aspiration	258.1	3111 B [18th, 19th]		I–3630–85		973.53 [3]
ICP/AES	200.7 [5]	3120 B [18th, 19th, 20th].				
Flame photometric, or		3500–K B [20th] and 3500–K D [18th, 19th].				
Colorimetric						317 B [17]
53. Residue—Total, mg/L: Gravimetric, 103–105°	160.3	2540 B [18th, 19th, 20th]		I–3750–85.		
54. Residue—filterable, mg/L: Gravimetric, 180°	160.1	2540 C [18th, 19th, 20th]		I–1750–85.		
55. Residue—nonfilterable (TSS), mg/L:						
Gravimetric, 103–105° post washing of residue.	160.2	2540 D [18th, 19th, 20th]		I–3765–85.		
56. Residue—settleable, mg/L: Volumetric, (Imhoff cone), or gravimetric.	160.5	2540 F [18th, 19th, 20th].				
57. Residue—Volatile, mg/L: Gravimetric, 550°	160.4			I–3753–85.		
58. Rhodium-Total,[4] mg/L; Digestion[4] followed by:						
AA direct aspiration, or	265.1	3111 B [18th, 19th].				

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1, 35]	*Standard Methods* [Edition(s)]	ASTM	USGS [2]	Other
AA furnace	265.2.				
59. Ruthenium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration, or	267.1	3111 B [18th, 19th].			
AA furnace	267.2.				
60. Selenium—Total,[4] mg/L; Digestion[4] followed by:					
AA furnace	270.2	3113 B [18th, 19th]	D3859–98(B)	I–4668–98[49].	
ICP/AES,[36] or	200.7[5]	3120 B [18th, 19th, 20th].			
AA gaseous hydride		3114 B [18th, 19th]	D3859–98(A)	I–3667–85.	
61. Silica[37]—Dissolved, mg/L; 0.45 micron filtration followed by:					
Colorimetric, Manual or	370.1	4500–SiO_2 C [20th] and 4500–Si D [18th, 19th].	D859–94	I–1700–85.	
Automated (Molybdosilicate), or.				I–2700–85.	
ICP	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50].	
62. Silver—Total,[4] mg/L: Digestion[4 29] followed by:					
AA direct aspiration	272.1	3111 B or C [18th, 19th]		I–3720–85	974.27,[3] p. 37[9]
AA furnace	272.2	3113 B [18th, 19th]		I–4724–89[51]	
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP					Note 34.
63. Sodium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	273.1	3111 B [18th, 19th]		I–3735–85	973.54[3]
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50]	
DCP, or					Note 34.
Flame photometric		3500 Na B [20th] and 3500 Na D [18th, 19th].			
64. Specific conductance, micromhos/cm at 25 °C:					
Wheatstone bridge	120.1	2510 B [18th, 19th, 20th]	D1125–95(A)	I–2781–85	973.40[3]
65. Sulfate (as SO_4), mg/L:					
Automated colorimetric (barium chloranilate).	375.1.				
Gravimetric	375.3	4500–SO_4^{-2}C or D [18th, 19th, 20th].			925.54[3]
Turbidimetric	375.4		D516–90		426C[30]
66. Sulfide (as S), mg/L:					
Titrimetric (iodine), or	376.1	4500–S^{-2}F [19th, 20th] or 4500–S^{-2}E [18th].		I–3840–85.	

Colorimetric (methylene blue).	376.2	4500–S^{-2}D [18th, 19th, 20th].			
67. Sulfite (as SO_3), mg/L: Titrimetric (iodine-iodate)	377.1	4500–SO_3^{-2}B [18th, 19th, 20th].			
68. Surfactants, mg/L: Colorimetric (methylene blue).	425.1	5540 C [18th, 19th, 20th]	D2330–88.		
69. Temperature, °C: Thermometric	170.1	2550 B [18th, 19th, 20th]			Note 32.
70. Thallium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	279.1	3111 B [18th, 19th].			
AA furnace	279.2.				
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th].			
71. Tin—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	282.1	3111 B [18th, 19th]		I–3850–78[8].	
AA furnace, or	282.2	3113 B [18th, 19th].			
ICP/AES	200.7[5].				
72. Titanium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	283.1	3111 D [18th, 19th].			
AA furnace	283.2.				
DCP					Note 34.
73. Turbidity, NTU: Nephelometric	180.1	2130 B [18th, 19th, 20th]	D1889–94(A)	I–3860–85.	
74. Vanadium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	286.1	3111 D [18th, 19th].			
AA furnace	286.2		D3373–93.		
ICP/AES	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50].	
DCP, or			D4190–94		Note 34.
Colorimetric (Gallic Acid)		3500–V B [20th] and 3500–V D [18th, 19th].			
75. Zinc—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	289.1	3111 B or C [18th, 19th]	D1691–95(A or B)	I–3900–85	974.27,[3] p. 37[9]
AA furnace	289.2.				
ICP/AES[36]	200.7[5]	3120 B [18th, 19th, 20th]		I–4471–97[50].	
DCP,[36] or			D4190–94		Note 34.
Colorimetric (Dithizone) or (Zincon)		3500–Zn E [18th, 19th]. 3500–Zn B [20th] and 3500–Zn F [18th, 19th].			Note 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, 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, 15th ed. (1990).

[4] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983". One (Section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (Section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials may also benefit by this vigorous digestion, however, vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICP/AES as well as determinations for certain elements such as antimony, arsenic, the noble metals, mercury, selenium, silver, tin, and titanium require a modified sample digestion procedure and in all cases the method write-up should be consulted for specific instructions and/or cautions.

NOTE TO TABLE 1B NOTE 4: If the digestion procedure for direct aspiration AA included in one of the other approved references is different than the above, the EPA procedure must be used. Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses, provided the sample solution to be analyzed meets the following criteria:

a. has a low COD (<20)
b. is visibly transparent with a turbidity measurement of 1 NTU or less
c. is colorless with no perceptible odor, and
d. is of one liquid phase and free of particulate or suspended matter following acidification.

[5] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[6] Manual distillation is not required if comparability data on representative effluent samples are on company 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, Apr. 2, 1975. Available from ANSI, 25 West 43rd Street, 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, PO Box 2980, College Station, TX 77840.

[14] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, PO 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, Biocinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

[20] After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 7.6 should be replaced with the buffer 7.6 found in Method 335.2.

[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, PO 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, PO 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] EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.
[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] ''Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals'', CEM Corporation, PO 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 Trichlorotrifluorethane (1,1,2-trichloro-1,2,2-trifluoroethane; CFC–113) extraction solvent when determining Total Recoverable Oil and Grease (analogous to EPA Method 413.1). Only use *n*-hexane extraction solvent when determining Hexane Extractable Material (analogous to EPA Method 1664A). Use of other extraction solvents is strictly prohibited.
[39] Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, PO 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, PO 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, PO 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; Non-polar 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, Virginia 22161.
[43] 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). 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, PO 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.

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)]	ASTM	Other
1. Acenaphthene	610	625, 1625B	610	6440 B [18th, 19th, 20th].	D4657–92	Note 9, p.27.
2. Acenaphthylene	610	625, 1625B	610	6440 B, 6410 B [18th, 19th, 20th].	D4657–92	Note 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].	D4657–92	Note 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)]	ASTM	Other
6. Benzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].		
7. Benzidine		625[5], 1625B	605			Note 3, p.1.
8. Benzo(a)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
9. Benzo(a)pyrene	610,	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
10. Benzo(b)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
11. Benzo(g, h, i)perylene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
12. Benzo(k)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
13. Benzyl chloride						Note 3, p 130: Note 6, p. S102.
14. Benzyl butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
15. Bis(2-chloroethoxy) methane	611	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
16. Bis(2-chloroethyl) ether	611	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
17. Bis(2-ethylhexyl) phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
18. Bromodichloromethane	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].		
19. Bromoform	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].		
20. Bromomethane	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th].		
21. 4-Bromophenylphenyl ether	611	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
22. Carbon tetrachloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th].		Note 3, p. 130.
23. 4-Chloro-3-methylphenol	604	625,1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 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].		Note 3, p. 130.
25. Chloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
26. 2-Chloroethylvinyl ether	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
27. Chloroform:	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		Note 3, p 130.
28. Chloromethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200C [20th] and 6230 B [18th, 19th].		
29. 2-Chloronaphthalene	612	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
30. 2-Chlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 9, p. 27.
31. 4-Chlorophenylphenyl ether	611	625, 1625B		6410 B, [18th, 19th, 20th].		Note 9, p. 27.
32. Chrysene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
33. Dibenzo(a,h)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
34. Dibromochloromethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th].		
35. 1,2-Dichlorobenzene	601, 602, 612	624, 625, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th], 6410 B [18th, 19th, 20th].		Note 9, p 27.
36. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th], 6410 B [18th, 19th, 20th].		Note 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)]	ASTM	Other
37. 1,4-Dichlorobenzene	601, 602, 612	624, 625, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th], 6410 B [18th, 19th, 20th].		Note 9, p. 27.
38. 3,3-Dichlorobenzidine		625, 1625B	605	6410 B [18th, 19th, 20th].		
39. Dichlorodifluoromethane	601			6200 C [20th] and 6230 B [18th, 19th].		
40. 1,1-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
41. 1,2-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
42. 1,1-Dichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
43. trans-1,2-Dichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
44. 2,4-Dichlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 9, p. 27.
45. 1,2-Dichloropropane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
46. cis-1,3-Dichloropropene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
47. trans-1,3-Dichloropropene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].		
48. Diethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.

49. 2,4-Dimethylphenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 9, p. 27.
50. Dimethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
51. Di-n-butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
52. Di-n-octyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
53. 2,3-Dinitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		
54. 2,4-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
55. 2,6-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
56. Epichlorohydrin						Note 3, p. 130; Note 6, p. S102.
57. Ethylbenzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	D4657–92	Note 9, p. 27.
58. Fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
59. Fluorene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
60. 1,2,3,4,6,7,8-Heptachloro- dibenzofuran		1613B				
61. 1,2,3,4,7,8,9-Heptachloro- dibenzofuran		1613B				
62. 1,2,3,4,6,7,8-Heptachloro- dibenzo-p-dioxin		1613B				
63. Hexachlorobenzene	612	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
64. Hexachlorobutadiene	612	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
65. Hexachlorocyclopentadiene	612	[5]625, 1625B		6410 [18th, 19th, 20th]		Note 9, p. 27.
66. 1,2,3,4,7,8-Hexachloro- dibenzofuran		1613B.				
67. 1,2,3,6,7,8-Hexachloro- dibenzofuran		1613B.				
68. 1,2,3,7,8,9-Hexachloro- dibenzofuran		1613B.				
69. 2,3,4,6,7,8-Hexachloro- dibenzofuran		1613B.				
70. 1,2,3,4,7,8-Hexachloro- dibenzo-p-dioxin		1613B.				
71. 1,2,3,6,7,8-Hexachloro- dibenzo-p-dioxin		1613B.				
72. 1,2,3,7,8,9-Hexachloro- dibenzo-p-dioxin		1613B.				
73. Hexachloroethane	616	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
74. Ideno(1,2,3-cd) pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
75. Isophorone	609	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27.
76. Methylene chloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th].		Note 3, p. 130.
77. 2-Methyl-4,6-dinitrophenol	604	625, 1625B		6420 B, 6410 B [18th, 19th, 20th].		Note 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)]	ASTM	Other
78. Naphthalene	610	625, 1625B	610	6440 B, 6410 B [18th, 19th, 20th].		Note 9, p. 27.
79. Nitrobenzene	609	625, 1625B		6410 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27.
80. 2-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 9, p. 27
81. 4-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th].		Note 9, p. 27
82. N-Nitrosodimethylamine	607	625[5], 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27
83. N-Nitrosodi-n-propylamine	607	625, 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27
84. N-Nitrosodiphenylamine	607	625[5], 1625B		6410 B [18th, 19th, 20th].		Note 9, p. 27
85. Octachlorodibenzofuran		1613B.				
86. Octachlorodibenzo-p-dioxin		1613B.				
87. 2,2'-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether].	611	625, 1625B		6410 B [18th, 19th, 20th].		
88. PCB–1016	608	625		6410 B [18th, 19th, 20th].		Note 3, p. 43
89. PCB–1221	608	625		6410 B [18th, 19th, 20th].		Note 3, p. 43
90. PCB–1232	608	625		6410 B [18th, 19th, 20th].		Note 3, p. 43
91. PCB–1242	608	625		6410 B [18th, 19th, 20th].		Note 3, p. 43
92. PCB–1248	608	625.				
93. PCB–1254	608	625		6410 B [18th, 19th, 20th].		Note 3, p. 43
94. PCB–1260	608	625		6410 B, 6630 B [18th, 19th, 20th].		Note 3, p. 43
95. 1,2,3,7,8-Pentachloro- dibenzofuran		1613B.				
96. 2,3,4,7,8-Pentachloro- dibenzofuran		1613B.				
97. 1,2,3,7,8,-Pentachlorodibenzo-p-dioxin		1613B.				
98. Pentachlorophenol	604	625, 1625B		6410 B, 6630 B [18th, 19th, 20th].		Note 3, p. 140; Note 9, p. 27
99. Phenanthrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th].	D4657–92	Note 9, p. 27
100. Phenol	604	625, 1625B		6420 B, 6410 B [18th, 19th, 20th].		Note 9, p. 27
101. Pyrene	610	625, 1625B	610	6440 B, 6410 B D4675–92 [18th, 19th, 20th].	D4675–92	Note 9, p. 27

102. 2,3,7,8-Tetrachloro- dibenzofuran	.....	1613B.			.....	
103. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	.....	613, 1613B.			.....	
104. 1,1,2,2-Tetrachloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	Note 3, p. 130
105. Tetrachloroethene	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	Note 3, p. 130
106. Toluene	602	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th].	.....	
107. 1,2,4-Trichlorobenzene	612	625, 1625B	.....	6410 B [18th, 19th, 20th].	.....	Note 3, p. 130; Note 9, p. 27.
108. 1,1,1-Trichloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	
109. 1,1,2-Trichloroethane	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	Note 3, p. 130
110. Trichloroethene	601	624, 1624B	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	
111. Trichlorofluoromethane	601	624	.....	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th].	.....	
112. 2,4,6-Trichlorophenol	604	625, 1625B	.....	6420 B, 6410 B [18th, 19th, 20th].	.....	Note 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].	.....	

Table IC notes:

[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.

[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 and cannot be reported to demonstrate regulatory compliance.

NOTE: These warning limits are promulgated as an "interim final action with a request for comments."

[8] "Organochlorine Pesticides and PCBs in Wastewater Using Empore TM 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.

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

Parameter	Method	EPA [2, 7]	*Standard Methods* 18th, 19th, 20th Ed.	ASTM	Other
1. Aldrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B		
2. Ametryn	GC				Note 3, p. 83; Note 6, p S68.
3. Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4. Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC				Note 3, p. 83; Note 6, p. S68; Note 9.
6. Azinphos methyl	GC				Note 3, p. 25; Note 6, p. S51.
7. Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8. α-BHC	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 8.
	GC/MS	625 [5]	6410 B.		
9. β-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	625 [5]	6410 B.		
10. δ-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	625 [5]	6410 B.		
11. γ-BHC (Lindane)	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
12. Captan	GC		6630 B	D3086–90	Note 3, p. 7.
13. Carbaryl	TLC				Note 3, p. 94, Note 6, p. S60.
14. Carbophenothion	GC				Note 4, p. 27; Note 6, p. S73.
15. Chlordane	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
16. Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		6640 B		Note 3, p. 115; Note 4, p. 40.
18. 4,4′-DDD	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
19. 4,4′-DDE	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
20. 4,4′-DDT	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
21. Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC				Note 3, p. 25; Note 6, p. S51.
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 27; Note 6, p. S51.
24. Dicamba	GC				Note 3, p. 115.
25. Dichlofenthion	GC				Note 4, p. 27; Note 6, p. S73.
26. Dichloran	GC		6630 B & C		Note 3, p. 7.

27. Dicofol	GC	608	6630 B & C	D3086–90.	Note 3, p. 7; Note 4, p. 27; Note 8.
28. Dieldrin	GC	608	6630 B & C		
	GC/MS	625	6410 B.		
29. Dioxathion	GC				Note 4, p. 27; Note 6, p. S73.
30. Disulfoton	GC				Note 3, p. 25; Note 6 p. S51.
31. Diuron	TLC				Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625 [5]	6410 B.		
33. Endosulfan II	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 8.
	GC/MS	625 [5]	6410 B.		
34. Endosulfan Sulfate	GC	608	6630 C		Note 8.
	GC/MS	625	6410 B.		
35. Endrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625 [5]	6410 B.		Note 8.
36. Endrin aldehyde	GC	608			
	GC/MS	625.			
37. Ethion	GC				Note 4, p. 27; Note 6, p. S73.
38. Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39. Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC	608	6630 B & C	3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410 B.		
41. Heptachlor epoxide	GC	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 6, p. S73; Note 8.
	GC/MS	625	6410 B.		Note 4, p. 27; Note 6, p. S73.
42. Isodrin	GC				Note 3, p. 104; Note 6, p. S64.
43. Linuron	GC				Note 3, p. 25; Note 4, p. 27; Note 6, p. S51
44. Malathion	TLC				Note 3, p. 94; Note 6, p. S60.
45. Methiocarb	GC		6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 27; Note 8.
46. Methoxychlor	TLC				Note 3, p. 94; Note 6, p. S60.
47. Mexacarbate	GC		6630 B & C		Note 3, p. 7; Note 4, p. 27 .
48. Mirex	TLC				Note 3, p. 104; Note 6, p. S64.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
50. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
51. Nuburon	GC		6630 C		Note 3, p. 25; Note 4, p. 27.
52. Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 27.
53. Parathion ethyl	GC		6630 B & C		Note 3, p. 7.
54. PCNB	GC			D3086–90	Note 4, p. 27.
55. Perthane	GC				Note 3, p. 83; Note 6, p. S68; Note 9.
56. Prometron	GC				Note 3, p. 83; Note 6, p. S68; Note 9.
57. Prometryn	GC				Note 3, p. 83; Note 6, p. S68; Note 9.
58. Propazine	TLC				Note 3, p. 104; Note 6, p. S64.
59. Propham	TLC				Note 3, p. 94; Note 6, p. S60.
60. Propoxur	TLC				Note 3, p. 83; Note 6, p. S68.
61. Secbumeton	TLC				Note 3, p. 104; Note 6, p. S64.
62. Siduron	GC				Note 3, p. 83; Note 6, p. S68; Note 9.
63. Simazine	GC		6630 B & C		Note 3, p. 7.
64. Strobane	TLC				Note 3, p. 104; Note 6, p. S64.
65. Swep	GC		6640 B		Note 3, p. 115; Note 4, p. 40.
66. 2,4,5-T					

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

Parameter	Method	EPA[2, 7]	*Standard Methods* 18th, 19th, 20th Ed.	ASTM	Other
67. 2,4,5-TP (Silvex)	GC		6640 B		Note 3, p. 115; Note 4, p. 40.
68. Terbuthylazine	GC				Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC	608	6630 B & C	D3086—90	Note 3, p. 7; Note 4, p. 27; Note 8.
	GC/MS	625	6410B.		
70. Trifluralin	GC		6630 B		Note 3, p. 7; Note 9.

Table ID notes:

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

[2] The full text of Methods 608 and 625 are given at Appendix A. "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 and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

NOTE: These warning limits are promulgated as an "Interim final action with a request for comments."

[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 PROCEDURES

Parameter and units	Method	Reference (method number or page)			
		EPA[1]	*Standard Methods* 18th, 19th, 20th Ed.	ASTM	USGS[2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900	7110 B	D1943–90	pp. 75 and 78[3]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	D1943–90	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	D1890–90	pp. 75 and 78[3]
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	D1890–90	p. 79
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	D2460–90	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500Ra C	D3454–91	p. 81

TABLE 1E NOTES:

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

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

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

TABLE IF—LIST OF APPROVED METHODS FOR PHARMACEUTICAL POLLUTANTS

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.

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, IE,and IF are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources. The full texts of all the test procedures cited are available for inspection at the National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, 26 West Martin Luther King Dr., Cincinnati, OH 45268 and 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 from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB–290329/AS. Cost: $36.95. Table IA, 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: Amer. Publ. Hlth. Assoc., 1015 15th Street, NW., Washington, DC 20005. Table IA, Note 4. Tables IB, IC, ID, IE.

(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) Annual Book of ASTM Standards, Water, and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994, 1996, 1999, and Volume 11.02, 2000 in 40 CFR 136.3, Tables IA, IB, IC, ID, and IE.

(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. Cost: $18.00. Table IA, Note 5.

(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) ''Official Methods of Analysis of the Association of Official Analytical

Chemicals'', Methods manual, 15th Edition (1990). Price: $240.00. Available from: The Association of Official Analytical Chemists, 2200 Wilson Boulevard, Suite 400, Arlington, VA 22201. Table IB, Note 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 from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108488. Table IA, Note 29.

(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 from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108489. Table IA, Note 30.

(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 from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Pub. No. PB2002–108490. Table IA, Note 31.

(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.

(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.

(54) USEPA. 2002. 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 D.C. September 2002, EPA–821–R–02–020. Available at NTIS, PB2003–100125. Table IA, Note 20.

(55) USEPA. 2002. 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 D.C. September 2002, EPA–821–R–02–021. Available at NTIS, PB2003–100126. Table IA, Note 24.

(56) USEPA. 2002. 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 September 2002, EPA–821–R–02–023. Available at NTIS, PB2003–100128. Table IA, Note 21.

(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 IA, Note 22.

(58) USEPA. 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 D.C. September 2002, EPA 821–R–02–024. Available from NTIS, PB2003–100129. Table IA, Note 22.

(59) USEPA. 2002. 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 D.C. September 2002, EPA–821–R–02–022. Available from NTIS, PB2003–100127. Table IA, Note 25.

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

Available from NTIS, PB2002–108709. Table IA, Note 26.

(61) USEPA. 2001. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC April 2001, EPA–821–R–01–025. Available from NTIS, PB2002–108710. Table IA, Note 27.

(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.

(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 the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. 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 Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

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

Parameter No./name	Container [1]	Preservation [2, 3]	Maximum holding time [4]
Table IA—Bacteria Tests:			
1–5 Coliform, total, fecal, and *E. coli*	PP, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5].	6 hours.
6 Fecal *streptococci*	PP, G	Cool, <10° 0.0008% $Na_2S_2O_3$[5]	6 hours.
7 *Enterococci*	PP, G	Cool, <10° 0.0008% $Na_2S_2O_3$[5]	6 hours.
Table IA—Protozoa Tests:			
8 *Cryptosporidium*	LDPE	0–8 °C	96 hours. [17]
9 *Giardia*	LDPE	0–8 °C	96 hours. [17]
Table IA—Aquatic Toxicity Tests:			
6–10 Toxicity, acute and chronic	P,G	Cool, 4 °C [16]	36 hours.

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

Parameter No./name	Container [1]	Preservation [2, 3]	Maximum holding time [4]
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	....do	Do.
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz.	HNO_3 TO pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	....do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23–24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid [5].	14 days.[6]
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
Metals:[7]			
18. Chromium VI [7]	P, G	Cool, 4 °C	24 hours.
35. Mercury [17]	P, G	HNO_3 to pH<2	28 days.
3, 5–8, 12,13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals except boron, chromium VI and mercury [7].	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H_2SO4 or H_3PO4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winkler	....do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	....do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	....do	7 days.
56. Residue, Settleable	P, G	....do	48 hours.
57. Residue, volatile	P, G	....do	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	....do	Do.
65. Sulfate	P, G	....do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P ,G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests [8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5].	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	....do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] HCl to pH2[9].	Do.
3, 4. Acrolein and acrylonitrile	....do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] adjust pH to 4–5[10].	Do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11].	G, Teflon-lined cap..	Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction; 40 days after extraction.
7, 38. Benzidines [11]	....do	....do	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters [11]	....do	Cool, 4 °C	7 days until extraction; 40 days after extraction.

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

Parameter No./name	Container [1]	Preservation [2, 3]	Maximum holding time [4]
82–84. Nitrosamines [11 14]	.....do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
88–94. PCBs [11]	.....do	Cool, 4 °C	Do.
54, 55, 75, 79. Nitroaromatics and isophorone [11].	.....do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11].	.....do	.....do	Do.
15, 16, 21, 31, 87. Haloethers [11]	.....do	Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5]	Do.
29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons [11].	.....do	Cool, 4 °C	Do.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs [11].			
aqueous: field and lab preservation.	G	Cool, 0–4 °C, pH<9, 0.008% $Na_2S_2O_3$ [5].	1 year.
Solids, mixed phase, and tissue: field preservation..	.....do	Cool, <4 °C	7 days.
Solids, mixed phase, and tissue: lab preservation.	.....do	Freeze, < −10 °C	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides [11]	.....do	Cool, 4°C, pH 5–9 [15]	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months.

Table II Notes

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (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 analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term ''analyze immediately'' usually means within 15 minutes or less of sample collection.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

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

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

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

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

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

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ 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 4C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

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

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987; 55 FR 24534, June 15, 1990; 55 FR 33440, Aug. 15, 1990; 56 FR 50759, Oct. 8, 1991; 57 FR 41833, Sept. 11, 1992; 58 FR 4505, Jan. 31, 1994; 60 FR 17160, Apr. 4, 1995; 60 FR 39588, 39590, Aug. 2, 1995; 60 FR 44672, Aug. 28, 1995; 60 FR 53542, 53543, Oct. 16, 1995; 62 FR 48403, 48404, Sept. 15, 1997; 63 FR 50423, Sept. 21, 1998; 64 FR 4978, Feb. 2, 1999; 64 FR 10392, Mar. 4, 1999; 64 FR 26327, May 14, 1999; 64 FR 30433, 30434, June 8, 1999; 64 FR 73423, Dec. 30, 1999; 66 FR 32776, June 18, 2001; 67 FR 65226, Oct. 23, 2002; 67 FR 65886, Oct. 29, 2002; 67 FR 69971, Nov. 19, 2002; 68 FR 43278, July 21, 2003; 68 FR 54934, Sept. 19, 2003; 69 FR 18803, Apr. 9, 2004]

§ 136.4 Application for alternate test procedures.

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

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

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

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

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

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

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

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

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

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

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

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

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

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

(b) Within thirty days of receipt of an application, the Director will forward such application proposed by the responsible person or firm making the discharge, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application,