

§ 136.3

40 CFR Ch. I (7–1–23 Edition)

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, IG, and IH of this section. The methods listed in Tables IA, IB, IC, ID, IE, IF, IG, and IH are incorporated by reference, see paragraph (b) of this section, with the exception of EPA Methods 200.7, 601–613, 624.1, 625.1, 1613, 1624, and 1625. The full texts of Methods 601–613, 624.1, 625.1, 1613, 1624, and 1625 are printed in appendix A of this part, and the full text of Method 200.7 is printed in appendix C of this part. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part. In the event of a conflict between the reporting requirements of 40 CFR parts 122 and 125 and any reporting requirements associated with

the methods listed in these tables, the provisions of 40 CFR parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full texts of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG, and IH. The date after the method number indicates the latest editorial change of the method. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, IE, IF, IG, and IH or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under certain circumstances (paragraph (c) of this section, § 136.5(a) through (d) or 40 CFR 401.13,) other additional or alternate test procedures may be used.

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

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria					
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN), 5 tube, 3 dilution, or	p. 132. ³ 1680, ^{11 15} 1681 ^{11 20}	9221 E-2014.		
2. Coliform (fecal), number per 100 mL.	Membrane filter (MF) ^{2 5} , single step MPN, 5 tube, 3 dilution, or	p. 124. ³ p. 132. ³	9222 D-2015 ²⁹ 9221 E-2014; 9221 F-2014 ³³	B-0050-85 ⁴	Colilert-18 [®] , ^{13 18 28}
3. Coliform (total), number per 100 mL.	Multiple tube/multiple well, or				
	MF ^{2 5} , single step ⁵	p. 124. ³	9222 D-2015 ²⁹		
	MPN, 5 tube, 3 dilution, or	p. 114. ³	9221 B-2014.		
4. <i>E. coli</i> , number per 100 mL	MF ^{2 5} , single step or two step	p. 108. ³	9222 B-2015 ³⁰	B-0025-85 ⁴	
	MF ^{2 5} , with enrichment	p. 111. ³	9222 B-2015 ³⁰		
	MPN ^{6 8 16} multiple tube, or		9221 B2014/9221 F-2014 ^{12 14 33}		
	multiple tube/multiple well, or		9223 B-2016 ¹³	991.15 ¹⁰	Colilert [®] 13 18 Colilert-18 [®] 13 17 18
5. Fecal streptococci, number per 100 mL.	MF ^{2 5 6 7 8} , two step, or		9222 B-2015/9222 I-2015 ³¹		m-CollBlue24 [®] , ¹⁹
	Single step	1603 ²¹			
	MPN, 5 tube, 3 dilution, or	p. 139. ³	9230 B-2013.		
6. Enterococci, number per 100 mL.	MF ² , or	p. 136. ³	9230 C-2013 ³²	B-0055-85 ⁴	
	Plate count	p. 143. ³			
	MPN, 5 tube, 3 dilution, or	p. 139. ³	9230 B-2013.		
	MPN ^{6 8} , multiple tube/multiple well, or		9230 D-2013	D6503-99 ⁹	Enterolert [®] , ^{13 23}
7. <i>Salmonella</i> , number per gram dry weight ¹¹ .	MF ^{2 5 6 7 8} single step or	1600 ²⁴	9230 C-2013 ³²		
	Plate count	p. 143. ³			
	MPN multiple tube	1682 ²²			
Aquatic Toxicity					
8. Toxicity, acute, fresh water organisms, LC ₅₀ , percent effluent.	Water flea, <i>Cladocera</i> , <i>Ceriodaphnia dubia</i> acute.	2002.0 ²⁵			

TABLE 1A—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE—Continued

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
9. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC ₅₀ , percent effluent.	Water fleas, Cladocerans, <i>Daphnia pulex</i> and <i>Daphnia magna</i> acute.	2021.0 ²⁵ .			
	Fish, Fathead minnow, <i>Pimephales promelas</i> , and Bannertin shiner, <i>Cyprinella leedsi</i> , acute.	2000.0 ²⁵ .			
	Fish, Rainbow trout, <i>Oncorhynchus mykiss</i> , and brook trout, <i>Salvelinus fontinalis</i> , acute.	2019.0 ²⁵ .			
	Mysid, <i>Mysidopsis bahia</i> , acute	2007.0 ²⁵ .			
10. Toxicity, chronic, fresh water organisms, NOEC or IC ₂₅ , percent effluent.	Fish, Sheepshead minnow, <i>Cyprinodon variegatus</i> , acute.	2004.0 ²⁵ .			
	Fish, Silverside, <i>Menidia beryllina</i> , <i>Menidia menidia</i> , and <i>Menidia peninsulae</i> , acute..	2006.0 ²⁵ .			
	Fish, Fathead minnow, <i>Pimephales promelas</i> , larval survival and growth.	1000.0 ²⁶ .			
	Fish, Fathead minnow, <i>Pimephales promelas</i> , embryo-larval survival and teratogenicity.	1001.0 ²⁶ .			
11. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC ₂₅ , percent effluent.	Water flea, Cladoceran, <i>Ceriodaphnia dubia</i> , survival and reproduction.	1002.0 ²⁶ .			
	Green alga, <i>Selenastrum capricornutum</i> , growth.	1003.0 ²⁶ .			
	Fish, Sheepshead minnow, <i>Cyprinodon variegatus</i> , larval survival and growth.	1004.0 ²⁷ .			
	Fish, Sheepshead minnow, <i>Cyprinodon variegatus</i> , embryo-larval survival and teratogenicity.	1005.0 ²⁷ .			
	Fish, Inland silverside, <i>Menidia beryllina</i> , larval survival and growth.	1006.0 ²⁷ .			

Mysid, <i>Mysidopsis bahia</i> , survival, growth, and fecundity.	1007.0 ²⁷ .		
Sea urchin, <i>Arbacia punctulata</i> , fertilization.	1008.0 ²⁷ .		

Table 1A notes:

- 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 Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA/600/8-78/017, 1978, U.S. EPA.
- ⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, 1989, USGS.
- ⁵ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.
- ⁶ Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.
- ⁷ When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
- ⁸ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current *Standard Methods for the Examination of Water and Wastewater* or EPA alternate test procedure (ATP) guidelines.
- ⁹ Annual Book of ASTM Standards-Water and Environmental Technology, Section 11.02, 2000, 1999, 1996, ASTM International.
- ¹⁰ Official Methods of Analysis of AOAC International, 16th Edition, 4th Revision, 1998, AOAC International.
- ¹¹ Recommended for enumeration of target organism in sewage sludge.
- ¹² The multiple-tube fermentation test is used in 9221B.2-2014. 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.
- ¹⁴ After prior enrichment in a presumptive medium for total coliform using 9221B.2-2014, 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-2014. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.
- ¹⁵ Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium, EPA-821-R-14-009, September 2014, U.S. EPA.
- ¹⁶ Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Coli-Test[®] may be enumerated with the multiple-well procedures, Quanti-Tray[®] or Quanti-Tray[®]/2000 and the MPN calculated from the table provided by the manufacturer.
- ¹⁷ Coli-Test[®] is an optimized formulation of the Coli-Test[®] 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 Coli-Test[®] test and is recommended for marine water samples.
- ¹⁸ Descriptions of the Coli-Test[®], Coli-Test[®]-18[®], Quanti-Tray[®], and Quanti-Tray[®]/2000 may be obtained from IDEXX Laboratories, Inc.
- ¹⁹ A description of the mColiBlue24[®] test is available from Hach Company.
- ²⁰ Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using A-1 Medium, EPA-821-R-06-013, July 2006, U.S. EPA.
- ²¹ Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified Membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC), EPA-821-R-14-010, September 2014, U.S. EPA.
- ²² Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium, EPA-821-R-14-012, September 2014, U.S. EPA.
- ²³ A description of the Enterolert[®] test may be obtained from IDEXX Laboratories Inc.
- ²⁴ Method 1600: Enterococci in Water by Membrane Filtration Using Membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA-821-R-14-011, September 2014, U.S. EPA.

²⁵ Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA-821-R-02-012, Fifth Edition, October 2002, U.S. EPA; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821-R-02-012-ES, December 2016.

²⁶ Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, EPA-821-R-02-013, Fourth Edition, October 2002, U.S. EPA; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821-R-02-012-ES, December 2016.

²⁷ Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, EPA-821-R-02-014, Third Edition, October 2002, U.S. EPA; and U.S. EPA Whole Effluent Toxicity Methods Errata Sheet, EPA 821-R-02-012-ES, December 2016.

²⁸ To use Colilert-18® to assay for fecal coliforms, the incubation temperature is 44.5 ± 0.2 °C, and a water bath incubator is used.

²⁹ On a monthly basis, at least ten blue colonies from positive samples must be verified using Lauryl Tryptose Broth and EC broth, followed by count adjustment based on these results; and representative non-blue colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

³⁰ On a monthly basis, at least ten sheen colonies from positive samples must be verified using lauryl tryptose broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using lauryl tryptose broth. Where possible, verifications should be done from randomized sample sources.

³¹ Subject coliform positive samples determined by 9222 B-2015 or other membrane filter procedure to 9222 I-2015, using NA-MUG media.

³² Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional. As per the Errata to the 23rd Edition of *Standard Methods for the Examination of Water and Wastewater*, Growth on a BHI agar plate incubated at 10 ± 0.5 °C for 48 ± 3 h is further verification that the colony belongs to the genus *Enterococcus*.

³³ 9221 F.2-2014 allows for simultaneous detection of *E. coli* and thermotolerant fecal coliforms by adding inverted vials to EC-MUG; the inverted vials collect gas produced by thermotolerant fecal coliforms.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
1. Acidity, as CaCO ₃ , mg/L.	Electrometric endpoint or phenolphthalein endpoint.		2310 B-2011	D1067-16	I-1020-85. ²
2. Alkalinity, as CaCO ₃ , mg/L.	Electrometric or Colorimetric titration to pH 4.5, Manual.		2320 B-2011	D1067-16	973.43, ³ I-1030-85. ²
3. Aluminum—Total, ⁴ mg/L.	Automatic	310.2 (Rev. 1974) ¹ ..			I-2030-85. ²
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶		3111 D-2011 or 3111 E-2011, 3113 B-2010.		I-3051-85. ²
	AA furnace				
	STGFAA	200.9, Rev. 2.2 (1994)/		D1976-12	I-4471-97. ⁵⁰
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994), 200.8, Rev. 5.4 (1994).	3120 B-2011	D5673-16	993.14, ³ I-4472-97. ⁸¹
	ICP/MS		3125 B-2011	D4190-15	See footnote. ³⁴
4. Ammonia (as N), mg/L.	Direct Current Plasma (DCP) ³⁶				
	Colorimetric (Eriochrome cyanine R)		3500-AI B-2011, 4500-NH ₃ B-2011 ..		973.49. ³
	Manual distillation ⁶ or gas diffusion (pH > 11), followed by any of the following:	350.1, Rev. 2.0 (1993).			
	Nesslerization			D1426-15 (A)	973.49, ³ I-3520-85. ²

5. Antimony—Total, ⁴ mg/L.	Titration		4500-NH ₃ C-2011. 4500-NH ₃ D-2011 or E-2011. 4500-NH ₃ F-2011 ..	D1426-15 (B).	See footnote. ⁶⁰
	Electrode				
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods.				
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction-based methods.	350.1, ³⁰ Rev. 2.0 (1993).	4500-NH ₃ G-2011 4500-NH ₃ H-2011.		I-4523-85, ² I-2522-90. ⁸⁰
	Automated electrode				See footnote. ⁷
	Ion Chromatography			D6919-17.	
	Automated gas diffusion, followed by conductivity cell analysis.				Timberline Ammonia-001. ⁷⁴
	Automated gas diffusion followed by fluorescence detector analysis.				FI/Ab100. ⁸²
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶		3111 B-2011. 3113 B-2010.		
6. Arsenic-Total, ⁴ mg/L.	AA furnace	200.9, Rev. 2.2 (1994).	3120 B-2011	D1976-12.	
	STGFAA	200.5, Rev 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3125 B-2011	D5673-16	993.14, ³ I-4472-97. ⁸¹
	ICP/AES ³⁶	200.8, Rev. 5.4 (1994).			
	ICP/MS	206.5 (issued 1978) ¹ .	3114 B-2011 or 3114 C-2011. 3113 B-2010	D2972-15 (B)	I-3062-85. ²
	Digestion, ⁴ followed by any of the following:			D2972-15 (C)	I-4063-98. ⁴⁹
	AA gaseous hydride				
	AA furnace	200.9, Rev. 2.2 (1994).	3120 B-2011	D1976-12.	
	STGFAA	200.5, Rev 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3125 B-2011	D5673-16	993.14, ³ I-4020-05. ⁷⁰
	ICP/AES ³⁶	200.8, Rev. 5.4 (1994).	3500-As B-2011	D2972-15 (A)	I-3060-85. ²
	ICP/MS				
7. Barium-Total, ⁴ mg/L.	Colorimetric (SDDC)		3111 D-2011		I-3084-85. ²
	Digestion, ⁴ followed by any of the following:		3113 B-2010	D4382-18.	
	AA direct aspiration ³⁶				
	AA furnace				

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
8. Beryllium—Total, ⁴ mg/L.	ICP/AES ³⁶	200.5, Rev 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3120 B–2011		I–4471–97. ⁵⁰
	ICP/MS		3125 B–2011	D5673–16	993.14, ³ I–4472– 97. ⁸¹
	DCP ³⁶				See footnote. ³⁴
	Digestion, ⁴ followed by any of the fol- lowing:				
	AA direct aspiration		3111 D–2011 or 3111 E–2011.	D3645–15 (A)	I–3095–85. ²
9. Biochemical oxy- gen demand (BOD ₅), mg/L.	AA furnace		3113 B–2010	D3645–15 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).		D1976–12	I–4471–97. ⁵⁰
	ICP/AES	200.5, Rev 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3120 B–2011		
	ICP/MS		3125 B–2011	D5673–16	993.14, ³ I–4472– 97. ⁸¹
	DCP			D4190–15	See footnote. ³⁴
10. Boron—Total, ³⁷ mg/L.	Colorimetric (aluminon)		See footnote. ⁶¹ .		973.44, ³ p. 17, ⁹ I– 1578–78. ⁸ See footnote. ^{10, 63}
	Dissolved Oxygen Depletion		5210 B–2016 ⁸⁵		I–3112–85. ²
	Colorimetric (curcumin)		4500–B B–2011		
	ICP/AES	200.5, Rev 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3120 B–2011	D1976–12	I–4471–97. ⁵⁰
	ICP/MS		3125 B–2011	D5673–16	993.14, ³
11. Bromide, mg/L	DCP			D4190–15	S7ee footnote. ³⁴
	Electrode			D1246–16	I–1125–85. ²
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).	4110 B–2011, C– 2011, D–2011.	D4327–17	993.30, ³ I–2057– 85. ⁷⁹
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴
	Digestion, ⁴ followed by any of the fol- lowing:				
12. Cadmium— Total, ⁴ mg/L.					

AA direct aspiration ³⁶		3111 B-2011 or 3111 C-2011.	D3557-17 (A or B) ..	974.27, ³ p. 37, ⁹ I-3135-85 ² or I-3136-85 ² I-4138-89. ⁵¹
AA furnace			D3557-17 (D)	
STGFAA				
ICP/AES ³⁶	200.9, Rev. 2.2 (1994).	3113 B-2010	D1976-12	I-1472-85 ² or I-4471-97. ⁵⁰
ICP/MS	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011	D5673-16	993.14, ³ I-4472-97. ⁸¹
ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011	D4190-15	See footnote. ³⁴
DCP ³⁶			D3557-17 (C).	
Voltammetry ¹¹		3500-Cd-D-1990.		
Colorimetric (Dithizone)				
Digestion, ⁴ followed by any of the following:				
AA direct aspiration		3111 B-2011	D511-14 (B)	I-3152-85. ²
ICP/AES	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011		I-4471-97. ⁵⁰
ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011	D5673-16	993.14, ³
DCP				See footnote. ³⁴
Titrimetric (EDTA)		3500-Ca B-2011	D511-14 (A).	
Ion Chromatography			D6919-17.	
Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B-2016 ⁸⁵		See footnote. ³⁵ 63
Titrimetric	410.3 (Rev. 1978) ¹ ..	5220 B-2011 or C-2011.	D1252-06(12) (A)	973.46, ³ p. 17, ⁹ I-3560-85. ²
Spectrophotometric, manual or automatic	410.4, Rev. 2.0 (1993).	5220 D-2011	D1252-06(12) (B)	See footnote. ¹³ 14 83, I-3561-85. ²
Titrimetric: (silver nitrate)		4500-Cl- B-2011	D512-12 (B)	I-1183-85. ²
(Mercuric nitrate)		4500-Cl- C-2011	D512-12 (A)	973.51, ³ I-1184-85. ²
Colorimetric: manual				I-1187-85. ²
Automated (ferrocyanide)		4500-Cl- E-2011		I-2187-85. ²
Potentiometric Titration		4500-Cl- D-2011.		
Ion Selective Electrode			D512-12 (C).	
Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B-2011 or 4110 C-2011.	D4327-17	993.30, ³ I-2057-90. ⁵¹

13. Calcium—Total,⁴ mg/L.

14. Carbonaceous biochemical oxygen demand (CBOD₅), mg/L¹².

15. Chemical oxygen demand (COD), mg/L.

16. Chloride, mg/L

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
17. Chlorine—Total residual, mg/L.	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴
	Amperometric direct		4500–Cl D–2011	D1253–14.	
	Amperometric direct (low level)		4500–Cl E–2011.		
	Iodometric direct		4500–Cl B–2011.		
	Back titration ether end-point ¹⁵		4500–Cl C–2011.		
17A. Chlorine-Free Available, mg/L.	DPD–FAS		4500–Cl F–2011.		See footnote. ¹⁶
	Spectrophotometric, DPD		4500–Cl G–2011.		
	Electrode				
	Amperometric direct		4500–Cl D–2011	D1253–14.	
	Amperometric direct (low level)		4500–Cl E–2011.		
18. Chromium VI dissolved, mg/L.	DPD–FAS		4500–Cl F–2011.		I–1232–85. ² 993.23. ³
	Spectrophotometric, DPD		4500–Cl G–2011.		
	0.45-micron filtration followed by any of the following:				
	AA chelation-extraction		3111 C–2011		
	Ion Chromatography	218.6, Rev. 3.3 (1994).	3500–Cr C–2011	D5257–17	
19. Chromium—Total, ⁴ mg/L.	Colorimetric (diphenyl-carbazide)		3500–Cr B–2011	D1687–17 (A)	I–1230–85. ²
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶		3111 B–2011	D1687–17 (B)	
	AA chelation-extraction		3111 C–2011.		
	AA furnace		3113 B–2010	D1687–17 (C)	
20. Cobalt—Total, ⁴ mg/L.	STGFAA	200.9, Rev. 2.2 (1994).			974.27, ³ I–3236–85. ² I–3233–93. ⁴⁶
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	
	DCP ³⁶				
	Colorimetric (diphenyl-carbazide)		3500–Cr B–2011.	D4190–15	
20. Cobalt—Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the following:				993.14, ³ I–4020–05 ⁷⁰ I–4472–97. ⁸¹ See footnote. ³⁴
	AA direct aspiration		3111 B–2011 or 3111 C–2011.	D3558–15 (A or B) ..	
	AA furnace		3113 B–2010	D3558–15 (C)	
					
					

STGFAA	200.9, Rev. 2.2 (1994).		3120 B-2011	D1976-12	I-4471-97. ⁵⁰
ICP/AES	200.7, Rev. 4.4 (1994).		3125 B-2011	D5673-16	993.14, ³ I-4020-05, ⁷⁰ I-4472-97. ⁸¹
ICP/MS	200.8, Rev. 5.4 (1994).		2120 F-2011 ⁷⁸ .	D4190-15	See footnote. ³⁴
DCP					
Colorimetric (ADMI)			2120 B-2011		I-1250-85. ²
Platinum cobalt visual comparison					See footnote ¹⁸
Spectrophotometric			3111 B-2011 or 3111 C-2011.	D1688-17 (A or B)	974.27, ³ p. 37, ⁹ I-3270-85. ² or I-3271-85. ²
AA direct aspiration ³⁶			3113 B-2010	D1688-17 (C)	I-4274-89. ⁵¹
AA furnace	200.9, Rev. 2.2 (1994).		3120 B-2011	D1976-12	I-4471-97. ⁵⁰
STGFAA	200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).		3125 B-2011	D5673-16	993.14, ³ I-4020-05, ⁷⁰ I-4472-97. ⁸¹
ICP/AES ³⁶	200.8, Rev. 5.4 (1994).			D4190-15	See footnote. ³⁴
ICP/MS			3500-Cu B-2011.		See footnote. ¹⁹
DCP ³⁶			3500-Cu C-2011		Kelada-01. ⁵⁵
Colorimetric (Neocuproine)				D7511-12(17).	
Colorimetric (Bathocuproine)			4500-CN- B-2016 and C-2016.	D2036-09(15)(A), D7284-13(17).	10-204-00-1-X. ⁵⁶
Automated UV digestion/distillation and Colorimetry.				D2036-09(15)(A) D7284-13(17).	
Segmented Flow Injection, In-Line Ultra-violet Digestion, followed by gas diffusion amperometry.			4500-CN- D-2016	D2036-09(15)(A)	p. 22. ⁹
Manual distillation with MgCl ₂ , followed by any of the following:	335.4, Rev. 1.0 (1993) ⁵⁷ .		4500-CN- E-2016	D2036-09(15)(A)	I-3300-85. ²
Flow injection, gas diffusion amperometry			4500-CN- N-2016		10-204-00-1-X. ⁵⁶ I-4302-85. ²
Titrimetric					
Spectrophotometric, manual					
Semi-Automated ²⁰	335.4, Rev. 1.0 (1993) ⁵⁷ .				

21. Color, platinum cobalt units or dominant wave-length, hue, luminance purity

22. Copper—Total,⁴ mg/L.

23. Cyanide—Total, mg/L.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
24. Cyanide-Available, mg/L.	Ion Chromatography			D2036–09(15)(A).	
	Ion Selective Electrode		4500-CN F–2016 ..	D2036–09(15)(A).	
	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl ₂ , followed by Titrimetric or Spectrophotometric.		4500-CN G–2016 ..	D2036–09(15)(B).	
24.A Cyanide-Free, mg/L.	Flow injection and ligand exchange, followed by gas diffusion amperometry ⁵⁹ .			D6888–16	OIA–1677–09. ⁴⁴
	Automated Distillation and Colorimetry (no UV digestion).				Kelada-01. ⁵⁵
25. Fluoride—Total, mg/L.	Flow injection, followed by gas diffusion amperometry.			D7237–15 (A)	OIA–1677–09. ⁴⁴
	Manual micro-diffusion and colorimetry			D4282–15.	
26. Gold—Total, ⁴ mg/L.	Manual distillation, ⁶ followed by any of the following:		4500-F B–2011	D1179–16 (A).	
	Electrode, manual		4500-F C–2011	D1179–16 (B).	I–4327–85. ²
	Electrode, automated		4500-F D–2011.		
	Colorimetric, (SPADNS)		4500-F E–2011.	D4327–17	993.30. ³
	Automated complexone		4110 B–2011 or C–2011.		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997).		D6508–15	D6508, Rev. 2. ⁵⁴
	CIE/UV		4140 B–2011		
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration		3111 B–2011.		
	AA furnace	231.2 (Issued 1978) ¹	3113 B–2010.	D5673–16	993.14. ³
27. Hardness—Total, as CaCO ₃ , mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011		See footnote. ³⁴
	DCP				
	Automated colorimetric	130.1 (Issued 1971) ¹ .		D1126–17	973.52B ³ , I–1338–85. ²
	Titrimetric (EDTA)		2340 C–2011		
	Ca plus Mg as their carbonates, by any approved method for Ca and Mg (See Parameters 13 and 33), provided that the sum of the lowest point of quantitation for Ca and Mg is below the NPDES permit requirement for Hardness.		2340 B–2011.		

28. Hydrogen ion (pH), pH units.	Electrometric measurement		4500-H+ B-2011	D1293-99 (A or B) ..	973.41, ³ I-1586-85. ²
29. Iridium—Total, ⁴ mg/L.	Automated electrode	150.2 (Dec. 1982) ¹ ..			See footnote. ²¹ I-2587-85. ²
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration		3111 B-2011.		
	AA furnace	235.2 (Issued 1978) ¹ ..	3125 B-2011.		
30. Iron—Total, ⁴ mg/L.	ICP/MS				
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶		3111 B-2011 or 3111 C-2011.	D1068-15 (A)	974.27, ³ I-3381-85. ²
	AA furnace		3113 B-2010	D1068-15 (B).	
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011	D1976-12	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011	D5673-16	993.14, ³
31. Kjeldahl Nitrogen ⁵ —Total, (as N), mg/L.	DCP ³⁶			D4190-15	See footnote. ³⁴
	Colorimetric (Phenanthroline)		3500-Fe B-2011	D1068-15 (C)	See footnote. ²²
	Manual digestion ²⁰ and distillation or gas diffusion, followed by any of the following:		4500-N _{org} B-2011 or C-2011 and 4500-NH ₃ B-2011.	D3590-17 (A)	I-4515-91. ⁴⁵
	Titration		4500-NH ₃ C-2011 ..		973.48. ³
	Nesslerization			D1426-15 (A).	
	Electrode		4500-NH ₃ D-2011 or E-2011.	D1426-15 (B).	
	Semi-automated phenate	350.1, Rev. 2.0 (1993).	4500-NH ₃ G-2011 4500-NH ₃ H-2011.		See footnote. ⁶⁰
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods.		4500-NH ₃ F-2011 ..		
	Automated gas diffusion, followed by conductivity cell analysis.				
	Automated gas diffusion followed by fluorescence detector analysis.				
	Automated Methods for TKN that do not require manual distillation				Timberline Ammonia-001. ⁷⁴ FIA/lab 100. ⁸²

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
32. Lead—Total, ⁴ mg/L.	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation).	351.1 (Rev. 1978) ¹ ..			I-4551–78. ⁸
	Semi-automated block digester colorimetric (distillation not required).	351.2, Rev. 2.0 (1993).	4500-N _{org} D-2011 ..	D3590–17 (B)	I-4515–91 ⁴⁵
	Block digester, followed by Auto distillation and Titration.				See footnote. ³⁹
	Block digester, followed by Auto distillation and Nesslerization.				See footnote. ⁴⁰
	Block Digester, followed by Flow injection gas diffusion (distillation not required).				See footnote. ⁴¹
	Digestion with peroxodisulfate, followed by Spectrophotometric (2,6-dimethyl phenol).				Hach 10242. ⁷⁶
	Digestion with persulfate, followed by Colorimetric.				NCASI TNTP W10900. ⁷⁷
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶				
	AA furnace				
33. Magnesium— Total, ⁴ mg/L.	STGFAA	200.9, Rev. 2.2 (1994).	3111 B-2011 or 3111 C-2011. 3113 B-2010	D3559–15 (A or B) .. D3559–15 (D)	974.27, ³ I-3399-85. ² I-4403–89. ⁵¹
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011	D1976–12	I-4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011	D5673–16	993.14, ³ I-4472–97. ⁸¹
	DCP ³⁶				See footnote. ³⁴
	Voltammetry ¹¹				
	Colorimetric (Dithizone)				
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration				
	ICP/AES	200.5, Rev. 4.2 (2003); ⁶⁶ 200.7, Rev. 4.4 (1994).	3111 B-2011	D511–14 (B)	974.27, ³ I-3447–85. ²
	ICP/MS	200.8, Rev. 5.4 (1994).	3120 B-2011	D1976–12	I-4471–97. ⁵⁰
			3125 B-2011	D5673–16	993.14. ³

34. Manganese— Total, ⁴ mg/L.	DCP					See footnote. ³⁴
	Ion Chromatography				D6919-17.	
	Digestion, ⁴ followed by any of the following:				D858-17 (A or B)	974.27, ³ I-3454-85. ²
	AA direct aspiration ³⁶		3111 B-2011		D858-17 (C).	
	AA furnace		3113 B-2010		D1976-12	I-4471-97. ⁵⁰
35. Mercury—Total, mg/L.	STGFAA	200.9, Rev. 2.2 (1994).				
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011		D5673-16	993.14, ³ I-4472- 97. ⁸¹
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011		D4190-15	See footnote. ³⁴
	DCP ³⁶					920.203. ³
	Colorimetric (Persulfate)		3500-Mn B-2011		D3223-17	See footnote. ²³
36. Molybdenum— Total, ⁴ mg/L.	Colorimetric (Periodate)					977.22, ³ I-3462-85. ²
	Cold vapor, Manual	245.1, Rev. 3.0 (1994).	3112 B-2011			
	Cold vapor, Automated	245.2 (Issued 1974) ¹ .				I-4464-01. ⁷¹
	Cold vapor atomic fluorescence spectrometry (CVAFS).	245.7 Rev. 2.0 (2005) ¹⁷ .				
	Purge and Trap CVAFS	1631E ⁴³ .				
37. Nickel—Total, ⁴ mg/L	Digestion, ⁴ followed by any of the following:					
	AA direct aspiration		3111 D-2011			I-3490-85. ²
	AA furnace		3113 -2010			I-3492-96. ⁴⁷
	ICP/AES	200.7, Rev. 4.4 (1994).	3120 B-2011		D1976-12	I-4471-97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011		D5673-16	993.14, ³ I-4472- 97. ⁸¹
38. Nickel—Total, ⁴ mg/L	DCP					See footnote. ³⁴
	Digestion, ⁴ followed by any of the following:					
	AA direct aspiration ³⁶		3111 B-2011 or 3111 C-2011.		D1886-14 (A or B) ..	I-3499-85. ²
	AA furnace		3113 B-2010		D1886-14 (C)	I-4503-89. ⁵¹
	STGFAA	200.9, Rev. 2.2 (1994).			D1976-12	I-4471-97. ⁵⁰
39. Nickel—Total, ⁴ mg/L	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B-2011			
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B-2011		D5673-16	993.14, ³ I-4020- 05. ⁷⁰ I-4472-97. ⁸¹

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
38. Nitrate (as N), mg/L.	DCP ³⁶	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C– 2011.	D4190–15	See footnote. ³⁴ 993.30. ³
	Ion Chromatography	4140 B–2011		D4327–17	
	CIE/UV		D6508–15	D6508, Rev. 2. ⁵⁴ .	973.50, ³ 419D ¹ 7, p. 28. ⁹ Hach 10206 ⁷⁵
	Ion Selective Electrode	352.1 (Issued 1971) ¹	4500–NO ₃ [–] D–2016.		
39. Nitrate-nitrite (as N), mg/L.	Colorimetric (Brucine sulfate)				
	Spectrophotometric (2,6-dimethylphenol) ..				
	Nitrate-nitrite N minus Nitrite N (See pa- rameters 39 and 40).				
	Cadmium reduction, Manual		4500–NO ₃ [–] E–2016 ..	D3867–16 (B).	I–2545–90. ⁵¹
	Cadmium reduction, Automated	353.2, Rev. 2.0 (1993).	4500–NO ₃ [–] F–2016 4500–NO ₃ [–] I–2016.	D3867–16 (A)	
	Automated hydrazine		4500–NO ₃ [–] H–2016.		See footnote. ⁶² 993.30. ³
	Reduction/Colorimetric	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C– 2011.	D4327–17	
	Ion Chromatography		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴ I–2547–11. ⁷² I–2548–11. ⁷² N07–0003. ⁷³
40. Nitrite (as N), mg/ L.	CIE/UV			D7781–14	
	Enzymatic reduction, followed by auto- mated colorimetric determination.				Hach 10206. ⁷⁵ See footnote. ²⁵
	Enzymatic reduction, followed by manual colorimetric determination.		4500–NO ₃ [–] J–2018.		I–4540–85, ² See footnote. ⁶² I– 2540–90. ⁸⁰ I–4545–85. ²
	Spectrophotometric (2,6-dimethylphenol) ..				
	Spectrophotometric: Manual		4500–NO ₂ [–] B–2011		
	Automated (Diazotization)				
	Automated (*bypass cadmium reduction) ..	353.2, Rev. 2.0 (1993).	4500–NO ₃ [–] F–2016 4500–NO ₃ [–] I–2016.	D3867–16 (A)	
	Manual (*bypass cadmium or enzymatic reduction).		4500–NO ₃ [–] E–2016.	D3867–16 (B).	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4500–NO ₃ [–] J–2018. 4110 B–2011 or C– 2011.	D4327–17	993.30. ³
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴

41. Oil and grease— Total recoverable, mg/L.	Automated (*bypass Enzymatic reduction) Hexane extractable material (HEM); <i>n</i> - Hexane extraction and gravimetry.	1664 Rev. A; 1664 Rev. B ⁴² .	5520 B–2011 ³⁸ .	D7781–14	I–2547–11 ⁷² I– 2548–11 ⁷² N07– 0003. ⁷³
42. Organic carbon— Total (TOC), mg/L.	Silica gel treated HEM (SGT–HEM); Silica gel treatment and gravimetry.	1664 Rev. A; 1664 Rev. B ⁴² .	5520 B–2011 ³⁸ and 5520 F–2011 ³⁸ . 5310 B–2014	D7573–09(17)	973.47, ³ p. 14. ²⁴
43. Organic nitrogen (as N), mg/L.	Heated persulfate or UV persulfate oxida- tion.		5310 C–2014 5310 D–2011.	D4839–03(17)	973.47, ³ p. 14. ²⁴
44. Ortho-phosphate (as P), mg/L.	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4). Ascorbic acid method:				
	Automated	365.1, Rev. 2.0 (1993).	4500–P F–2011 or G–2011.		973.56, ³ I–4601–85, ² I–2601–90. ⁸⁰
	Manual, single-reagent	365.3 (Issued 1978) ¹ .	4500–P E–2011	D515–88 (A)	973.55. ³
	Manual, two-reagent				
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C– 2011.	D4327–17	993.30. ³
45. Osmium—Total, ⁴ mg/L.	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴
	Digestion, ⁴ followed by any of the fol- lowing:				
	AA direct aspiration	252.2 (Issued 1978) ¹ .	3111 D–2011.		
	AA furnace				
46. Oxygen, dis- solved, mg/L.	Winkler (Azide modification)		4500–O (B–F)–2016	D888–12 (A)	973.45B, ³ I–1575– 78. ⁸
	Electrode		4500–O G–2016	D888–12 (B)	I–1576–78. ⁸
	Luminescence-Based Sensor		4500–O H–2016	D888–12 (C)	See footnote. ⁶³ See footnote. ⁶⁴
47. Palladium— Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the fol- lowing:		3111 B–2011.		
	AA direct aspiration	253.2 (Issued 1978) ¹ .	3125 B–2011.		
	AA furnace		5530 B–2010	D1783–01(12).	See footnote. ³⁴
48. Phenols, mg/L	ICP/MS				
	DCP	420.1 (Rev. 1978) ¹ ..			
	Manual distillation, ²⁶ followed by any of the following:				

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
49. Phosphorus (elemental), mg/L. 50. Phosphorus—Total, mg/L.	Colorimetric (4AAP) manual	420.1 (Rev. 1978) ¹ ..	5530 D–2010 ²⁷	D1783–01(12) (A or B).	
	Automated colorimetric (4AAP)	420.4 Rev. 1.0 (1993).			See footnote. ²⁸
	Gas-liquid chromatography				973.55. ³
	Digestion, ²⁰ followed by any of the following: Manual	365.3 (Issued 1978) ¹	4500–P B (5)–2011		
	Automated ascorbic acid reduction	365.1 Rev. 2.0 (1993).	4500–P E–2011 4500–P (F–H)–2011	D515–88 (A).	973.56, ³ I–4600–85. ²
51. Platinum—Total, ⁴ mg/L.	ICP/AES ⁴ ³⁶	200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97 ⁵⁰
	Semi-automated block digester (TKP digestion).	365.4 (Issued 1974) ¹		D515–88 (B)	I–4610–91. ⁴⁸
	Digestion with persulfate, followed by Colorimetric.				NCASI TNTP W10900. ⁷⁷
	Digestion, ⁴ followed by any of the following: AA direct aspiration	255.2 (Issued 1978) ¹ .	3111 B–2011.		
	AA furnace		3125 B–2011.		See footnote. ³⁴
52. Potassium—Total, ⁴ mg/L.	ICP/MS				
	DCP				
	Digestion, ⁴ followed by any of the following: AA direct aspiration	200.7, Rev. 4.4 (1994).	3111 B–2011 3120 B–2011.		973.53, ³ I–3630–85. ²
	ICP/AES	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14. ³
	ICP/MS				
53. Residue—Total, mg/L. 54. Residue—filterable, mg/L.	Flame photometric Electrode		3500–K B–2011. 3500–K C–2011.		
	Ion Chromatography		2540 B–2015	D6919–17.	I–3750–85. ²
	Gravimetric, 103–105°				I–1750–85. ²
	Gravimetric, 180°		2540 C–2015	D5907–13	

55. Residue—non-filterable (TSS), mg/L.	Gravimetric, 103–105° post-washing of residue.		2540 D–2015	D5907–13	I–3765–85. ²
56. Residue—settleable, mL.	Volumetric (Imhoff cone), or gravimetric		2540 F–2015.		
57. Residue—Volatile, mg/L.	Gravimetric, 550°	160.4 (Issued 1971) ¹	2540 E–2015		I–3753–85. ²
58. Rhodium—Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the following: AA direct aspiration, or AA furnace	265.2 (Issued 1978) ¹	3111 B–2011.		
59. Ruthenium—Total, ⁴ mg/L.	ICP/MS		3125 B–2011.		
	Digestion, ⁴ followed by any of the following: AA direct aspiration, or AA furnace		3111 B–2011.		
60. Selenium—Total, ⁴ mg/L.	ICP/MS	267.2 ¹	3125 B–2011.		
	Digestion, ⁴ followed by any of the following: AA furnace		3113 B–2010	D3859–15 (B)	I–4668–98. ⁴⁹
	STGFAA	200.9, Rev. 2.2 (1994).			
	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12.	
61. Silica—Dissolved, ³⁷ mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14, ³ I–4020–05 ⁷⁰ I–4472–97. ⁸¹
	AA gaseous hydride		3114 B–2011, or 3114 C–2011.	D3859–15 (A)	I–3667–85. ²
	0.45-micron filtration followed by any of the following: Colorimetric, Manual		4500-SiO ₂ C–2011 ..	D859–16	I–1700–85. ²
	Automated (Molybdosilicate)		4500-SiO ₂ E–2011 or F–2011.		I–2700–85. ²
	ICP/AES	200.5, Rev. 4.2 (2003); ⁶⁶ 200.7, Rev. 4.4 (1994).	3120 B–2011		I–4471–97. ⁵⁰
62. Silver—Total, ^{4,31} mg/L.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14, ³
	Digestion, ^{4,29} followed by any of the following: AA direct aspiration		3111 B–2011 or 3111 C–2011.		974.27, ³ p. 37, ⁹ I–3720–85. ²

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
63. Sodium—Total, ⁴ mg/L.	AA furnace STGFAA	200.9, Rev. 2.2 (1994).	3113 B–2010		I–4724–89. ⁵¹
	ICP/AES	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B–2011	D1976–12	I–4471–97. ⁵⁰
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14, ³ I–4472– 97. ⁸¹ See footnote. ³⁴
	DCP				
	Digestion, ⁴ followed by any of the fol- lowing: AA direct aspiration ICP/AES	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3111 B–2011 3120 B–2011		973.54, ³ I–3735–85. ² I–4471–97. ⁵⁰
64. Specific conduct- ance, micromhos/ cm at 25 °C.	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14, ³ See footnote. ³⁴
	DCP				
	Flame photometric		3500–Na B–2011.		
	Ion Chromatography			D6919–17.	
	Wheatstone bridge	120.1 (Rev. 1982) ¹ ..	2510 B–2011	D1125–95(99) (A)	973.40, ³ I–2781–85. ²
65. Sulfate (as SO ₄), mg/L.	Automated colorimetric	375.2, Rev. 2.0 (1993).	4500–SO ₄ ²⁻ F–2011 or G–2011.		
	Gravimetric		4500–SO ₄ ²⁻ C–2011 or D–2011.		925.54. ³
	Turbidimetric		4500–SO ₄ ²⁻ E–2011	D516–16.	
	Ion Chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997).	4110 B–2011 or C– 2011.	D4327–17	993.30, ³ I–4020– 05. ⁷⁰
	CIE/UV		4140 B–2011	D6508–15	D6508, Rev. 2. ⁵⁴
66. Sulfide (as S), mg/L.	Sample Pretreatment		4500–S ²⁻ B, C–2011.		
	Titrimetric (iodine)		4500–S ²⁻ F–2011		I–3840–85. ²
	Colorimetric (methylene blue)		4500–S ²⁻ D–2011.		
	Ion Selective Electrode		4500–S ²⁻ G–2011	D4658–15.	
	Titrimetric (iodine-iodate)		4500–SO ₃ ²⁻ B–2011.		
67. Sulfite (as SO ₃), mg/L.	Colorimetric (methylene blue)		5540 C–2011	D2330–02.	
68. Surfactants, mg/L					

69. Temperature, °C	Thermometric			2550 B-2010			See footnote. ³²
70. Thallium-Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the fol- lowing: AA direct aspiration AA furnace STGFAA ICP/AES ICP/MS		 279.2 (Issued 1978), ¹ 200.9, Rev. 2.2 (1994). 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3111 B-2011. 3113 B-2010. 3120 B-2011 3125 B-2011		D1976-12. D5673-16	993.14, ³ I-4471- 97 ⁵⁰ I-4472-97. ⁸¹
71. Tin-Total, ⁴ mg/L	Digestion, ⁴ followed by any of the fol- lowing: AA direct aspiration AA furnace STGFAA ICP/AES		 200.9, Rev. 2.2 (1994). 200.5, Rev. 4.2 (2003) ⁶⁸ , 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3111 B-2011 3113 B-2010.			I-3850-78. ⁸
72. Titanium-Total, ⁴ mg/L.	ICP/MS Digestion, ⁴ followed by any of the fol- lowing: AA direct aspiration AA furnace ICP/AES		 283.2 (Issued 1978) ¹ . 200.7, Rev. 4.4 (1994). 200.8, Rev. 5.4 (1994).	3125 B-2011 3111 D-2011.		D5673-16	993.14, ³
73. Turbidity, NTU ⁵³	ICP/MS DCP Nephelometric		 180.1, Rev. 2.0 (1993).	3125 B-2011 2130 B-2011		D5673-16 D1889-00	993.14, ³ See footnote. ³⁴ I-3860-85 ² See footnote. ⁶⁵ See footnote. ⁶⁶ See footnote. ⁶⁷
74. Vanadium-Total, ⁴ mg/L.	Digestion, ⁴ followed by any of the fol- lowing: AA direct aspiration AA furnace ICP/AES		 200.5, Rev. 4.2 (2003), ⁶⁸ 200.7, Rev. 4.4 (1994).	3111 D-2011. 3113 B-2010 3120 B-2011		D3373-17. D1976-12	I-4471-97 ⁵⁰

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard methods ⁸⁴	ASTM	USGS/AOAC/Other
75. Zinc-Total, ⁴ mg/L	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	993.14, ³ I–4020–05, ⁷⁰
	DCP			D4190–15	See footnote. ³⁴
	Colorimetric (Gallic Acid)		3500–V B–2011.		
	Digestion, ⁴ followed by any of the following:				
	AA direct aspiration ³⁶		3111 B–2011 or 3111 C–2011.	D1691–17 (A or B)	974.27, ³ p. 37, ⁹ I–3900–85. ²
	AA furnace	289.2 (issued 1978) ¹ .		D1976–12	I–4471–97. ⁵⁰
76. Acid Mine Drainage.	ICP/AES ³⁶	200.5, Rev. 4.2 (2003); ⁶⁸ 200.7, Rev. 4.4 (1994).	3120 B–2011		993.14, ³ I–4020–05, ⁷⁰ I–4472–97. ⁸¹
	ICP/MS	200.8, Rev. 5.4 (1994).	3125 B–2011	D5673–16	See footnote. ³⁴
	DCP ³⁶			D4190–15	See footnote. ³³
	Colorimetric (Zincon)	1627 ⁶⁹ .	3500 Zn B–2011		

Table IB Notes:

¹Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, Revised March 1983 and 1979, where applicable. U.S. EPA.
²Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1, unless otherwise stated. 1989, USGS.

³Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International.

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

⁵Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.

- ⁶ Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step (for a total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as < 20% RPD for all tested matrices). Alternatively, the two populations of spike recovery percentages may be compared using a recognized statistical test.
- ⁷ Industrial Method Number 379–75 WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II, February 19, 1976. Bran & Luebbe Analyzing Technologies Inc.
- ⁸ The approved method is that cited in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1, 1979, USGS.
- ⁹ American National Standard on Photographic Processing Effluents, April 2, 1975. American National Standards Institute.
- ¹⁰ In-Situ Method 1003–8–2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe, 2009. In-Situ Incorporated.
- ¹¹ The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- ¹² Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test method which measures “total 5-day BOD.” The addition of the nitrification inhibitor is not a procedural option but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger’s permit specifically states CBOD₅ is required can the permittee report data using a nitrification inhibitor.
- ¹³ OIC Chemical Oxygen Demand Method, 1978. Oceanography International Corporation.
- ¹⁴ Method 8000, Chemical Oxygen Demand, Hach Handbook of Water Analysis, 1979. Hach Company.
- 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. 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 Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-05-001, Revision 2.0, February 2005. US EPA.
- ¹⁸ National Council of the Paper Industry for Air and Stream Improvement (NCASI) Technical Bulletin 253 (1971) and Technical Bulletin 803, May 2000.
- ¹⁹ Method 8506, Bicinchoninate Method for Copper, Hach Handbook of Water Analysis, 1979. Hach Company.
- ²⁰ When using a method with block digestion, this treatment is not required.
- ²¹ Industrial Method Number 378–75WA, Hydrogen ion (pH) Automated Electrode Method, Bran & Luebbe (Technicon) Autoanalyzer II, October 1976. Bran & Luebbe Analyzing Technologies.
- ²² Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, 1980. Hach Company.
- ²³ Method 8034, Periodate Oxidation Method for Manganese, Hach Handbook of Wastewater Analysis, 1979. Hach Company.
- ²⁴ 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, (1972 Revised 1987). 1987. USGS.
- ²⁵ Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater, 1979. Hach Company.
- ²⁶ Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- ²⁷ The colorimetric reaction must be conducted at a pH of 10.0 ± 0.2.
- ²⁸ Addison, R.F., and R.G. Ackman. 1970. Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography, *Journal of Chromatography*, 47(3):421–426.
- 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 Na₂S₂O₃ 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 use of EDTA decreases method sensitivity. Analysts may omit EDTA or replace with another suitable complexing reagent provided that all method-specified quality control acceptance criteria are met.

- ³¹ For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH_4OH , 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I_2 to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH>7 with NH_4OH . Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.
- ³² "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, USGS.
- ³³ Method 8009, Zincon Method for Zinc, Hach Handbook of Water Analysis, 1979, Hach Company.
- ³⁴ Method AES0029, Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, 1986-Revised 1991, Thermo Jarrell Ash Corporation.
- ³⁵ In-Situ Method 1004-8-2009, Carbonaceous Biochemical Oxygen Demand (CBOD) Measurement by Optical Probe, 2009, In-Situ Incorporated.
- ³⁶ Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, April 16, 1992, CEM Corporation.
- ³⁷ When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
- ³⁸ Only use *n*-hexane (85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L) extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Methods 1664 Rev. A and 1664 Rev. B). Use of other extraction solvents is prohibited.
- ³⁹ Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection, Revised December 22, 1994, OI Analytical.
- ⁴⁰ Method PAI-DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection, Revised December 22, 1994, OI Analytical.
- ⁴¹ Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion, Revised December 22, 1994, OI Analytical.
- ⁴² Method 1664 Rev. B is the revised version of EPA Method 1664 Rev. A. U.S. EPA, February 1999, Revision A. Method 1664, *n*-Hexane Extractable Material (HEM); Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-98-002. U.S. EPA, February 2010, Revision B. Method 1664, *n*-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-10-001.
- ⁴³ Method 1631, Revision E, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-02-019, Revision E, August 2002, U.S. EPA. The application of clean techniques described in EPA's 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 Method OIA-1677-09, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA), 2010, OI Analytical.
- ⁴⁵ Open File Report 00-170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion, 2000, USGS.
- ⁴⁶ Open File Report 93-449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry, 1993, USGS.
- ⁴⁷ Open File Report 97-198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry, 1997, USGS.
- ⁴⁸ Open File Report 92-146, 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, 1992, USGS.
- ⁴⁹ Open File Report 98-639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry, 1999, USGS.
- ⁵⁰ Open File Report 98-165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry, 1998, USGS.
- ⁵¹ Open File Report 93-125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments, 1993, USGS.
- ⁵² Unless otherwise indicated, all EPA methods, excluding EPA Method 300.1, are published in U.S. EPA, May 1994, *Methods for the Determination of Metals in Environmental Samples*, Supplement 1, EPA/600/R-94/111; or U.S. EPA, August 1993, *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100. EPA Method 300.1 is U.S. EPA, Revision 1.0, 1997, including errata cover sheet April 27, 1999, *Determination of Inorganic Ions in Drinking Water by Ion Chromatography*.
- ⁵³ Styrene divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StablCal™ or equivalent) are acceptable substitutes for formazin.

- ⁵⁴ Method D6508–15, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte, 2015, ASTM.
- ⁵⁵ Kelada-01, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate, EPA 821-B-01–009, Revision 1.2, August 2001. U.S. EPA. Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.
- ⁵⁶ QuikChem Method 10–204–00–1–X, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis, Revision 2.2, March 2005, Lachat Instruments.
- ⁵⁷ When using sulfide removal test procedures described in EPA Method 335.4–1, reconstitute particulate that is filtered with the sample prior to distillation.
- ⁵⁸ Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation “followed by” analysis with a method, approved digestion and/or distillation are required prior to analysis.
- ⁵⁹ Samples analyzed for available cyanide using OI Analytical method OIA–1677–09 or ASTM method D6888–16 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample filtration to no more than 30 minutes to preclude settling of materials in samples.
- ⁶⁰ Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction (“phenol-hypochlorite reaction”) colorimetric ammonium determination methods. For example, when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch, March 1977, *Anal. Chem.* 49:464–469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980, *The Analyst* 105:305–316.
- ⁶¹ If atomic absorption or ICP instrumentation is not available, the aluminum colorimetric method detailed in the 19th Edition of *Standard Methods for the Examination of Water and Wastewater* may be used. This method has poorer precision and bias than the methods of choice.
- ⁶² Easy (1-Reagent) Nitrate Method, Revision November 12, 2011, Craig Chinchilla.
- ⁶³ Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and CBOD₅, Revision 1.2, October 2011, Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table IB for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).
- ⁶⁴ In-Situ Method 1002–8–2009, Dissolved Oxygen (DO) Measurement by Optical Probe, 2009, In-Situ Incorporated.
- ⁶⁵ Mitchell Method M5331, Determination of Turbidity by Nephelometry, Revision 1.0, July 31, 2008, Leck Mitchell.
- ⁶⁶ Mitchell Method M5271, Determination of Turbidity by Nephelometry, Revision 1.0, July 31, 2008, Leck Mitchell.
- ⁶⁷ Orion Method AQ4500, Determination of Turbidity by Nephelometry, Revision 5, March 12, 2009, Thermo Scientific.
- ⁶⁸ EPA Method 200.5, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA/600/R–06/115, Revision 4.2, October 2003, U.S. EPA.
- ⁶⁹ Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA–821–R–09–002, December 2011, U.S. EPA.
- ⁷⁰ Techniques and Methods Book 5–B1, Determination of Elements in Natural-Water, Biota, Sediment and Soil Samples Using Collision/Reaction Cell Inductively Coupled Plasma-Mass Spectrometry, Chapter 1, Section B, Methods of the National Water Quality Laboratory, Book 5, Laboratory Analysis, 2006, USGS.
- ⁷¹ Water-Resources Investigations Report 01–4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water with Cold Vapor-Atomic Fluorescence Spectrometry, 2001, USGS.
- ⁷² USGS Techniques and Methods 5–B8, Chapter 8, Section B, Methods of the National Water Quality Laboratory Book 5, Laboratory Analysis, 2011 USGS.
- ⁷³ NECi Method N07–0003, “Nitrate Reductase Nitrate-Nitrogen Analysis,” Revision 9.0, March 2014, The Nitrate Elimination Co., Inc.
- ⁷⁴ Timberline Instruments, LLC Method Ammonia-001, “Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Conductivity Cell Analysis,” June 2011, Timberline Instruments, LLC.
- ⁷⁵ Hach Company Method 10206, “Spectrophotometric Measurement of Nitrate in Water and Wastewater,” Revision 2.1, January 2013, Hach Company.
- ⁷⁶ Hach Company Method 10242, “Simplified Spectrophotometric Measurement of Total Kjeldahl Nitrogen in Water and Wastewater,” Revision 1.1, January 2013, Hach Company.
- ⁷⁷ National Council for Air and Stream Improvement (NCASI) Method TNTP-W10900, “Total (Kjeldahl) Nitrogen and Total Phosphorus in Pulp and Paper Biologically Treated Effluent by Alkaline Persulfate Digestion,” June 2011, National Council for Air and Stream Improvement, Inc.
- ⁷⁸ The pH adjusted sample is to be adjusted to 7.6 for NPDES reporting purposes.
- ⁷⁹ 1–2057–85 U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Chap. A11989, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, 1989.

- ⁸⁰ Methods I-2522–90, I-2540–90, and I-2601–90 U.S. Geological Survey Open-File Report 93–125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments, 1993.
- ⁸¹ Method I-1472–97, U.S. Geological Survey Open-File Report 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments, 1998.
- ⁸² FIALab Instruments, Inc. Method FIALab 100, "Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis", April 4, 2018, FIALab Instruments, Inc.
- ⁸³ MACHEREY-NAGEL GmbH and Co. Method 036/038 NANOCOLOR® COD LR/HR, "Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater", Revision 1.5, May 2018, MACHEREY-NAGEL GmbH and Co. KG.
- ⁸⁴ Please refer to the following applicable Quality Control Sections: Part 2000 Methods, Physical and Aggregate Properties 2020 (2017); Part 3000 Methods, Metals, 3020 (2017); Part 4000 Methods, Inorganic Nonmetallic Constituents, 4020 (2014); Part 5000 Methods, and Aggregate Organic Constituents, 5020 (2017). These Quality Control Standards are available for download at www.standardmethods.org at no charge.
- ⁸⁵ Each laboratory may establish its own control limits by performing at least 25 glucose-glutamic acid (GGA) checks over several weeks or months and calculating the mean and standard deviation. The laboratory may then use the mean $\pm$ 3 standard deviations as the control limit for future GGA checks. However, GGA acceptance criteria can be no wider than 198 ± 30.5 mg/L for BOD₅. GGA acceptance criteria for CBOD must be either 198 ± 30.5 mg/L, or the lab may develop control charts under the following conditions:
- Dissolved oxygen uptake from the seed contribution is between 0.6–1.0 mg/L.
 - Control charts are performed on at least 25 GGA checks with three standard deviations from the derived mean.
 - The RSD must not exceed 7.5%.
 - Any single GGA value cannot be less than 150 mg/L or higher than 250 mg/L.

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

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
1. Acenaphthene	GC	610.	6410 B-2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, 1625B	6440 B-2005	D4657–92 (98).	
2. Acenaphthylene	HPLC	610			
	GC	610	6410 B-2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, 1625B	6440 B-2005	D4657–92 (98).	
3. Acrolein	HPLC	610			
	GC	603			
	GC/MS	624.1, ⁴ 1624B.			
4. Acrylonitrile	GC	603			
	GC/MS	624.1, ⁴ 1624B			O-4127–96. ¹³
5. Anthracene	GC	610.			
	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
	HPLC	610	6440 B-2005	D4657–92 (98).	
6. Benzene	GC	602	6200 C-2011.		O-4127–96 ¹³ , O-4436–16. ¹⁴
	GC/MS	624.1, 1624B	6200 B-2011		See footnote ³ , p.1.
7. Benzidine	Spectrophotometric				
	GC/MS	625.1, ⁵ 1625B	6410 B-2000.		
	HPLC	605.			
	GC	610.			
8. Benzo(a)anthracene	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.

9. Benzo(a)pyrene	HPLC	610	6440 B-2005	D4657-92 (98)	See footnote ⁹ , p. 27.
GC/MS	610. 625.1, 1625B	6410 B-2000			
HPLC	610	6440 B-2005		D4657-92 (98)	
10. Benzo(b)fluoranthene	GC	610			
GC/MS	625.1, 1625B	6410 B-2000		D4657-92 (98)	See footnote ⁹ , p. 27.
HPLC	610	6440 B-2005			
11. Benzo(g,h,i)perylene	GC	610. 625.1, 1625B	6410 B-2000	D4657-92 (98)	See footnote ⁹ , p. 27.
GC/MS	610	6440 B-2005			
12. Benzo(k)fluoranthene	HPLC	610	6410 B-2000	D4657-92 (98)	
GC	610. 625.1, 1625B	6440 B-2005			
13. Benzyl chloride	GC/MS	610	6410 B-2000	D4657-92 (98)	See footnote ⁹ , p. 27.
GC	610	6440 B-2005			
14. Butyl benzyl phthalate	GC/MS	606. 625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
GC	611. 625.1, 1625B	6410 B-2000			
15. bis(2-Chloroethoxy) methane	GC	611. 625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
GC	611. 625.1, 1625B	6410 B-2000			
16. bis(2-Chloroethyl) ether	GC	606. 625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
17. bis(2-Ethylhexyl) phthalate	GC/MS	601. 624.1, 1624B	6200 C-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴
GC	601. 624.1, 1624B	6200 C-2011			O-4127-96 ¹³ , O-4436-16. ¹⁴
18. Bromodichloromethane	GC/MS	601. 624.1, 1624B	6200 C-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴
GC	611. 625.1, 1625B	6410 B-2000			
19. Bromoform	GC	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
20. Bromomethane	GC/MS	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	611. 625.1, 1625B	6410 B-2000			
21. 4-Bromophenyl phenyl ether	GC/MS	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
22. Carbon tetrachloride	GC	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
23. 4-Chloro-3-methyl phenol	GC/MS	601. 624.1, 1624B	6200 C-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴
GC	601. 624.1, 1624B	6200 C-2011			
24. Chlorobenzene	GC/MS	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
25. Chloroethane	GC/MS	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC	601. 624.1, 1624B	6200 C-2011			
26. 2-Chloroethylvinyl ether	GC/MS	601. 624.1, 1624B	6200 C-2011		O-4127-96 ¹³
GC	601. 624.1, 1624B	6200 C-2011			
27. Chloroform	GC	601. 624.1, 1624B	6200 C-2011		See footnote ⁹ , p. 27.
GC/MS	601. 624.1, 1624B	6200 C-2011			
28. Chloromethane	GC	601. 624.1, 1624B	6200 C-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴

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

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
29. 2-Chloronaphthalene	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC	612			
30. 2-Chlorophenol	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	GC	604	6420 B–2000		
31. 4-Chlorophenyl phenyl ether	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	GC	611			
32. Chrysene	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	GC	610			
33. Dibenzo(a,h)anthracene	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98)	
	GC	610			
34. Dibromochloromethane	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	HPLC	610	6440 B–2005	D4657–92 (98)	
	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
35. 1,2-Dichlorobenzene	GC/MS	624.1, 1624B	6200 B–2011		
	GC	601, 602	6200 C–2011		See footnote ⁹ , p. 27; O–4127–96, ¹³
	GC/MS	624.1, 1625B	6200 B–2011		O–4436–16, ¹⁴
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C–2011		
	GC/MS	624.1, 1625B	6200 B–2011		See footnote ⁹ , p. 27; O–4127–96, ¹³
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C–2011		
	GC/MS	624.1, 1625B	6200 B–2011		See footnote ⁹ , p. 27; O–4127–96, ¹³
38. 3,3'-Dichlorobenzidine	GC/MS	625.1, 1625B	6410 B–2000		O–4436–16, ¹⁴
	HPLC	605			
39. Dichlorodifluoromethane	GC	601			O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC/MS		6200 C–2011		
40. 1,1-Dichloroethane	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC/MS	624.1, 1624B	6200 B–2011		
41. 1,2-Dichloroethane	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC/MS	624.1, 1624B	6200 B–2011		
42. 1,1-Dichloroethene	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC/MS	624.1, 1624B	6200 B–2011		
43. <i>trans</i> -1,2-Dichloroethene	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴
	GC/MS	624.1, 1624B	6200 B–2011		
44. 2,4-Dichlorophenol	GC	604	6420 B–2000		
	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27.
45. 1,2-Dichloropropane	GC	601	6200 C–2011		
	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 ¹³ , O–4436–16, ¹⁴

46. <i>cis</i> -1,3-Dichloropropene	GC	601	6200 C-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴
47. <i>trans</i> -1,3-Dichloropropene	GC/MS	624.1, 1624B	6200 B-2011		O-4127-96 ¹³ , O-4436-16. ¹⁴
48. Diethyl phthalate	GC	601	6200 C-2011		See footnote ⁹ , p. 27.
49. 2,4-Dimethylphenol	GC/MS	624.1, 1624B	6200 B-2011		See footnote ⁹ , p. 27.
50. Dimethyl phthalate	GC	606	6410 B-2000		See footnote ⁹ , p. 27.
51. Di- <i>n</i> -butyl phthalate	GC/MS	625.1, 1625B	6420 B-2000		See footnote ⁹ , p. 27.
52. Di- <i>n</i> -octyl phthalate	GC	604	6410 B-2000		See footnote ⁹ , p. 27.
53. 2, 4-Dinitrophenol	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
54. 2,4-Dinitrotoluene	GC	606	6410 B-2000		See footnote ⁹ , p. 27.
55. 2,6-Dinitrotoluene	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
56. Epichlorohydrin	GC	609	6410 B-2000		See footnote ⁹ , p. 27.
57. Ethylbenzene	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
58. Fluoranthene	GC	602	6200 C-2011		See footnote ³ , p. 130.
59. Fluorene	GC/MS	624.1, 1624B	6200 B-2011		See footnote ⁶ , p. S102.
60. 1,2,3,4,6,7,8-Heptachloro- dibenzofuran	GC	610	6410 B-2000		O-4127-96, ¹³ O-4436-16. ¹⁴
61. 1,2,3,4,7,8,9-Heptachloro- dibenzofuran	GC/MS	625.1, 1625B	6440 B-2005	D4657-92 (98).	See footnote ⁹ , p. 27.
62. 1,2,3,4,6,7,8-Heptachloro- dibenzo- <i>p</i> -dioxin	HPLC	610	6410 B-2000		See footnote ⁹ , p. 27.
63. Hexachlorobenzene	GC	625.1, 1625B	6440 B-2005	D4657-92 (98).	See footnote ⁹ , p. 27.
64. Hexachlorobutadiene	GC/MS	1613B	6410 B-2000		See footnote ⁹ , p. 27.
65. Hexachlorocyclopentadiene	GC	612	6410 B-2000		See footnote ⁹ , p. 27; O-4127-96. ¹³
66. 1,2,3,4,7,8-Hexachloro- dibenzofuran	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27; O-4127-96. ¹³

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

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
67. 1,2,3,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B.			
68. 1,2,3,7,8,9-Hexachloro-dibenzofuran.	GC/MS	1613B.			
69. 2,3,4,6,7,8-Hexachloro-dibenzofuran.	GC/MS	1613B.			
70. 1,2,3,4,7,8-Hexachloro-dibenzo- <i>p</i> -dioxin.	GC/MS	1613B.			
71. 1,2,3,6,7,8-Hexachloro-dibenzo- <i>p</i> -dioxin.	GC/MS	1613B.			
72. 1,2,3,7,8,9-Hexachloro-dibenzo- <i>p</i> -dioxin.	GC/MS	1613B.			
73. Hexachloroethane	GC	612.			
74. Indeno(1,2,3-c,d) pyrene	GC/MS	625.1, 1625B	6410 B–2000		See footnote ⁹ , p. 27; O–4127–96. ¹³
75. Isophorone	GC	610.	6410 B–2000		See footnote ⁹ , p. 27.
76. Methylene chloride	GC/MS	625.1, 1625B	6440 B–2005	D4657–92 (98).	
77. 2-Methyl-4,6-dinitrophenol	HPLC	609.	6410 B–2000		See footnote ⁹ , p. 27.
78. Naphthalene	GC	625.1, 1625B	6200 C–2011		See footnote ⁹ , p. 130.
79. Nitrobenzene	GC/MS	624.1, 1624B	6420 B–2000.		O–4127–96 ¹³ , O–4436–16. ¹⁴
80. 2-Nitrophenol	GC	604	6410 B–2000		See footnote ⁹ , p. 27.
81. 4-Nitrophenol	GC	610.	6410 B–2000		See footnote ⁹ , p. 27.
82. N-Nitrosodimethylamine	HPLC	625.1, 1625B	6440 B–2005.		See footnote ⁹ , p. 27.
83. N-Nitrosodi- <i>n</i> -propylamine	GC	609.	6410 B–2000	D4657–92 (98).	
84. N-Nitrosodiphenylamine	HPLC	625.1, 1625B	6420 B–2000.		See footnote ⁹ , p. 27.
	GC	604	6410 B–2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, 1625B	6420 B–2000.		See footnote ⁹ , p. 27.
	GC	607.	6410 B–2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, ⁵ 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	GC	607.	6410 B–2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, ⁵ 1625B	6410 B–2000		See footnote ⁹ , p. 27.
	GC	607.	6410 B–2000		See footnote ⁹ , p. 27.
	GC/MS	625.1, ⁵ 1625B	6410 B–2000		See footnote ⁹ , p. 27.

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85. Octachlorodibenzofuran	GC/MS	1613B. ¹⁰	6410 B-2000		See footnote ⁹ , p. 27.
86. Octachlorodibenzo- <i>p</i> -dioxin	GC/MS	1613B. ¹⁰	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
87. 2,2'-oxybis(1-chloropropane) ¹² [also known as bis(2-Chloro-1- methylethyl) ether].	GC	611.	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
88. PCB-1016	GC/MS	625.1, 1625B	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
89. PCB-1221	GC	608.3	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
90. PCB-1232	GC/MS	625.1	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
91. PCB-1242	GC	608.3	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
92. PCB-1248	GC/MS	625.1	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
93. PCB-1254	GC	608.3	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
94. PCB-1260	GC/MS	625.1	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
95. 1,2,3,7,8-Pentachloro- dibenzofuran.	GC/MS	608.3	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
96. 2,3,4,7,8-Pentachloro- dibenzofuran.	GC	625.1	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
97. 1,2,3,7,8-Pentachloro-dibenzo- <i>p</i> - dioxin.	GC/MS	608.3	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
98. Pentachlorophenol	GC/MS	1613B.	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
99. Phenanthrene	GC/MS	1613B.	6410 B-2000.		See footnote ³ , p. 43; See footnote. ⁸
100. Phenol	GC	604	6410 B-2000		See footnote ³ , p. 140.
101. Pyrene	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
102. 2,3,7,8-Tetrachloro- dibenzofuran.	GC	610.	6410 B-2000		See footnote ⁹ , p. 27.
103. 2,3,7,8-Tetrachloro-dibenzo- <i>p</i> - dioxin.	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
104. 1,1,2,2-Tetrachloroethane	HPLC	604	6410 B-2000		See footnote ⁹ , p. 27.
105. Tetrachloroethene	GC/MS	625.1, 1625B	6410 B-2000		See footnote ⁹ , p. 27.
	GC/MS	610	6410 B-2000		See footnote ⁹ , p. 27.
	GC/MS	1613B. ¹⁰	6410 B-2000		See footnote ⁹ , p. 27.
	GC/MS	613, 625.1, ^{5a}	6410 B-2000		See footnote ³ , p. 130.
	GC	1613B.	6410 B-2000		O-4127-96. ¹³
	GC/MS	601	6410 B-2000		See footnote ³ , p. 130.
	GC/MS	624.1, 1624B	6410 B-2000		O-4127-96. ¹³ , O-4436-16. ¹⁴
	GC	601	6410 B-2000		
	GC/MS	624.1, 1624B	6410 B-2000		

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

Parameter ¹	Method	EPA ^{2,7}	Standard methods	ASTM	Other
106. Toluene	GC	602	6200 C–2011.		O–4127–96 ¹³ , O–4436–16. ¹⁴
107. 1,2,4-Trichlorobenzene	GC/MS	624.1, 1624B	6200 B–2011		See footnote ³ , p. 130.
	GC	612			See footnote ⁹ , p. 27; O–4127–96, ¹³
	GC/MS	625.1, 1625B	6410 B–2000		O–4436–16. ¹⁴
108. 1,1,1-Trichloroethane	GC	601	6200 C–2011.		O–4127–96 ¹³ , O–4436–16. ¹⁴
109. 1,1,2-Trichloroethane	GC/MS	624.1, 1624B	6200 B–2011		See footnote ³ , p. 130.
	GC	601	6200 C–2011		O–4127–96 ¹³ , O–4436–16. ¹⁴
110. Trichloroethene	GC/MS	624.1, 1624B	6200 B–2011		O–4127–96 ¹³ , O–4436–16. ¹⁴
111. Trichlorofluoromethane	GC/MS	624.1, 1624B	6200 C–2011.		O–4127–96 ¹³ , O–4436–16. ¹⁴
112. 2,4,6-Trichlorophenol	GC	601	6200 B–2011		O–4127–96. ¹³
113. Vinyl chloride	GC/MS	604	6420 B–2000.		See footnote ⁹ , p. 27.
	GC/MS	625.1, 1625B	6410 B–2000		O–4127–96 ¹³ , O–4436–16. ¹⁴
	GC	601	6200 C–2011.		
114. Nonylphenol	GC/MS	624.1, 1624B	6200 B–2011		
115. Bisphenol A (BPA)	GC/MS				
116. <i>p</i> -tert-Octylphenol (OP)	GC/MS				
117. Nonylphenol Monoethoxylate (NP1EO)	GC/MS				
118. Nonylphenol Diethoxylate (NP2EO)	GC/MS				
119. Adsorbable Organic Halides (AOX)	Adsorption and Coulometric Titration.	1650. ¹¹			
120. Chlorinated Phenolics	In Situ Acetylation and GC/MS.	1653. ¹¹			

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). The full text of Methods 601–613, 1613B, 1624B, and 1625B are provided at appendix A, Test Procedures for Analysis of Organic Pollutants. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B of this part, Definition and Procedure for the Determination of the Method Detection Limit. These methods are available at: <https://www.epa.gov/cwa-methods> as individual PDF files.

² Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA.

³ Method 624.1 may be used for quantitative determination of acrolein and acrylonitrile, provided that the laboratory has documentation to substantiate the ability to detect and quantify these analytes at levels necessary to comply with any associated regulations. In addition, the use of sample introduction techniques other than simple purge-and-trap may be required. QC acceptance criteria from Method 603 should be used when analyzing samples for acrolein and acrylonitrile in the absence of such criteria in Method 624.1.

⁵Method 625.1 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, N-nitrosodi-n-propylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.

^{5a}Method 625.1 screening only.

⁶Selected 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. American Public Health Association (APHA).

⁷Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 1624B, and 1625B in accordance with procedures in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624.1 and 625.1 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 quality control (QC) acceptance criteria in the pertinent method, analytical results for that parameter in the unspiked sample are suspect. The results should be reported but cannot be used to demonstrate regulatory compliance. If the method does not contain QC acceptance criteria, control limits of $\pm$ three standard deviations around the mean of a minimum of five replicate measurements must be used. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

⁸Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk, Revised October 28, 1994. 3M Corporation.

⁹Method O-3116-87 is in Open File Report 93-125, Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.

¹⁰Analysts may use Fluid Management Systems, Inc. Power-Prep system in place of manual cleanup provided the analyst meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. Method 1613, Revision B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994. U.S. EPA. The full text of this method is provided in appendix A to this part and at <https://www.epa.gov/cwa-methods/approved-cwa-test-methods-organic-compounds>.

¹¹Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration, Revision C, 1997 U.S. EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GC/MS, Revision A, 1997 U.S. EPA. The full text for both of these methods is provided at appendix A in part 430 of this chapter, The Pulp, Paper, and Paperboard Point Source Category.

¹²The compound was formerly inaccurately labeled as 2,2'-oxybis(2-chloropropane) and bis(2-chloroisopropyl) ether. Some versions of Methods 611, and 1625 inaccurately list the analyte as "bis(2-chloroisopropyl)ether," but use the correct CAS number of 108-60-1.

¹³Method O-4127-96, U.S. Geological Survey Open-File Report 97-829, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of 86 volatile organic compounds in water by gas chromatography/mass spectrometry, including detections less than reporting limits, 1998, USGS.

¹⁴Method O-4436-16 U.S. Geological Survey Techniques and Methods, book 5, chap. B12, Determination of heat purgeable and ambient purgeable volatile organic compounds in water by gas chromatography/mass spectrometry, 2016, USGS.

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
1. Aldrin	GC	617, 608.3	6630 B-2007 & C-2007	D3086-90, D5812-96 (02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
2. Ametryn	GC/MS	625.1	6410 B-2000.		See footnote, ³ p. 83; See footnote, ⁹ O-3106-93; See footnote, ⁶ p. S68.
	GC	507, 619			See footnote, ¹⁴ O-1121-91.
3. Aminocarb	GC/MS	525.2, 625.1			See footnote, ³ p. 94; See footnote, ⁶ p. S60.
	TLC				
	HPLC	632.			
4. Atraton	GC	619			See footnote, ³ p. 83; See footnote, ⁶ p. S68.

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
5. Atrazine	GC/MS GC	625.1. 507, 619, 608.3			See footnote, ³ p. 83; See footnote, ⁹ O-3106–93. See footnote, ⁹ O-3106–93.
6. Azinphos methyl	HPLC/MS GC/MS GC	525.1, 525.2, 625.1 614, 622, 1657			See footnote, ¹² O-2060–01. See footnote, ¹¹ O-1126–95. See footnote, ³ p. 25; See footnote, ⁶ p. S51.
7. Barban	GC/MS TLC	625.1			See footnote, ¹¹ O-1126–95. See footnote, ³ p. 104; See footnote, ⁶ p. S64.
8. α-BHC	HPLC GC/MS GC	632. 625.1, 617, 608.3	6630 B–2007 & C–2007	D3086–90, D5812–96(02).	See footnote, ³ p. 7; See footnote, ⁸ 3M0222.
9. β-BHC	GC/MS GC	625.1 ⁵ 617, 608.3	6410 B–2000 6630 B–2007 & C–2007	D3086–90, D5812–96(02).	See footnote, ¹¹ O-1126–95. See footnote, ⁸ 3M0222.
10. δ-BHC	GC/MS GC	625.1 617, 608.3	6410 B–2000 6630 B–2007 & C–2007	D3086–90, D5812–96(02).	See footnote, ⁸ 3M0222.
11. γ-BHC (Lindane)	GC/MS GC	625.1 617, 608.3	6410 B–2000 6630 B–2007 & C–2007	D3086–90, D5812–96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104–83; See footnote, ⁸ 3M0222.
12. Captan	GC/MS GC	625.1 ⁵ 617, 608.3	6410 B–2000 6630 B–2007	D3086–90, D5812–96(02).	See footnote, ¹¹ O-1126–95. See footnote, ³ p. 7.
13. Carbaryl	TLC				See footnote, ³ p. 94; See footnote, ⁶ p. S60.
14. Carbophenothion	HPLC HPLC/MS GC/MS GC	531.1, 632. 553 625.1 617, 608.3			See footnote, ¹² O-2060–01. See footnote, ¹¹ O-1126–95. See footnote, ⁴ page 27; See footnote, ⁶ p. S73.
15. Chlordane	GC/MS GC	625.1, 617, 608.3	6630 B–2007 & C–2007	D3086–90, D5812–96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104–83; See footnote, ⁸ 3M0222.
	GC/MS	625.1	6410 B–2000.		

16. Chloropropham	TLC				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
17. 2,4-D	HPLC	632.			See footnote, ³ p. 115; See footnote, ⁴ O-3105-83.
	GC/MS	625.1.			See footnote, ¹² O-2060-01.
	GC	615.			See footnote, ³ p. 7; See footnote, ⁴ O-3105-83; See footnote, ⁸ 3M0222.
18. 4,4'-DDD	HPLC/MS	617, 608.3		D3086-90, D5812-96(02).	
	GC				
19. 4,4'-DDE	GC/MS	625.1.		D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
	GC	617, 608.3			
20. 4,4'-DDT	GC/MS	625.1.		D3086-90, D5812-96(02).	See footnote, ¹¹ O-1126-95.
	GC	617, 608.3			See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
21. Demeton-O	GC/MS	625.1.			See footnote, ³ p. 25; See footnote, ⁶ p. S51.
	GC	614, 622			
22. Demeton-S	GC/MS	625.1.			See footnote, ³ p. 25; See footnote, ⁶ p. S51.
	GC	614, 622			
23. Diazinon	GC/MS	625.1.			See footnote, ³ p. 25; See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S51.
	GC	507, 614, 622, 1657			
24. Dicamba	GC/MS	525.2, 625.1			See footnote, ¹¹ O-1126-95.
	GC	615.			See footnote, ³ p. 115.
25. Dichlofenthion	HPLC/MS				See footnote, ¹² O-2060-01.
	GC	622.1			See footnote, ⁴ page 27; See footnote, ⁶ p. S73.
26. Dichloran	GC	608.2, 617, 608.3			See footnote, ³ p. 7.
27. Dicofof	GC	617, 608.3			See footnote, ⁴ O-3104-83.
28. Dieldrin	GC	617, 608.3		D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
29. Dioxathion	GC/MS	625.1.			See footnote, ¹¹ O-1126-95.
	GC	614.1, 1657			See footnote, ⁴ page 27; See footnote, ⁶ p. S73.
30. Disulfoton	GC	507, 614, 622, 1657			See footnote, ³ p. 25; See footnote, ⁶ p. S51.
	GC/MS	525.2, 625.1			See footnote, ¹¹ O-1126-95.

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
31. Diuron	TLC				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
32. Endosulfan I	HPLC	632			See footnote, ¹² O-2060-01.
	HPLC/MS	553			See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
	GC	617, 608.3	6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ¹³ O-2002-01.
33. Endosulfan II	GC/MS	625.1 ⁵	6410 B-2000		See footnote, ³ p. 7; See footnote, ⁸ 3M0222.
	GC	617, 608.3	6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ¹³ O-2002-01.
34. Endosulfan Sulfate	GC/MS	625.1 ⁵	6410 B-2000		See footnote, ⁸ 3M0222.
	GC	617, 608.3	6630 C-2007		See footnote, ¹³ O-2002-01.
	GC/MS	625.1	6410 B-2000		See footnote, ⁸ 3M0222.
35. Endrin	GC	505, 508, 617, 1656, 608.3	6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
	GC/MS	525.1, 525.2, 625.1 ⁵	6410 B-2000		See footnote, ⁸ 3M0222.
36. Endrin aldehyde	GC	617, 608.3	6630 C-2007		See footnote, ⁴ page 27; See footnote, ⁶ p. S73.
	GC/MS	625.1			See footnote, ¹³ O-2002-01.
37. Ethion	GC	614, 614.1, 1657			See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	GC/MS	625.1			See footnote, ¹² O-2060-01.
38. Fenuron	TLC				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
39. Fenuron-TCA	HPLC	632			See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
	HPLC/MS				See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S64.
	TLC				See footnote, ¹² O-2060-01.
40. Heptachlor	HPLC	632	6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁸ 3M0222.
	GC	505, 508, 617, 1656, 608.3			See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73; See footnote, ⁸ 3M0222.
41. Heptachlor epoxide	GC/MS	525.1, 525.2, 625.1	6410 B-2000		See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73; See footnote, ⁸ 3M0222.
	GC	617, 608.3	6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73.
42. Isodrin	GC/MS	625.1	6410 B-2000		See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73.
	GC	617, 608.3	6630 B-2007 & C-2007		See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73.
43. Isodrin	GC/MS	625.1	6410 B-2000		See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73.
	GC	617, 608.3	6630 B-2007 & C-2007		See footnote, ⁴ O-3104-83; See footnote, ⁶ p. S73.

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43. Linuron	GC					See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	HPLC	632.				See footnote, ¹² O-2060-01.
	HPLC/MS	553				See footnote, ¹¹ O-1126-95.
44. Malathion	GC/MS	614, 1657		6630 B-2007		See footnote, ³ p. 25; See footnote, ⁶ p. S51.
	GC					See footnote, ¹¹ O-1126-95.
45. Methiocarb	GC/MS	625.1				See footnote, ³ p. 94; See footnote, ⁶ p. S60.
	TLC					
	HPLC	632.				See footnote, ¹² O-2060-01.
46. Methoxychlor	HPLC/MS	505, 508, 608.2, 617, 1656, 608.3.		6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83; See footnote, ⁹ 3M0222.
	GC					See footnote, ¹¹ O-1126-95.
47. Mexacarbate	GC/MS	525.1, 525.2, 625.1				See footnote, ³ p. 94; See footnote, ⁶ p. S60.
	TLC					
	HPLC	632.		6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7; See footnote, ⁴ O-3104-83.
48. Mirex	GC/MS	625.1.				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	GC	617, 608.3				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	GC/MS	625.1.				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
49. Monuron	TLC					See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	HPLC	632.				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
50. Monuron-TCA	TLC					See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	HPLC	632.				See footnote, ³ p. 104; See footnote, ⁶ p. S64.
51. Neburon	TLC					See footnote, ³ p. 104; See footnote, ⁶ p. S64.
	HPLC	632.				See footnote, ¹² O-2060-01.
52. Parathion methyl	HPLC/MS			6630 B-2007		See footnote, ⁴ page 27; See footnote, ³ p. 25.
	GC	614, 622, 1657				See footnote, ¹¹ O-1126-95.
53. Parathion ethyl	GC/MS	625.1		6630 B-2007		See footnote, ⁴ page 27; See footnote, ³ p. 25.
	GC	614				See footnote, ¹¹ O-1126-95.
54. PCNB	GC/MS	608.1, 617, 608.3		6630 B-2007 & C-2007	D3086-90, D5812-96(02).	See footnote, ³ p. 7.
	GC					See footnote, ⁴ O-3104-83.
55. Perthane	GC	617, 608.3			D3086-90, D5812-96(02).	

TABLE ID—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹—Continued

Parameter	Method	EPA ^{2,7,10}	Standard methods	ASTM	Other
56. Prometon	GC	507, 619			See footnote, ³ p. 83; See footnote, ⁶ p. S68; See footnote, ⁹ O-3106–93.
57. Prometryn	GC/MS GC	525.2, 625.1 507, 619			See footnote, ¹¹ O-1126–95. See footnote, ³ p. 83; See footnote, ⁶ p. S68; See footnote, ⁹ O-3106–93.
58. Propazine	GC/MS GC	525.1, 525.2, 625.1 507, 619, 1656, 608.3			See footnote, ¹³ O-2002–01. See footnote, ³ p. 83; See footnote, ⁶ p. S68; See footnote, ⁹ O-3106–93.
59. Propham	GC/MS TLC	525.1, 525.2, 625.1			See footnote, ³ p. 104; See footnote, ⁶ p. S64.
60. Propoxur	HPLC HPLC/MS TLC	632.			See footnote, ¹² O-2060–01. See footnote, ³ p. 94; See footnote, ⁶ p. S60.
61. Seccumeton	HPLC TLC	632.			See footnote, ³ p. 83; See footnote, ⁶ p. S68.
62. Siduron	GC TLC	619.			See footnote, ³ p. 104; See footnote, ⁶ p. S64.
63. Simazine	HPLC HPLC/MS GC	632. 505, 507, 619, 1656, 608.3.			See footnote, ¹² O-2060–01. See footnote, ³ p. 83; See footnote, ⁶ p. S68; See footnote, ⁹ O-3106–93.
64. Strobane	GC/MS GC	525.1, 525.2, 625.1			See footnote, ¹¹ O-1126–95.
65. Swep	TLC	617, 608.3	6630 B-2007 & C-2007		See footnote, ³ p. 7. See footnote, ³ p. 104; See footnote, ⁶ p. S64.
66. 2,4,5-T	HPLC GC	632. 615	6640 B-2006		See footnote, ³ p. 115; See footnote, ⁴ O-3105–83.
67. 2,4,5-TP (Silvex)	GC	615	6640 B-2006		See footnote, ³ p. 115; See footnote, ⁴ O-3105–83.
68. Terbutylazine	GC	619, 1656, 608.3			See footnote, ³ p. 83; See footnote, ⁶ p. S68.

69. Toxaphene	GC/MS				
	GC	505, 508, 617, 1656, 608.3.	6630 B-2007 & C-2007	D3086-90, D5812- 96(02).	See footnote, ¹³ O-2002-01. See footnote, ³ p. 7; See footnote, ⁸ See footnote, ⁴ O-3105-83.
70. Trifluralin	GC/MS	525.1, 525.2, 625.1	6410 B-2000.		See footnote, ³ p. 7; See footnote, ⁹ O-3106-93.
	GC	508, 617, 627, 1656, 608.3.	6630 B-2007		See footnote, ¹¹ O-1126-95.
	GC/MS	525.2, 625.1			

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 IC of this section, where entries are listed by chemical name.

² The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B of this part, Definition and Procedure for the Determination of the Method Detection Limit.

³ Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater. September 1978. U.S. EPA. This EPA publication includes thin-layer chromatography (TLC) methods.

⁴ Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3. 1987. USGS.

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 608.3 is the preferred method.

⁶ 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. American Public Health Association (APHA).

⁷ Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608.3 and 625.1 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 5% of all samples analyzed with Method 608.3 or 5% of all samples analyzed with Method 625.1 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

⁸ Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk. Revised October 28, 1994. 3M Corporation.

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

¹⁰ EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater. EPA 821-R-92-002. April 1992. U.S. EPA. EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, 1993, U.S. EPA. EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry. Revision 2.0, 1995, U.S. EPA. EPA methods 1656 and 1657 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, 1993, U.S. EPA. Methods 608.3 and 625.1 are available at <https://www.epa.gov/cwa-methods/approved-cwa-test-methods-organic-compounds>.

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

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

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

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

TABLE IE—LIST OF APPROVED RADIOLOGIC TEST TEST PROCEDURES

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

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

² Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of 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."

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TABLE IF—LIST OF APPROVED METHODS FOR PHARMACEUTICAL POLLUTANTS

Pharmaceuticals pollutants	CAS registry No.	Analytical method number
Acetonitrile	75-05-8	1666/1671/D3371/D3695/624.1
n-Amyl acetate	628-63-7	1666/D3695
n-Amyl alcohol	71-41-0	1666/D3695
Benzene	71-43-2	D4763/D3695/502.2/524.2/624.1
n-Butyl acetate	123-86-4	1666/D3695
tert-Butyl alcohol	75-65-0	1666/624.1
Chlorobenzene	108-90-7	502.2/524.2/624.1
Chloroform	67-66-3	502.2/524.2/551/624.1
o-Dichlorobenzene	95-50-1	1625C/502.2/524.2/624.1
1,2-Dichloroethane	107-06-2	D3695/502.2/524.2/624.1
Diethylamine	109-89-7	1666/1671
Dimethyl sulfoxide	67-68-5	1666/1671
Ethanol	64-17-5	1666/1671/D3695/624.1
Ethyl acetate	141-78-6	1666/D3695/624.1
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/D3695/624.1
Methyl Cellosolve® (2-Methoxy ethanol)	109-86-4	1666/1671
Methylene chloride	75-09-2	502.2/524.2/624.1
Methyl formate	107-31-3	1666
4-Methyl-2-pentanone (MIBK)	108-10-1	1624C/1666/D3695/D4763/524.2/624.1
Phenol	108-95-2	D4763
n-Propanol	71-23-8	1666/1671/D3695/624.1
2-Propanone (Acetone)	67-64-1	D3695/D4763/524.2/624.1
Tetrahydrofuran	109-99-9	1666/524.2/624.1
Toluene	108-88-3	D3695/D4763/502.2/524.2/624.1
Triethylamine	121-44-8	1666/1671
Xylenes	(Note 1)	1624C/1666/624.1

Table IF note:

¹ 1624C: *m*-xylene 108-38-3, *o*-xylene, E-14095 (Not a CAS number; this is the number provided in the Environmental Monitoring Methods Index [EMMI] database.); 1666: *m,p*-xylene 136777-61-2, *o*-xylene 95-47-6.

TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS

[40 CFR part 455]

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ³
8	Triadimefon	43121-43-3	507/633/525.1/525.2/1656/625.1.
12	Dichlorvos	62-73-7	1657/507/622/525.1/525.2/625.1.
16	2,4-D; 2,4-D Salts and Esters [2,4-Dichloro-phenoxyacetic acid].	94-75-7	1658/515.1/615/515.2/555.
17	2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid].	94-82-6	1658/515.1/615/515.2/555.
22	Mevinphos	7786-34-7	1657/507/622/525.1/525.2/625.1.
25	Cyanazine	21725-46-2	629/507/608.3/625.1.
26	Propachlor	1918-16-7	1656/508/608.1/525.1/525.2/608.3/625.1.
27	MCPA; MCPA Salts and Esters [2-Methyl-4-chlorophenoxyacetic acid]	94-74-6	1658/615/555.
30	Dichlorprop; Dichlorprop Salts and Esters [2-(2,4-Dichlorophenoxy) propionic acid].	120-36-5	1658/515.1/615/515.2/555.
31	MCPP; MCPP Salts and Esters [2-(2-Methyl-4-chlorophenoxy) propionic acid].	93-65-2	1658/615/555.
35	TCMTB [2-(Thiocyanomethylthio) benzo-thiazole].	21564-17-0	637.
39	Pronamide	23950-58-5	525.1/525.2/507/633.1/625.1.
41	Propanil	709-98-8	632.1/1656/608.3.
45	Metribuzin	21087-64-9	507/633/525.1/525.2/1656/608.3/625.1.
52	Acephate	30560-19-1	1656/1657/608.3.
53	Acifluorfen	50594-66-6	515.1/515.2/555.
54	Alachlor	15972-60-8	505/507/645/525.1/525.2/1656/608.3/625.1.
55	Aldicarb	116-06-3	531.1.
58	Ametryn	834-12-8	507/619/525.2/625.1.
60	Atrazine	1912-24-9	505/507/619/525.1/525.2/1656/ 608.3/625.1.
62	Benomyl	17804-35-2	631.

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TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued
[40 CFR part 455]

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ^a
68	Bromacil; Bromacil Salts and Esters	314–40–9	507/633/525.1/525.2/1656/608.3/625.1.
69	Bromoxynil	1689–84–5	1625/1661/625.1.
69	Bromoxynil Octanoate	1689–99–2	1656/608.3.
70	Butachlor	23184–66–9	507/645/525.1/525.2/1656/608.3/625.1.
73	Captafol	2425–06–1	1656/608.3/625.1.
75	Carbaryl [Sevin]	63–25–2	531.1/632/553/625.1.
76	Carbofuran	1563–66–2	531.1/632/625.1.
80	Chloroneb	2675–77–6	1656/508/608.1/525.1/525.2/608.3/625.1.
82	Chlorothalonil	1897–45–6	508/608.2/525.1/525.2/1656/608.3/625.1.
84	Stirofos	961–11–5	1657/507/622/525.1/525.2/625.1.
86	Chlorpyrifos	2921–88–2	1657/508/622/625.1.
90	Fenvalerate	51630–58–1	1660.
103	Diazinon	333–41–5	1657/507/614/622/525.2/625.1.
107	Parathion methyl	298–00–0	1657/614/622/625.1.
110	DCPA [Dimethyl 2,3,5,6-tetrachloro-terephthalate].	1861–32–1	508/608.2/525.1/525.2/515.1 ² /515.2 ² /1656/608.3/625.1.
112	Dinoseb	88–85–7	1658/515.1/615/515.2/555/625.1.
113	Dioxathion	78–34–2	1657/614.1.
118	Nabonate [Disodium cyanodithioimidocarbonate].	138–93–2	630.1.
119	Diuron	330–54–1	632/553.
123	Endothall	145–73–3	548/548.1.
124	Endrin	72–20–8	1656/505/508/617/525.1/525.2/608.3/625.1.
125	Ethalfuralin	55283–68–6	1656/627/608.3 See footnote 1.
126	Ethion	563–12–2	1657/614/614.1/625.1.
127	Ethoprop	13194–48–4	1657/507/622/525.1/525.2/625.1.
132	Fenarimol	60168–88–9	507/633.1/525.1/525.2/1656/608.3/625.1.
133	Fenthion	55–38–9	1657/622/625.1.
138	Glyphosate [N-(Phosphonomethyl) glycine]	1071–83–6	547.
140	Heptachlor	76–44–8	1656/505/508/617/525.1/525.2/608.3/625.1.
144	Isopropalin	33820–53–0	1656/627/608.3.
148	Linuron	330–55–2	553/632.
150	Malathion	121–75–5	1657/614/625.1.
154	Methamidophos	10265–92–6	1657.
156	Methomyl	16752–77–5	531.1/632.
158	Methoxychlor	72–43–5	1656/505/508/608.2/617/525.1/525.2/608.3/625.1.
172	Nabam	142–59–6	630/630.1.
173	Naled	300–76–5	1657/622/625.1.
175	Norflurazon	27314–13–2	507/645/525.1/525.2/1656/608.3/625.1.
178	Benfluralin	1861–40–1	1656/627/608.3 See footnote 1.
182	Fensulfothion	115–90–2	1657/622/625.1.
183	Disulfoton	298–04–4	1657/507/614/622/525.2/625.1.
185	Phosmet	732–11–6	1657/622.1/625.1.
186	Azinphos Methyl	86–50–0	1657/614/622/625.1.
192	Organo-tin pesticides	12379–54–3	Ind-01/200.7/200.9.
197	Bolstar	35400–43–2	1657/622.
203	Parathion	56–38–2	1657/614/625.1.
204	Pendimethalin	40487–42–1	1656.
205	Pentachloronitrobenzene	82–68–8	1656/608.1/617/608.3/625.1.
206	Pentachlorophenol	87–86–5	1625/515.2/555/515.1/525.1/525.2/625.1.
208	Permethrin	52645–53–1	608.2/508/525.1/525.2/1656/1660/608.3 ⁴ /625.1 ⁴ .
212	Phorate	298–02–2	1657/622/625.1.
218	Busan 85 [Potassium dimethyldithiocarbamate].	128–03–0	630/630.1.
219	Busan 40 [Potassium N-hydroxymethyl-N-methyldithiocarbamate].	51026–28–9	630/630.1.
220	KN Methyl [Potassium N-methyldithiocarbamate].	137–41–7	630/630.1.
223	Prometon	1610–18–0	507/619/525.2/625.1.
224	Prometryn	7287–19–6	507/619/525.1/525.2/625.1.
226	Propazine	139–40–2	507/619/525.1/525.2/1656/608.3/625.1.
230	Pyrethrin I	121–21–1	1660.
232	Pyrethrin II	121–29–9	1660.
236	DEF [S,S,S-Tributyl phosphorotrithioate]	78–48–8	1657.
239	Simazine	122–34–9	505/507/619/525.1/525.2/1656/608.3/625.1.
241	Carbam-S [Sodium dimethyldithiocarbamate].	128–04–1	630/630.1.

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TABLE IG—TEST METHODS FOR PESTICIDE ACTIVE INGREDIENTS—Continued
[40 CFR part 455]

EPA survey code	Pesticide name	CAS No.	EPA analytical method No.(s) ³
243	Vapam [Sodium methyldithiocarbamate]	137-42-8	630/630.1.
252	Tebuthiuron	34014-18-1	507/525.1/525.2/625.1.
254	Terbacil	5902-51-2	507/633/525.1/525.2/1656/608.3/625.1.
255	Terbufos	13071-79-9	1657/507/614.1/525.1/525.2/625.1.
256	Terbutylazine	5915-41-3	619/1656/608.3.
257	Terbutryn	886-50-0	507/619/525.1/525.2/625.1.
259	Dazomet	533-74-4	630/630.1/1659.
262	Toxaphene	8001-35-2	1656/505/508/617/525.1/525.2/608.3/ 625.1.
263	Merphos [Tributyl phosphorothioate]	150-50-5	1657/507/525.1/525.2/622/625.1.
264	Trifluralin ¹	1582-09-8	1656/508/617/627/525.2/608.3/625.1.
268	Ziram [Zinc dimethyldithiocarbamate]	137-30-4	630/630.1.

Table IG notes:

¹ Monitor and report as total Trifluralin.

² Applicable to the analysis of DCPA degradates.

³ EPA Methods 608.1 through 645, 1645 through 1661, and Ind-01 are available in Methods for the Determination of Non-conventional Pesticides in Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, Revision I, August 1993, U.S. EPA. EPA Methods 200.9 and 505 through 555 are available in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, August 1993, U.S. EPA. The full text of Methods 608.3, 625.1, and 1625 are provided at appendix A of this part. The full text of Method 200.7 is provided at appendix C of this part. Methods 608.3 and 625.1 are available at <https://www.epa.gov/cwa-methods/approved-cwa-test-methods-organic-compounds>.

⁴ Permethrin is not listed within methods 608.3 and 625.1; however, *cis*-permethrin and *trans*-permethrin are listed. Permethrin can be calculated by adding the results of *cis*- and *trans*-permethrin.

TABLE 1H—LIST OF APPROVED MICROBIOLOGICAL METHODS FOR AMBIENT WATER

Parameter and units	Method ¹	EPA	Standard methods	AOAC, ASTM, USGS	Other
Bacteria					
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube, 3 dilution, or.	p. 132 ³	9221 E–2014, 9221 F–2014 ³² .		
2. Coliform (total), number per 100 mL.	Membrane filter (MF) ² , single step	p. 124 ³	9222 D–2015 ²⁶	B–0050–85 ⁴ .	
	MPN, 5 tube, 3 dilution, or	p. 114 ³	9221 B–2014	B–0025–85 ⁴ .	
3. <i>E. coli</i> , number per 100 mL.	MF ² , single step or two step	p. 108 ³	9222 B–2015 ²⁷ .		
	MF ² with enrichment	p. 111 ³	9222 B–2015 ²⁷ .		
	MPN ^{5,7,13} , multiple tube, or		9221 B.3–2014/9221 F–2014 ^{10,12,32} .		
	Multiple tube/multiple well, or		9223 B–2016 ¹¹	991.15 ⁹	Colilert® ^{11,15} , Colilert-18® ^{11,14,15}
	MF ^{2,5,6,7} , two step, or	1103.1 ¹⁸	9222 B–2015/9222 I–2015 ¹⁷ , 9213 D–2007.	D5392–93 ⁸ .	
	Single step	1603 ¹⁹ , 1604 ²⁰			m-ColiBlue24® ¹⁶ , KwikCount™ EC. ^{28,29}
4. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 139 ³	9230 B–2013	B–0055–85 ⁴ .	
5. Enterococci, number per 100 mL.	MF ² , or	p. 136 ³	9230 C–2013 ³⁰ .		
	Plate count	p. 143 ³			
	MPN ^{5,7} , multiple tube/multiple well, or		9230 D–2013	D6503–99 ⁸	Enterolert®. ^{11,21}
	MF ^{2,5,6,7} two step, or	1106.1 ²²	9230 C–2013 ³⁰	D5259–92 ⁸ .	
	Single step, or	1600 ²³	9230 C–2013 ³⁰ .		
	Plate count	p. 143 ³ .			
Protozoa					
6. <i>Cryptosporidium</i> ...	Filtration/IMS/FA	1622 ²⁴ , 1623 ²⁵ , 1623.1 ^{25,31} .			
7. <i>Giardia</i>	Filtration/IMS/FA	1623 ²⁵ , 1623.1 ^{25,31} .			

Table 1H notes:

- 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 Microbiological Methods for Monitoring the Environment, Water and Wastes. EPA/600/8-78/017. 1978. US EPA.
- ⁴ U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. 1989. USGS.
- ⁵ 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.
- ⁶ When the MF method has not been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
- ⁷ 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.
- ⁸ Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. 2000, 1999, 1996. ASTM International.
- ⁹ Official Methods of Analysis of AOAC International, 16th Edition, Volume 1, Chapter 17. 1995. AOAC International.
- ¹⁰ The multiple-tube fermentation test is used in 9221B.3-2014. 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.
- ¹¹ These tests are collectively known as defined enzyme substrate tests.
- ¹² After prior enrichment in a presumptive medium for total coliform using 9221B.3-2014, 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-2014. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.
- ¹³ Samples shall be enumerated by the multiple-tube or multiple-tube 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 Coli-Test[®] may be enumerated with the multiple-tube procedures, Quanti-Tray[®] or Quanti-Tray[®]/2000, and the MPN calculated from the table provided by the manufacturer.
- ¹⁴ Coli-Test[®] is an optimized formulation of the Coli-Test[®] 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 Coli-Test[®] test, and is recommended for marine water samples.
- ¹⁵ Descriptions of the Coli-Test[®], Coli-Test[®], Quanti-Tray[®], and Quanti-Tray[®]/2000 may be obtained from IDEXX Laboratories Inc.
- ¹⁶ A description of the mColiBlue24[®] test may be obtained from Hach Company.
- ¹⁷ Subject coliform positive samples determined by 9222B-2015 or other membrane filter procedure to 9222I-2015 using NA-MUG media.
- ¹⁸ Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC), EPA-821-R-10-002, March 2010. US EPA.
- ¹⁹ Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC), EPA-821-R-14-010. September 2014. US EPA.
- ²⁰ Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium), EPA 821-R-02-024, September 2002. US EPA.
- ²¹ A description of the Enterolert[®] test may be obtained from IDEXX Laboratories Inc.
- ²² Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA), EPA-821-R-09-015. December 2009. US EPA.
- ²³ Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI), EPA-821-R-14-011. September 2014. US EPA.
- ²⁴ Method 1622 uses a filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA, EPA-821-R-05-001. December 2005. US EPA.
- ²⁵ Methods 1623 and 1623.1 use a filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA, EPA-821-R-05-002. December 2005. US EPA. Method 1623.1: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA, EPA 816-R-12-001. January 2012. US EPA.

²⁶ On a monthly basis, at least ten blue colonies from positive samples must be verified using Lauryl Tryptose Broth and EC broth, followed by count adjustment based on these results; and representative non-blue colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

²⁷ On a monthly basis, at least ten sheen colonies from positive samples must be verified using Lauryl Tryptose Broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources.

²⁸ A description of KwikCount™ EC may be obtained from Micrology Laboratories LLC.

²⁹ Approved for the analyses of *E. coli* in freshwater only.

³⁰ Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional. As per the Errata to the 23rd Edition of *Standard Methods for the Examination of Water and Wastewater*, "Growth on a BHI agar plate incubated at 10 ± 0.5 °C for 48 ± 3 h is further verification that the colony belongs to the genus *Enterococcus*."

³¹ Method 1623.1 includes updated acceptance criteria for IPR, OPR, and MS/MSD and clarifications and revisions based on the use of Method 1623 for years and technical support questions.

³² 9221 F.2-2014 allows for simultaneous detection of *E. coli* and thermotolerant fecal coliforms by adding inverted vials to EC-MUG; the inverted vials collect gas produced by thermotolerant fecal coliforms.

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(b) Certain material is incorporated by reference into this section with the approval of the Director of the Federal Register under 5 U.S.C. 552(a) and 1 CFR part 51. All approved material may be inspected at EPA's Water Docket, EPA West, 1301 Constitution Avenue NW, Room 3334, Washington, DC 20004, (Telephone: 202-566-2426). It is also available for inspection at 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/cfr/ibr-locations.html>.

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

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

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

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

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

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

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

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

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

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

(ii) [Reserved]

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

(5) Office of Research and Development, Cincinnati OH. U.S. Environ-

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

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

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

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

(A) Method 120.1, Conductance, Specific Conductance, μmhos at 25 °C. Revision 1982. Table IB, Note 1.

(B) Method 130.1, Hardness, Total (mg/L as CaCO_3), Colorimetric, Automated EDTA. Issued 1971. Table IB, Note 1.

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

(D) Method 160.4, Residue, Volatile, Gravimetric, Ignition at 550 °C. Issued 1971. Table IB, Note 1.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

(U) Method 420.1, Phenolics, Total Recoverable, Spectrophotometric, Manual 4-AAP With Distillation. Revision 1978. Table IB, Note 1.

(iv) Prescribed Procedures for Measurement of Radioactivity in Drinking Water. 1980. EPA-600/4-80-032. Table IE.

(A) Method 900.0, Gross Alpha and Gross Beta Radioactivity. Table IE.

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

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

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

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

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

(ii) [Reserved]

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

(i) Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrom-

etry. Revision E, August 2002. EPA-821-R-02-019, Pub. No. PB2002-108220. Table IB, Note 43.

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

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

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

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

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

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

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

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

(C) Method 614, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.

(D) Method 614.1, Organophosphorus Pesticides. Table ID, Note 10; Table IG, Note 3.

(E) Method 615, Chlorinated Herbicides. Table ID, Note 10; Table IG, Note 3.

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(lxv) ASTM D7237–15a, Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection. June 2015. Table IB.

(lxvi) ASTM D7284–13 (Reapproved 2017), Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection. July 2017. Table IB.

(lxvii) ASTM D7365–09a, Standard Practice for Sampling, Preservation, and Mitigating Interferences in Water Samples for Analysis of Cyanide. October 2009. Table II, Notes 5 and 6.

(lxviii) ASTM D7511–12 (Reapproved 2017), Standard Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection. July 2017. Table IB.

(lxix) ASTM D7573–09 (Reapproved 2017), Standard Test Method for Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection, February 2017. Table IB.

(lxx) ASTM D7781–14 Standard Test Method for Nitrate-Nitrite in Water by Nitrate Reductase, May 2014. Table IB.

(16) Bran & Luebbe Analyzing Technologies, Inc., Elmsford NY 10523.

(i) Industrial Method Number 378–75WA, Hydrogen Ion (pH) Automated Electrode Method, Bran & Luebbe (Technicon) Auto Analyzer II. October 1976. Table IB, Note 21.

(ii) [Reserved]

(17) CEM Corporation, P.O. Box 200, Matthews NC 28106–0200.

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(i) Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals. April 16, 1992. Table IB, Note 36.

(ii) [Reserved]

(18) Craig R. Chinchilla, 900 Jorie Blvd., Suite 35, Oak Brook IL 60523. Telephone: 630-645-0600.

(i) Nitrate by Discrete Analysis Easy (1-Reagent) Nitrate Method, (Colorimetric, Automated, 1 Reagent). Revision 1, November 12, 2011. Table IB, Note 62.

(ii) [Reserved]

(19) FIALab Instruments, Inc., 2151 N. Northlake Way, Seattle, WA 98103. Telephone: 425-376-0450.

(i) Method 100, Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Fluorescence Detector Analysis, April 4, 2018. Table IB, Note 82.

(ii) [Reserved]

(20) Hach Company, P.O. Box 389, Loveland CO 80537.

(i) Method 8000, Chemical Oxygen Demand. Hach Handbook of Water Analysis. 1979. Table IB, Note 14.

(ii) Method 8008, 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water. 1980. Table IB, Note 22.

(iii) Method 8009, Zincon Method for Zinc. Hach Handbook for Water Analysis. 1979. Table IB, Note 33.

(iv) Method 8034, Periodate Oxidation Method for Manganese. Hach Handbook for Water Analysis. 1979. Table IB, Note 23.

(v) Method 8506, Bicinchoninate Method for Copper. Hach Handbook of Water Analysis. 1979. Table IB, Note 19.

(vi) Method 8507, Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater. 1979. Table IB, Note 25.

(vii) Method 10206, Hach Company TNTplus 835/836 Nitrate Method 10206, Spectrophotometric Measurement of Nitrate in Water and Wastewater. Revision 2.1, January 10, 2013. Table IB, Note 75.

(viii) Method 10242, Hach Company TNTplus 880 Total Kjeldahl Nitrogen Method 10242, Simplified Spectrophotometric Measurement of Total Kjeldahl Nitrogen in Water and Wastewater. Revision 1.1, January 10, 2013. Table IB, Note 76.

(ix) Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and cBOD₅. Revision 1.2, October 2011. Table IB, Note 63.

(x) m-ColiBlue24® Method, for total Coliforms and *E. coli*. Revision 2, 1999. Table IA, Note 18; Table IH, Note 17.

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

(i) Colilert. 2013. Table IA, Notes 17 and 18; Table IH, Notes 14, 15 and 16.

(ii) Colilert-18. 2013. Table IA, Notes 17 and 18; Table IH, Notes 14, 15 and 16.

(iii) Enterolert. 2013. Table IA, Note 24; Table IH, Note 12.

(iv) Quanti-Tray Insert and Most Probable Number (MPN) Table. 2013. Table IA, Note 18; Table IH, Notes 14 and 16.

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

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

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

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

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

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

(ii) [Reserved]

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

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

(ii) [Reserved]

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

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

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

(26) MACHEREY-NAGEL GmbH and Co., 2850 Emrick Blvd. Bethlehem, PA 18020. Telephone: 888-321-6224.

(i) Method 036/038 NANOCOLOR® COD LR/HR, Spectrophotometric Measurement of Chemical Oxygen Demand in Water and Wastewater, Revision 1.5, May 2018. Table IB, Note 83.

(ii) [Reserved]

(27) Micrology Laboratories, LLC, 1303 Eisenhower Drive, Goshen, IN 46526. Telephone: 574-533-3351.

(i) KwikCount™ EC Medium *E. coli* enzyme substrate test, Rapid Detection of *E. coli* in Beach Water By KwikCount™ EC Membrane Filtration. 2014. Table IH, Notes 28 and 29.

(ii) [Reserved]

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

(i) NCASI Method TNTP-W10900, Total Nitrogen and Total Phosphorus in Pulp and Paper Biologically Treated Effluent by Alkaline Persulfate Digestion. June 2011. Table IB, Note 77.

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

(iii) NCASI Technical Bulletin No. 803, An Update of Procedures for the Measurement of Color in Pulp Mill Wastewaters. May 2000. Table IB, Note 18.

(29) The Nitrate Elimination Co., Inc. (NECI), 334 Hecla St., Lake Linden MI 49945.

(i) NECI Method N07-0003, Method for Nitrate Reductase Nitrate-Nitrogen Analysis. Revision 9.0. March 2014. Table IB, Note 73.

(ii) [Reserved]

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

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

(ii) [Reserved]

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

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

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

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

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

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

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

(ii) [Reserved]

(33) Technicon Industrial Systems, Tarrytown NY 10591.

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

(ii) [Reserved]

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

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

(ii) [Reserved]

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

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

(ii) [Reserved]

(36) 3M Corporation, 3M Center Building 220-9E-10, St. Paul MN 55144-1000.

(i) Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk™ Test Method 3M 0222. Revised

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October 28, 1994. Table IC, Note 8; Table ID, Note 8.

(ii) [Reserved]

(37) Timberline Instruments, LLC, 1880 South Flatiron Ct., Unit I, Boulder CO 80301.

(i) Timberline Amonia-001, Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Conductivity Cell Analysis. June 24, 2011. Table IB, Note 74.

(ii) [Reserved]

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

(i) Colorimetric determination of nitrate plus nitrite in water by enzymatic reduction, automated discrete analyzer methods. U.S. Geological Survey Techniques and Methods, Book 5—Laboratory Analysis, Section B—Methods of the National Water Quality Laboratory, Chapter 8. 2011. Table IB, Note 72.

(ii) Determination of Heat Purgeable and Ambient Purgeable Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry. Chapter 12 of Section B, Methods of the National Water Quality Laboratory, of Book 5, Laboratory Analysis. 2016.

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

(iv) Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1989. Table IB, Notes 2 and 79.

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

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

(vii) OFR 91-519, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination

of Organonitrogen Herbicides in Water by Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry With Selected-Ion Monitoring. 1992. Table ID, Note 14.

(viii) OFR 92-146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by a Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. Table IB, Note 48.

(ix) OFR 93-125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. Table IB, Note 51 and 80; Table IC, Note 9.

(x) OFR 93-449, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1993. Table IB, Note 46.

(xi) OFR 94-37, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors. 1994. Table ID, Note 9.

(xii) OFR 95-181, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Pesticides in Water by C-18 Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry With Selected-Ion Monitoring. 1995. Table ID, Note 11.

(xiii) OFR 97-198, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum in Water by Graphite Furnace Atomic Absorption Spectrophotometry. 1997. Table IB, Note 47.

(xiv) OFR 97-829, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of 86 Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry, Including Detections Less Than Reporting Limits. 1999. Table IC, Note 13.

(xv) OFR 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-Water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. Table IB, Notes 50 and 81.

(xvi) OFR 98–639, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace—Atomic Absorption Spectrometry. 1999. Table IB, Note 49.

(xvii) OFR 00–170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. Table IB, Note 45.

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

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

(xx) Water-Resources Investigation Report 01–4098, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Moderate-Use Pesticides and Selected Degradates in Water by C–18 Solid-Phase Extraction and Gas Chromatography/Mass Spectrometry. 2001. Table ID, Note 13.

(xxi) Water-Resources Investigations Report 01–4132, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water With Cold Vapor-Atomic Fluorescence Spectrometry. 2001. Table IB, Note 71.

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

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

(39) Waters Corporation, 34 Maple Street, Milford MA 01757, Telephone: 508–482–2131, Fax: 508–482–3625.

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

(ii) [Reserved]

(c) Under certain circumstances, the Director may establish limitations on the discharge of a parameter for which there is no test procedure in this part or in 40 CFR parts 405 through 499. In these instances the test procedure shall be specified by the Director.

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

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters are cited in Tables IA, IB, IC, ID, IE, IF, IG, and IH are prescribed in Table II. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a change from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for such limited use changes may be made by letters to the Regional Alternative Test Procedure (ATP) Program Coordinator or the permitting authority in the Region in which the discharge will occur. Sufficient data should be provided to assure such changes in sample preservation, containers or holding

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times do not adversely affect the integrity of the sample. The Regional ATP Coordinator or permitting authority will review the application and then notify the applicant and the appropriate State agency of approval or rejection of the use of the alternate test procedure. A decision to approve or deny any request on deviations from

the prescribed Table II requirements will be made within 90 days of receipt of the application by the Regional Administrator. An analyst may not modify any sample preservation and/or holding time requirements of an approved method unless the requirements of this section are met.

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

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IA—Bacterial Tests			
1–4. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ^{22,23}
5. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
6. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
7. <i>Salmonella</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
Table IA—Aquatic Toxicity Tests			
8–11. Toxicity, acute and chronic.	P, FP, G	Cool, ≤6 °C ¹⁶	36 hours.
Table IB—Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
9. Biochemical oxygen demand.	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
10. Boron	P, FP, or Quartz	HNO ₃ to pH <2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
23–24. Cyanide, total or available (or CATC) and free.	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH >10 ^{5,6} , reducing agent if oxidizer present.	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH <2	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
Table IB—Metals ⁷			
18. Chromium VI	P, FP, G	Cool, ≤6 °C ¹⁸ , pH = 9.3–9.7 ²⁰ .	28 days.
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH <2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷ .	90 days. ¹⁷
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO ₃ to pH <2, or at least 24 hours prior to analysis ¹⁹ .	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.

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

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
41. Oil and grease	G	Cool to ≤6 °C ¹⁸ , HCl or H ₂ SO ₄ to pH <2.	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C ¹⁸ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH <2.	28 days.
44. Orthophosphate	P, FP, G	Cool, to ≤6 °C ^{18,24}	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe ..	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark ..	8 hours.
48. Phenols	G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
49. Phosphorus (elemental)	G	Cool, ≤6 °C ¹⁸	48 hours.
50. Phosphorus, total	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2.	28 days.
53. Residue, total	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
54. Residue, Filterable (TDS) ..	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
55. Residue, Nonfilterable (TSS).	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C ¹⁸	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C ¹⁸	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C ¹⁸	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C ¹⁸ , add zinc acetate plus sodium hydroxide to pH >9.	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.
69. Temperature	P, FP, G	None required	Analyze within 15 minutes.
73. Turbidity	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours.

Table IC—Organic Tests ⁵

13, 18–20, 22, 24, 25, 27, 28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹ .	14 days. ⁹
26. 2-Chloroethylvinyl ether	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons.	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹ .	14 days. ⁹
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ , pH to 4–5 ¹⁰ .	14 days. ¹⁰
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ .	7 days until extraction, 40 days after extraction.
7, 38. Benzidines ^{11,12}	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction. ¹³
14, 17, 48, 50–52. Phthalate esters ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines ^{11,14}	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
88–94. PCBs ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ .	7 days until extraction, 40 days after extraction.
29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons ¹¹ .	G, FP-lined cap	Cool, ≤6 °C ¹⁸	7 days until extraction, 40 days after extraction.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs ¹¹ .	G	See footnote 11	See footnote 11.
Aqueous Samples: Field and Lab Preservation.	G	Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH <9.	1 year.
Solids and Mixed-Phase Samples: Field Preservation.	G	Cool, ≤6 °C ¹⁸	7 days.
Tissue Samples: Field Preservation.	G	Cool, ≤6 °C ¹⁸	24 hours.

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

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation.	G	Freeze, ≤ -10 °C	1 year.
114–118. Alkylated phenols	G	Cool, <6 °C, H ₂ SO ₄ to pH <2	28 days until extraction, 40 days after extraction.
119. Adsorbable Organic Halides (AOX).	G	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , HNO ₃ to pH <2.	Hold at least 3 days, but not more than 6 months.
120. Chlorinated Phenolics	G, FP-lined cap	Cool, <6 °C, 0.008% Na ₂ S ₂ O ₃ , H ₂ SO ₄ to pH <2.	30 days until acetylation, 30 days after acetylation.

Table ID—Pesticides Tests

1–70. Pesticides ¹¹	G, FP-lined cap	Cool, ≤6 °C ¹⁸ , pH 5–9 ¹⁵	7 days until extraction, 40 days after extraction.
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Table IE—Radiological Tests

1–5. Alpha, beta, and radium ..	P, FP, G	HNO ₃ to pH <2	6 months.
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Table IH—Bacterial Tests

1, 2. Coliform, total, fecal	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
3. <i>E. coli</i>	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
4. Fecal streptococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²
5. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	8 hours. ²²

Table IH—Protozoan Tests

6. <i>Cryptosporidium</i>	LDPE; field filtration	1–10 °C	96 hours. ²¹
7. <i>Giardia</i>	LDPE; field filtration	1–10 °C	96 hours. ²¹

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

²Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.24(f)(14)(iv) and (v) (volatile organics).

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

⁴Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory have 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 ATP Coordinator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0–6 °C, with minimum head space.

⁵ ASTM D7365–09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analyses. Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (23rd edition) addresses dechlorination procedures for microbiological analyses.

⁶ Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365–09a (15). There may be interferences that are not mitigated by the analytical test methods or D7365–09a (15). Any technique for removal or suppression of interferences may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365–09a (15) or the analytical test method must be documented along with supporting data.

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

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

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

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

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

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

¹³ Extracts may be stored up to 30 days at $<0^{\circ}\text{C}$.

¹⁴ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵ The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

¹⁶ Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6°C prior to test initiation.

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

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

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

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

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

²² Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.

²³ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

²⁴ The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (i.e., that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (i.e., within 15 minutes of collection).

[38 FR 28758, Oct. 16, 1973]

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

§ 136.4 Application for and approval of alternate test procedures for nationwide use.

(a) A written application for review of an alternate test procedure (alternate method) for nationwide use may be made by letter via email or by hard copy in triplicate to the National Alternate Test Procedure (ATP) Program Coordinator (National Coordinator),