

PART 136
GUIDELINES ESTABLISHING TEST PROCEDURES
FOR THE ANALYSIS OF POLLUTANTS

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Catalyst Information Resources

June 1, 2021

[Editor's Note: This document highlights the changes to Part 136 as a result of the pre-publication 2021 Method Update Rule. Additions are shown in red font and deleted language is shown as strikeout. Some non-relevant language is not shown and minor formatting changes have been made to improve readability. Catalyst has attempted to capture all changes, but if errors are noted, please let us know.]

Edits since May 16 (Thank you Cathy Westerman!):

- Changes to Table 1A for Parameters 3 and 4
- Changes to Table 1B for CBOD, Manganese, and TOC and addition of footnote 85 (GGA check) to SM 5210B for BOD and CBOD.

136.1 Applicability.

a) The procedures shall, except as specified in 136.4, 136.5, and 136.6, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

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

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

c) For the purposes of the NPDES program, when more than one test procedure is approved under this part for the analysis of a pollutant or pollutant parameter, the test procedure must be sufficiently sensitive as defined at 122.21(e)(3) and 122.44(i)(1)(iv).

136.2 Definitions.

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

Detection limit means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is distinguishable from the method blank results as determined by the procedure set forth at appendix B of this part.

Director means the director as defined in 40 CFR 122.2

136.3 Identification of test procedures.

a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, IF, IG and 1H. The methods listed in Tables IA, IB, IC, ID, IE, IF, IG, and IH are incorporated by reference, see paragraph (b), with the exception of EPA Methods 200.7, 601–613, 624.1, 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, and the full text of Method 200.7 is printed in appendix C. The full text for determining the method detection limit when using the test procedures is given in appendix B. In the event of a conflict between the reporting requirements of Parts 122 and 125 and any reporting requirements associated with the methods listed in these tables, the provisions of Parts 122 and 125 are controlling and will determine a permittee's reporting requirements. The full

text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, IF, IG and 1H. The year **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, IE, IF, IG and 1H or by any alternate test procedure which has been approved by the Administrator. Under certain circumstances other additional or alternate test procedures may be used.

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

Parameter and units	Method ¹	EPA	Standard Methods	ASTM, AOAC, 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 G- E-14		
	Membrane filter (MF) ^{2, 5} , single step	p.124 ³	9222 D- 15²⁹	B-0050-85 ⁴	
	Multiple tube/multiple well, or				Colilert-18^{13, 18, 21, 29}
2. Coliform (fecal), number per 100 mL in presence of chlorine	MPN, 5 tube, 3 dilution, or	p.132 ³	9221 E- 14 9221 F-14³³		
	Multiple tube/multiple well, or				Colilert-18^{13, 18, 28}
	MF ^{2, 5} , single step ⁵	p. 124 ³	9222 D- 15²⁹		
3. Coliform (total), number per 100 mL in presence of chlorine	MF ^{2, 5} single step or two step	p. 108 ³	9222 B- 15³⁰	B-0050-85 ⁴	
	MPN, 5 tube, 3 dilution, or	p. 114 ³	9221 B- 14		
	MF ^{2, 5} single step or two step	p. 108 ³	9222 B- 15³⁰	B-0050-85 ⁴	
4. Coliform (total), in presence of chlorine, number per 100 mL	MF^{2, 5} with enrichment	p. 111³	9222 B-15³⁰		
	MPN, 5 tube, 3 dilution, or	p. 114³	9221 B-06		
	MF² with enrichment⁵	p. 111³	9222 (B+B.4c)-07		
4. <i>E. coli</i> , number per 100 mL	MPN ^{6,8,16} multiple tube, or		9221 B- 14 9221 F- 14^{12,14, 33}		
	multiple tube/multiple well, or		9223 B- 16¹³	991.15 ¹⁰	Colilert ^{13,18} Colilert-18 ^{13,17,18}
	MF ^{2, 5, 6,7,8} two single step, or		9222 B-15 9222 I-15³¹		
	single step	1603 ²¹			mColiBue 24 ¹⁹
5. Fecal streptococci, number per 100 mL	MPN, 5 tube, 3 dilution, or	p.139 ³	9230 B- 13		
	MF ² , or	p.136 ³	9230 C- 13³²	B-0055-85 ⁴	
	Plate count	p.143 ³			
6. Enterococci, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 139 ³	9230 B- 13		
	MPN ^{6, 8} multiple tube/multiple well, or		9230 D- 13	D6503-99 ⁹	Enterolert ^{13,23}
	MF ^{2, 5, 6,7,8} single step	1600 ²⁴	9230 C- 13³²		
7. Salmonella, number per gram dry weight ¹¹	Plate count	p.143 ³			
	MPN multiple tube	1682 ²²			
Aquatic Toxicity					
8. Toxicity, acute, fresh water organisms, LC ₅₀ , percent effluent	Water flea, Cladoceran, Ceriodaphnia dubia acute	2002.0 ²⁵			
	Water fleas, Cladocerans, Daphnia pulex and Daphnia magna acute	2021.0 ²⁵			
	Fish, Fathead minnow, Pimephales promelas, and Bannerfin shiner, Cyprinella leedsi, acute.	2000.0 ²⁵			

	Fish, Rainbow trout, <i>Oncorhynchus mykiss</i> , and brook trout, <i>Salvelinus fontinalis</i> , acute.	2019.0 ²⁵			
9. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC ₅₀ , percent effluent	Mysid, <i>Mysidopsis bahia</i> , acute	2007.0 ²⁵			
	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			
11. Toxicity, chronic, fresh water organisms, NOEC or IC ₂₅ , percent effluent	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			
	Water flea, Cladoceran, <i>Ceriodaphnia dubia</i> , survival and reproduction.	1002.0 ²⁶			
	Green alga, <i>Selenastrum capricornutum</i> , growth	1003.0 ²⁶			
14. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC ₂₅ , percent	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			

Notes to Table IA:

1. The method must be specified when results are reported.
2. A 0.45 um membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.
3. Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017. 1978 USEPA.
4. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, 1989 USGS.
5. Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.
6. Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.
7. When the MF method has not been used previously to test ambient waters with high turbidity, large number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
8. To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.
9. Annual Book of ASTM Standards--Water and Environmental Technology. Section 11.02. 2000, 1999, 1996 International. ASTM.
10. Official Methods of Analysis of AOAC International, 16th Edition, 4th Revision, 1998 AOAC.
11. ~~Approved~~ Recommended for enumeration of target organism in sewage sludge.
12. The multiple-tube fermentation test is used in 9221B.1-2014. 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, ~~where, for example, a substrate is used to detect the enzyme [beta]glucuronidase produced by E. coli.~~
14. After prior enrichment in a presumptive medium for total coliform using 9221B.1-2014, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h \pm 3 h of incubation shall be submitted to 9221F-2014. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 μ g/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. USEPA.
16. Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert may be enumerated with the multiple-well procedures, Quanti-Tray or Quanti-Tray 2000, and the MPN calculated from the table provided by the manufacturer.
17. Colilert-18 is an optimized formulation of the Colilert for the determination of total coliforms and E. coli that provides results within 18 h of incubation at 35 C rather than the 24 h required for the Colilert test and is recommended for marine water samples.
18. Descriptions of the Colilert, Colilert-18, Quanti-Tray, and Quanti-Tray 2000 may be obtained from IDEXX Laboratories, Inc.
19. A description of the mColiBlue24 test is available from Hach Company.
20. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-013. July 2006. USEPA.
- ~~21. Recommended for enumeration of target organism in wastewater effluent.~~
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.
22. Method 1682: Salmonella in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. EPA-821-R-14-012. July 2014. USEPA.
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-[beta]-D-Glucoside Agar (mEI). EPA-821-R-14-011. September 2014. USEPA.
25. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. EPA/821/R-02/012. October 2002. USEPA 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. Fourth Edition, U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/013. Fourth Edition October 2002. USEPA 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. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R-02/014. Third Edition October 2002. USEPA 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 \pm 0.2°C. This test is recommended for wastewater samples.
29. On a monthly basis, at least ten blue colonies from ~~the medium~~ 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 \pm 0.5 °C for 48 \pm 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 \pm 0.5 °C for 48 \pm 3 h is further verification that the colony belongs to the genus *Enterococcus*."
33. This procedure allows for simultaneous detection of E. coli and thermotolerant coliforms by adding inverted vials to EC-MUG; the inverted vials collect gas produced by thermotolerant 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-11	D1067- 16	I-1020-85 ²
2. Alkalinity , as CaCO ₃ , mg/L	Electrometric or Colorimetric titration to pH 4.5, manual		2320 B-11	D1067- 16	973.43 ³ I-1030-85 ²
	Automatic	310.2 (Rev 1974) ¹			I-2030-85 ²
3. Aluminum--Total , ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 D-11 or E-11		I-3051-85 ²
	AA furnace		3113 B-10		
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673- 16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	Direct Current Plasma (DCP) ³⁶			D4190- 15	See footnote 34
	Colorimetric (Eriochrome cyanine R)		3500-AI B-11		
4. Ammonia (as N), mg/L	Manual, distillation ⁶ or gas diffusion (at pH >11), followed by any of the following:	350.1 Rev. 2.0 (1993)	4500-NH ₃ B-11		973.49 ³
	Nesslerization			D1426- 15 (A)	973.49 ³ , I-3520-85 ²
	Titration		4500-NH ₃ C-11		
	Electrode		4500-NH ₃ D or E-11	D1426- 15 (B)	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods		4500-NH ₃ F -11		See footnote 60
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods	350.1 ⁵⁰ Rev. 2.0 (1993)	4500-NH ₃ G-11 4500-NH ₃ H -11		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
5. Antimony--Total , ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11		
	AA furnace		3113 B-10		
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673- 16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
6. Arsenic--Total , ⁴ mg/L	Digestion ⁴ followed by any of the following	206.5 (1978) ¹			
	AA gaseous hydride		3114 B-11 or 3114 C-11	D2972- 15 (B)	I-3062-85 ²
	AA furnace		3113 B -10	D2972- 15 (C)	I-4063-98 ⁴⁹
	STGFAA	200.9, Rev.2.2 (1994)			

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	
	ICP/MS	200.8, Rev 5.4 (1994)	3125 B--11	D5673--16	993.14 ³ , I-4020-05 ⁷⁰
	Colorimetric (SDDC)		3500-As B-11	D2972-15(A)	I-3060-85 ²
7. Barium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ¹⁴		3111D-11		I-3084-85 ²
	AA furnace		3113 B-10	D4382-18	
	ICP/AES ¹⁴	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11		I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP ³⁶				See footnote 34.
8. Beryllium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111D-11 or E-11	D3645-15 (A)	I-3095-85 ²
	AA furnace		3113 B-10	D3645-15(B)	
	STGFAA	200.9, Rev.2.2 (1994)			
	ICP/AES	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP, or			D4190-15	See footnote 34
	Colorimetric (aluminon)		See footnote 61		
9. Biochemical oxygen demand (BOD5), mg/L	Dissolved Oxygen Depletion		5210 B-16 ⁸⁵		973.44, ³ p. 17. ⁹ I-1578-78 ⁸ See footnote 10, 63
10. Boron ³⁷ --Total, mg/L	Colorimetric (curcumin)		4500-B B-11		I-3112-85 ²
	ICP/AES, or	200.5, Rev.4.2 (2003) ⁶⁸ 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-05	993.14 ³ , I-4471-97 ⁵⁰
	DCP			D4190-15	See footnote 34
	Colorimetric (aluminon)		See footnote 61		
11. Bromide, mg/L	Electrode			D1246-16	I-1125-85 ²
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, Rev. 1.0 (1997)	4110 B-11, C-11, D-11	D4327-17	993.30 ³ , I-2057-85 ⁷⁹
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
12. Cadmium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11 or C-11	D3557-17 (A or B)	974.27 ³ p.37 ⁹ I-3135-85 ² , I-3136-85 ²
	AA furnace		3113 B-10	D3557- 17 (D)	I-4138-89 ⁵¹
	STGFAA	200.9, Rev.2.2 (1994)			

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-1472-85, I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸¹ , I-4472-97 ⁸¹
	DCP ³⁶			D4190-15	See footnote 34
	Voltametry, ¹¹			D3557-17(C)	
	Colorimetric (Dithizone)		3500 Cd D-90		
13. Calcium --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following:				
	AA direct aspiration		3111 B-11	D511-14(B)	I-3152-85 ²
	ICP/AES	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11		I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	DCP				See footnote 34
	Titrimetric (EDTA)		3500-Ca B-11	D511-14 (A)	
	Ion chromatography			D6919-17	
14. Carbonaceous biochemical oxygen demand (CBOD₅), mg/L¹²	Dissolved Oxygen Depletion with nitrification inhibitor		5210 B-16 ⁸⁵		See footnote 35, 63
15. Chemical oxygen demand (COD), mg/L	Titrimetric	410.3 (Rev. 1978)	5220 B-11 or C-11	D1252-06 (12) (A)	973.46 ³ p 17 ⁹ I-3560-85 ²
	Spectrophotometric, manual or automated	410.4 Rev. 2.0, (1993)	5220 D-11	D1252-12 (B)	See footnote 13, 14, 83, I-3561-85 ²
16. Chloride, mg/L	Titrimetric (silver nitrate)		4500-Cl ⁻ B-11	D512-12 (B)	I-1183-85 ²
	(Mercuric nitrate)		4500-Cl ⁻ C-11	D512-12 (A)	973.51. ³ I-1184-85 ²
	Colorimetric, manual				I-1187-85 ²
	Automated (Ferricyanide)		4500-Cl ⁻ E-11		I-2187-85 ²
	Potentiometric Titration		4500-Cl ⁻ D-11		
	Ion Selective Electrode			D512-12 (C)	
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³ I-2057-90 ⁵¹
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
17. Chlorine --Total residual, mg/L	Amperometric direct		4500-Cl D-11	D1253-14	
	Amperometric direct (low level)		4500-Cl E-11		
	Iodometric direct		4500-Cl B-11		
	Back titration either endpoint ¹⁵ or		4500-Cl C-11		
	DPD-FAS		4500-Cl F-11		
	Spectrophotometric, DPD		4500-Cl G-11		
	Electrode				See footnote 16.
17A. Chlorine, Free available, mg/L	Amperometric direct		4500-Cl D-11	D1253-14	
	Amperometric direct (low level)		4500-Cl E-11		
	DPD-FAS		4500-Cl F-11		
	Spectrophotometric, DPD		4500-Cl G-11		
18. Chromium VI dissolved, mg/L	0.45 micron filtration followed by any of the following				

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
19. Chromium--Total, ⁴ mg/L	AA chelation-extraction or Ion chromatography	218.6, Rev 3.3 (1994)	3111 C -11	D5257-17	I-1232-85 ²
	Colorimetric (Diphenylcarbazide)		3500-Cr B-11	D1687-17(A)	993.23 ³
	Digestion ⁴ followed by any of the following				I-1230-85 ²
	AA direct aspiration ³⁶		3111 B-11	D1687-17 (B)	974.27 ³ , I-3236-85 ²
	AA chelation-extraction		3111 C-11		
	AA furnace		3113 B-10	D1687-17 (C)	I-3233-93 ⁴⁶
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
20. Cobalt--Total, ⁴ mg/L	DCP ³⁶			D4190-15	See footnote 34
	Colorimetric (Diphenylcarbazide)		3500-Cr B-11		
	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111B-11 or C-11	D3558-15 (A or B)	p. 37. ⁹ I-3239-85 ²
	AA furnace		3113 B-10	D3558-15 (C)	I-4243-89 ⁵¹
	STGFAA	200.9, Rev.2.2 (1994)			
	ICP/AES ³⁶	200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP		.	D4190-15	See footnote 34
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:	Colorimetric (ADMI)		2120 F-11 ⁷⁸		
	Platinum cobalt visual comparison		2120 B-11		I-1250-85 ²
	Spectrophotometric				See footnote 18.
22. Copper--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111B-11 or C-11	D1688-17 (A or B)	974.27 ³ p. 37. ⁹ I-3270-85 ² , I-3271-85 ²
	AA furnace		3113 B-10	D1688-17 (C)	I-4274-89 ⁵¹
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP ³⁶ or			D4190-15	See footnote 34
	Colorimetric (Neocuproine) or		3500-Cu B-11		
	Colorimetric (Bathocuproine)		3500-Cu C-11		See footnote 19
23. Cyanide--Total, mg/L	Automated Distillation and Colorimetry				Kelada-01 ⁵⁵
	Segmented Flow Injection, In-Line Ultraviolet Digestion followed by gas diffusion amperometry.			D7511-12(17)	
	Manual distillation with MgCl ₂ followed by any of the following	335.4, Rev. 1.0 (1993) ⁵⁷	4500-CN B-16 or C-16	D2036-09(15) (A) D7284-13(17)	10-204-00-1-X ⁵⁶
	Flow Injection, gas diffusion amperometry			D2036-09(15)(A)	

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
				D7284-13(17)	
	Titrimetric, or		4500-CN D-16	D2036-09(15) (A)	p. 22. ⁹
	Spectrophotometric, manual		4500-CN E-16	D2036-09(15) (A)	I-3300-85 ²
	Semi-Automated ²⁰	335.4, Rev. 1.0 (1993) ⁵⁷	4500-CN ⁻ N-16		10-204-00-1-X ⁵⁶ , I-4302-85 ²
	Ion Chromatography			D2036-09(15)(A)	
24. Cyanide, Available mg/L	Ion Selective Electrode		4500-CN F-16	D2036-09(15) (A)	
	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl ₂ followed by titrimetric or spectrophotometric		4500-CN G-16	D2036-09(15) (B)	
	Flow injection and ligand exchange, followed by amperometry ⁶¹			D6888-16	OIA-1677-09 ⁴⁴
	Automated Distillation and Colorimetry				Kelada-01 ⁵⁵
24A. Cyanide, Free, mg/L	Flow Injection, followed by gas diffusion amperometry			D7237-15 (A)	OIA-1677-09 ⁴⁴
	Manual micro-diffusion and colorimetry			D4282-15	
25. Fluoride--Total, mg/L	Manual distillation ⁶ followed by any of the following		4500-F B-11	D1179-16(A)	
	Electrode, manual		4500-F C-11	D1179-16 (B)	
	Electrode, Automated				I-4327-85 ²
	Colorimetric (SPADNS)		4500-F D-11	D1179-16(A)	
	Automated complexone		4500-F E-11		
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
26. Gold--Total, ⁴ mg/L -	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111B-11		
	AA furnace, or	231.2 (Issued 1978) ¹	3113 B-10		
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	DCP				See footnote 34
27. Hardness--Total, as CaCO ₃ , mg/L	Automated colorimetric,	130.1 (1971) ¹			
	Titrimetric (EDTA)		2340 C-11	D1126-17	973.52B. ³ , I-1338-85 ²
	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-11		
28. Hydrogen ion (pH), pH units	Electrometric measurement		4500-H ⁺ B-11	D1293-99 (A or B)	973.41. ³ , I-1586-85 ²
	Automated electrode	150.2 (Dec. 1982) ¹			See footnote 21, I-2587-85 ²
29. Iridium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration or		3111 B-11		
	AA furnace	235.2 (1978) ¹			
	ICP/MS		3125 B-11		
30. Iron--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11 or C-11	D1068-15 (A or B)	974.27. ³ , I-3381-85 ²

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	AA furnace		3113B-10	D1068-15 (C)	
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	DCP ³⁶ or Colorimetric (Phenanthroline)			D4190-15	See footnote 34
31. Kjeldahl Nitrogen⁵⁻⁻ Total, (as N), mg/L			3500-Fe B -11	D1068-15(C)	See footnote 22.
	Manual digestion ²⁰ and distillation or gas diffusion followed by any of the following		4500-N _{org} B-11 or C-11 and 4500-NH ₃ B-11	D3590-17 (A)	I-4515-91 ⁴⁵
	Titration		4500-NH ₃ C-11		973.48. ³
	Nesslerization			D1426-15(A)	
	Electrode		4500-NH ₃ D-11 or E-11	D1426-15 (B)	
	Semi-automated phenate	350.1 Rev. 2.0 (1993)	4500-NH ₃ G-11 or H-11		
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods		4500-NH ₃ F-11		See footnote 60
	Automated gas diffusion, followed by conductivity cell analysis				Timberline Ammonia-001 ⁷⁴
	Automated gas diffusion followed by fluorescence detector analysis				FIALab 100 ⁸²
	Automated methods for TKN that do not require manual digestion				
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation)	351.1 (1978) ¹			I-4551-78 ⁸
	Semi-automated block digester colorimetric (distillation not required)	351.2, Rev. 2.0 (1993)	4500-N _{org} D-11	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 peroxdisulfate, followed by Spectrophotometric (2,6- dimethyl phenol)				Hach 10242 ⁷⁶
	Digestion with persulfate, followed by Colorimetric				NCASI TNTP W10900 ⁷⁷
32. Lead--Total,⁴mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11 or C-11	D3559-15 (A or B)	974.27. ³ I-3399-85 ²
	AA furnace		3113 B-10	D3559-15 (D)	I-4403-89 ⁵¹
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸¹ , I-4472-97 ⁸¹
	DCP ³⁶			D4190-15	See footnote 34

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	Voltametry ¹¹ or			D3559-15 (C)	
	Colorimetric (Dithizone)		3500-Pb B-11		
33. Magnesium-- Total, ⁴ mg/L	Digestion ⁴ followed by any of the following:				
	AA direct aspiration		3111 B-11	D511-14 (B)	974.27 ³ I-3447-85 ²
	ICP/AES	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	DCP				See footnote 34.
	Ion chromatography			D6919-17	
34. Manganese-- Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11	D858-17 (A or B)	974.27. ³ , I-3454-85 ²
	AA furnace		3113 B-10	D858-17 (C)	
	STGFAA	200.9, Rev.2.2 (1994)			
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 ⁵ Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP ³⁶ or			D4190-15	See footnote 34
	Colorimetric (Persulfate)		3500-Mn-11		920.203 ³
	Colorimetric (Periodate)				See footnote 23
35. Mercury-- Total, ⁴ mg/L	Cold vapor, Manual	245.1, Rev. 3.0 (1994)	3112 B-11	D3223-17	977.22. ³ , I-3462-85 ²
	Cold vapor, Automated	245.2 (1974) ¹			
	Cold vapor atomic fluorescence spectrometry (CVAFS)	245.7, Rev. 2.0 (2005) ¹⁷			I-4464-01 ⁷¹
	Purge and trap CVAFS	1631E ⁴³			
36. Molybdenum-- Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 D-11		I-3490-85 ²
	AA furnace		3113 B-10		I-3492-96 ⁴⁷
	ICP/AES ³⁶	200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁸¹
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP				See footnote 34
37. Nickel-- Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11 or C-11	D1886-14(A or B)	I-3499-85 ²
	AA furnace		3113 B-10	D1886-14(C)	I-4503-89 ⁵¹
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP ³⁶ , or			D4190-15	See footnote 34.
38. Nitrate (as N), mg/L	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
	Ion Selective Electrode		4500-NO ₃ D-16		
	Colorimetric (Brucine sulfate)	352.1(1971) ¹			973.50 ³ 419 D ^{1,7} p. 28. ⁹
	Spectrophotometric (2,6- dimethylphenol)				Hach 10206 ⁷⁵
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).				
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction, Manual		4500-NO ₃ E-16	D3867-16(B)	
	Cadmium reduction, Automated	353.2, Rev. 2.0 (1993)	4500-NO ₃ F-16	D3867-16(A)	I-4545-85 ²
	Automated hydrazine		4500-NO ₃ H-16		
	Reduction/Colorimetric				See footnote 62
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
	Enzymatic reduction, followed by automated colorimetric determination			D7781-14	I-2547-11 ⁷² I-2548-11 ⁷² N07-0003 ⁷³
	Enzymatic reduction, followed by manual colorimetric determination		4500-NO ₃ J-18		
40. Nitrite (as N), mg/L;	Spectrophotometric (2,6-dimethylphenol)				Hach 10206 ⁷⁵
	Spectrophotometric: Manual or Automated (Diazotization)		4500-NO ₂ B-11		See footnote 25.
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)	4500-NO ₃ F-16 4500-NO ₃ I-16	D3867-16 (A)	I-4540-85 ² See footnote 62. I-2540-90 ⁸⁰ I-4545-85 ²
	Manual (*bypass cadmium reduction)		4500-NO ₃ E-11 4500-NO ₃ J-18	D3867-16 (B)	
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
	Automated (*bypass Enzymatic reduction)			D7781-14	I-2547-11 ⁷² I-2548-11 ⁷² N07-0003 ⁷³
41. Oil and grease--Total recoverable, mg/L	Hexane extractable material (HEM): n-Hexane extraction and gravimetry.	1664A, 1664B ⁴²	5520 B-11 ³⁸		
	Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry.	1664A, 1664B ⁴²	5520 B-11 ³⁸ and 5520 F-11 ³⁸		
42. Organic carbon--Total (TOC), mg/L	Combustion		5310 B-14	D7573-09 (17)	973.47 ³ , p. 14. ²⁴
	Heated persulfate or UV persulfate oxidation.		5310 C-14 or D-11	D4839-03 (17)	973.47 ³ , p. 14. ²⁴
43. Organic nitrogen (as N), mg/L	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)				
44. Orthophosphate (as P), mg/L	Ascorbic acid method				
	Automated	365.1, Rev. 2.0 (1993)	4500-P F -11 or G-11		973.56. ³ I-4601-85 ² , I-2601-90 ⁸⁰
	Manual single reagent		4500-P E-11	D515-88(A)	973.55 ³
	Manual two reagent	365.3 (Issued 1978) ¹			
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-00	D4327-17	993.30 ³
	CIE/UV		4140-11	D6508-15	D6508, Rev. 2 ⁵⁴

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
45. Osmium--Total ⁴ , mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration, or		3111 D-11		
	AA furnace	252.2 (1978) ¹			
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or		4500-O (B-F)-16	D888-12 (A)	973.45B ³ I-1575-78 ⁸
	Electrode		4500-O G-16	D888-12 (B)	I-1576-78 ⁸
	Luminescence Based Sensor			D888-12 (C) ⁶⁸	See footnote 63, 64
47. Palladium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration, or		3111 B-11		
	AA furnace	253.2 (1978) ¹			
	ICP/MS		3125 B-11		
	DCP				See footnote 34
48. Phenols, mg/L	Manual distillation ²⁶ followed by any of the following	420.1 (Rev. 1978) ¹	5530B-10	D1783-01(12)	
	Colorimetric (4AAP) manual, or	420.1 (Rev. 1978) ¹	5530D-10 ²⁷	D1783-01(12) (A or B)	
	Automated colorimetric (4AAP)	420.4 Rev. 1.0 (1993) ¹			
49. Phosphorus (elemental), mg/L	Gas-liquid chromatography				See footnote 28.
50. Phosphorus--Total, mg/L	Digestion ²⁰ followed by any of the following		4500-P B (5)- 11		973.55 ³
	Manual or	365.3 (1978) ¹	4500-P E -11	D515-88(A)	
	Automated ascorbic acid reduction	365.1, Rev. 2.0 (1993) ¹	4500-P (F-H)-11		973.56. ³ I-4600-85 ²
	ICP/AES ^{4, 36}	200.7 Rev 4.4 (1994)	3120 B-11		I-4471-97 ⁵⁰
	Semi-automated block digester (TKP Digestion)	365.4 (1974) ¹		D515-88 (B)	I-4610-91 ⁴⁸
	Digestion with persulfate, followed by Colorimetric				NCASI TNTP W10900 ⁷⁷
51. Platinum--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 B-11		
	AA furnace	255.2 (1978) ¹			
	ICP/MS		3125 B-11		
	DCP				See footnote 34.
52. Potassium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 B-11		973.53. ³ I-3630-85 ²
	ICP/AES ³⁶	200.7 Rev 4.4 (1994)	3120 B-11		
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	Flame photometric		3500-K B-11		
	Electrode		3500-K C-11		
	Ion chromatography			D6919-17	
53. Residue--Total, mg/L	Gravimetric, 103-105°		2540 B-15		I-3750-85 ²
54. Residue--filterable, mg/L	Gravimetric, 180°		2540 C-15	D5907-13	I-1750-85 ²
55. Residue--nonfilterable (TSS), mg/L	Gravimetric, 103-105° post washing of residue		2540 D-15	D5907-13	I-3765-85 ²

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
56. Residue --settleable, mg/L	Volumetric, (Imhoff cone), or gravimetric		2540 F-15		
57. Residue --Volatile, mg/L	Gravimetric, 550°	160.4 (1971) ¹	2540 E-15		I-3753-85 ²
58. Rhodium --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration, or		3111 B-11		
	AA furnace	265.2 (1978) ¹			
	ICP/MS		3125 B-11		
59. Ruthenium --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration, or		3111 B-11		
	AA furnace	267.2 ¹			
	ICP/MS		3125 B-11		
60. Selenium --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA furnace		3113 B-10	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	AA gaseous hydride		3114 B-11 or C-11	D3859-15 (A)	I-3667-85 ²
61. Silica ³⁷ --Dissolved, mg/L	0.45 micron filtration followed by any of the following				
	Colorimetric, Manual or		4500-SiO ₂ C-11	D859-16	I-1700-85 ²
	Automated (Molybdosilicate)		4500-SiO ₂ E-11 or F-11		I-2700-85 ²
	ICP/AES ³⁶	200.5, Rev.4.2 (2003), 200.7 Rev 4.4 (1994)	3120 B-11		I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
62. Silver --Total, ⁴ mg/L	Digestion ^{4,29} followed by any of the following				
	AA direct aspiration		3111 B-11 or C-11		974.27 ³ p. 37. ⁹ I-3720-85 ²
	AA furnace		3113 B-10		I-4724-89 ⁵¹
	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-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP				See footnote 34.
63. Sodium --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 B-11		973.54. ³ I-3735-85 ²
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11		I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁶⁴	ASTM	USGS/AOAC/Other
	DCP				See footnote 34.
	Flame photometric		3500 Na B-11		
	Ion chromatography			D6919-17	
64. Specific conductance, micromhos/cm at 25 °C	Wheatstone bridge	120.1 ¹ (1982)	2510 B-11	D1125-95(99) (A)	973.40. ³ I-1780-85 ²
65. Sulfate (as SO ₄), mg/L	Automated colorimetric	375.2 ¹ , Rev. 2.0 (1993)	4500-SO ₄ F-11 or G-11		
	Gravimetric		4500-SO ₄ C-97 or D-11		925.54 ³
	Turbidimetric		4500-SO ₄ E-11	D516-16	
	Ion chromatography	300.0, Rev. 2.1 (1993) 300.1, rev. 1.0 (1997)	4110 B-11 or C-11	D4327-17	993.30 ³ I-4020-05 ⁷⁰ .30 ₃
	CIE/UV		4140 B-11	D6508-15	D6508, Rev. 2 ⁵⁴
66. Sulfide (as S), mg/L	Sample pretreatment		4500-S ² B, C-11		
	Titrimetric (iodine)		4500-S ² F-11		I-3840-85 ²
	Colorimetric (methylene blue)		4500-S ² D-11		
	Ion Selective Electrode		4500-S ² G-11	D4658-15	
67. Sulfite (as SO ₃), mg/L	Titrimetric (iodine-iodate)		4500-SO ₃ ⁻² B-11		
68. Surfactants, mg/L	Colorimetric (methylene blue)		5540 C-11	D2330-02	
69. Temperature, °C	Thermometric		2550 B-11		See footnote 32.
70. Thallium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 B-11		
	AA furnace	279.2 ¹ (1978)	3113 B-10		
	STGFAA	200.9, Rev.2.2 (1994)			
	ICP/AES ³⁶	200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4471-97 ⁸¹ , I-4472-97 ⁸¹
71. Tin--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 B -11		I-3850-78 ⁸
	AA furnace, or		3113 B -10		
	STGFAA	200.9, Rev.2.2 (1994)			
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)			
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
72. Titanium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 D-11		
	AA furnace	283.2 ¹ (1978)			
	ICP/AES ³⁶	200.7 Rev 4.4 (1994)			
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³
	DCP				See footnote 34.
73. Turbidity, NTU	Nephelometric	180.1 ¹ , Rev. 2.0 (1993)	2130 B-11	D1889-00	I-3860-85 ² , See footnote 65, 66, 67.
74. Vanadium--Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration		3111 D -11		
	AA furnace		3113 B -10	D3373-17	

Parameter	Methodology ⁵⁸	EPA ⁵²	Standard Methods ⁸⁴	ASTM	USGS/AOAC/Other
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰
	DCP, or			D4190-15	See footnote 34.
	Colorimetric (Gallic acid)		3500-V B-11		
75. Zinc --Total, ⁴ mg/L	Digestion ⁴ followed by any of the following				
	AA direct aspiration ³⁶		3111 B-11 or C-11	D1691-17 (A or B)	974.27 ³ p. 37. ⁹ I-3900-85 ²
	AA furnace	289.2 ¹ (1978)			
	ICP/AES ³⁶	200.5, Rev.4.2 (2003) ⁶⁸ , 200.7 Rev 4.4 (1994)	3120 B-11	D1976-12	I-4471-97 ⁵⁰
	ICP/MS	200.8, Rev 5.4 (1998)	3125 B-11	D5673-16	993.14 ³ , I-4020-05 ⁷⁰ , I-4471-97 ⁸⁴ , I-4472-97 ⁸¹
	DCP ³⁶			D4190-15	See footnote 34.
	Colorimetric (Dithizone) (Zincon)		3500-Zn B-11		See footnote 33.
76. Acid Mine Drainage		1627 ⁶⁹			

Table IB Notes:

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983 and 1979 where applicable. U.S. EPA.
2. Methods 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
3. Official Methods of Analysis of the Association of Official Analytical Chemists, methods manual, Sixteenth Edition, 4th Revision, 1998. AOAC International
4. For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/111, May, 1994, and is reproduced in Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) use 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 Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.
5. Copper sulfate 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. (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, 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, USGS TWRI, Book 5, Chapter A1 (1979).
9. American National Standard on Photographic Processing Effluents, April 2, 1975.
10. In-Situ Method 1003-8-2009, Biochemical Oxygen Demand (BOD) Measurement by Optical Probe. 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 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, Rev. 2.0, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, Revision 2.0, February 2005, EPA-821-R-05-001.
18. National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 803, May 2000.
19. Method 8506, Biocinchoninate 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, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc.
22. Method 8008, 1,10-Phenanthroline Method for Iron 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 Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987)
25. Method 8507, Nitrogen, Nitrite Low Range, Diazotization Method for Water and Wastewater, 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. R.F. Addison and R. G. Ackman, Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography, *Journal of Chromatography*, Vol. 47(3), 421-426, 1970.
29. Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $\text{Na}_2\text{S}_2\text{O}_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
30. The 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.
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, 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 (*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 A and 1664B). 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 Revision B is the revised version of EPA Method 1664A. 2010 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, USEPA The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA-821-R-96-011) are recommended to preclude contamination at low-level, trace metal determinations.
44. Method OIA-1677, 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 Method 300.1, are published in USEPA. May 1994 Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111; or US EPA. August 1993 Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100. EPA Method 300.1 is 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 D6508-10, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte, 2010. 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. US 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 D6888-09 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, C.J. Patton and S.R. Crouch, Anal. Chem. (1977) 49, 464-469. These reaction parameters increase to pH >12.6 and 665 nm when salicylate is used as the color reagent--see, M.D. Krom, Analyst(1980) 105, 305-316.
61. If atomic absorption or ICP instrumentation is not available, the aluminon colorimetric method detailed in the 19th Edition of Standard Methods 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 (LDO) in Water and Wastewater, 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. USEPA.
69. Method 1627, Kinetic Test Method for the Prediction of Mine Drainage Quality, EPA-821-R-09-002. December 2011. USEPA.
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, Revision 9.0, March 2014, Method for Nitrate Reductase Nitrate-Nitrogen Analysis, 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.
78. The pH adjusted sample is to be adjusted to 7.6 for NPDES reporting purposes.
79. I-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			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
	HPLC	610	6440 B-05	D4657-92 (98)	
2. Acenaphthylene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
3. Acrolein	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-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
6. Benzene	GC	602	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
7. Benzidine	Spectro- photometric				See footnote 3, p.1.
	GC/MS	625.1 ⁵ , 1625B	6410 B-00		
	HPLC	605			
8. Benzo(a)anthracene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
9. Benzo(a)pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
10. Benzo(b)fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
11. Benzo(g,h,i)perylene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
12. Benzo(k)fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
13. Benzyl chloride	GC				See footnote 3, p. 130.
	GC/MS				See footnote 6, p. S102.
14. Butyl benzyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
15. bis(2-Chloroethoxy) methane	GC	611			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
16. bis(2-Chloroethyl) ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
17. bis(2-Ethylhexyl) phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
18. Bromodichloromethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
19. Bromoform	GC	601	6200 C-11		

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
20. Bromomethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
21. 4-Bromophenyl phenyl ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
22. Carbon tetrachloride	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
23. 4-Chloro-3-methyl phenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
24. Chlorobenzene	GC	601, 602	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
25. Chloroethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³
26. 2-Chloroethylvinyl ether	GC	601			
	GC/MS	624.1, 1624B			
27. Chloroform	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
28. Chloromethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
29. 2-Chloronaphthalene	GC	612			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
30. 2-Chlorophenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
31. 4-Chlorophenyl phenyl ether	GC	611			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
32. Chrysene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
33. Dibenzo(a,h)anthracene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
34. Dibromochloromethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³
35. 1,2-Dichlorobenzene	GC	601, 602	6200 C-11		
	GC/MS	624.1, 1625B	6200 B-11		See footnote 9, p. 27; O-4127-96 ¹³ , O-44436-16 ¹⁴
36. 1,3-Dichlorobenzene	GC	601, 602	6200 C-11		
	GC/MS	624.1, 1625B	6200 B-11		See footnote 9, p. 27 O-4127-96 ¹³ , O-44436-16 ¹⁴
37. 1,4-Dichlorobenzene	GC	601, 602	6200 C-11		
	GC/MS	624.1, 1625B	6200 B-11		See footnote 9, p. 27 O-4127-96 ¹³ , O-44436-16 ¹⁴
38. 3,3'-Dichlorobenzidine	GC/MS	625.1, 1625B	6410 B-00		

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
	HPLC	605			
39. Dichlorodifluoromethane	GC	601			
	GC/MS		6200 C-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
40. 1,1-Dichloroethane	GC	601	6200 B-11		
	GC/MS	624.1, 1624B	6200 C-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
41. 1,2-Dichloroethane	GC	601	6200 B-11		
	GC/MS	624.1, 1624B	6200 C-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
42. 1,1-Dichloroethene	GC	601	6200 B-11		
	GC/MS	624.1, 1624B	6200 C-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
43. trans-1,2-Dichloroethene	GC	601	6200 B-11		
	GC/MS	624.1, 1624B	2060 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
44. 2,4-Dichlorophenol	GC	604	6420 B-00.		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
45. 1,2-Dichloropropane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
46. cis-1,3-Dichloropropene	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
47. trans-1,3-Dichloropropene	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
48. Diethyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
49. 2,4-Dimethylphenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
50. Dimethyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
51. Di-n-butyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
52. Di-n-octyl phthalate	GC	606			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
53. 2, 4-Dinitrophenol	GC	604	6420 B-00		See footnote 9, p. 27
	GC/MS	625.1, 1625B	6410 B-00		
54. 2,4-Dinitrotoluene	GC	609			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
55. 2,6-Dinitrotoluene	GC	609			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
56. Epichlorohydrin	GC				See footnote 9, p. 27
	GC/MS				See footnote 9, p. 27
57. Ethylbenzene	GC	602	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
58. Fluoranthene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	
59. Fluorene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98)	

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran	GC/MS	1613B			
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran	GC/MS	1613B			
62. 1,2,3,4,6,7,8- Heptachloro- dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
63. Hexachlorobenzene	GC	612			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
64. Hexachlorobutadiene	GC	612			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27; O-4127-96 ¹³
65. Hexachlorocyclopentadiene	GC	612			
	GC/MS	625.1 ⁵ , 1625B	6410 B-00		See footnote 9, p. 27; O-4127-96 ¹³
66. 1,2,3,4,7,8-Hexachloro- dibenzofuran	GC/MS	1613B			
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			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27; O-4127-96 ¹³
74. Indeno(1,2,3-c,d) pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98).	
75. Isophorone	GC	609			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
76. Methylene chloride	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
77. 2-Methyl-4,6-dinitrophenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
78. Naphthalene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05		
79. Nitrobenzene	GC	609			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC			D4657-92 (98).	
80. 2-Nitrophenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
81. 4-Nitrophenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
82. N-Nitrosodimethylamine	GC	607			
	GC/MS	625.1 ⁵ , 1625B	6410 B-00		See footnote 9, p. 27
83. N-Nitrosodi-n-propylamine	GC	607			
	GC/MS	625.1 ⁵ , 1625B	6410 B-00		See footnote 9, p. 27
84. N-Nitrosodiphenylamine	GC	607			
	GC/MS	625.1 ⁵ , 1625B	6410 B-00		See footnote 9, p. 27

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
85. Octachlorodibenzofuran	GC/MS	1613B ¹⁰			
86. Octachlorodibenzo- <i>p</i> -dioxin	GC/MS	1613B ¹⁰			
87. 2,2'-oxybis(1-chloropropane) ¹²	GC	611			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
88. PCB-1016	GC	608.3			See footnote 3, p. 43; See footnote. 8
	GC/MS	625.1	6410 B-00		
89. PCB-1221	GC	608.3			See footnote 3, p. 43; See footnote 8
	GC/MS	625.1	6410 B-00		
90. PCB-1232	GC	608.3			See footnote 3, p. 43; See footnote 8.
	GC/MS	625.1	6410 B-00		
91. PCB-1242	GC	608.3			See footnote 3, p. 43; See footnote 8.
	GC/MS	625.1	6410 B-00		
92. PCB-1248	GC	608.3			See footnote 3, p. 43; See footnote 8.
	GC/MS	625.1	6410 B-00		
93. PCB-1254	GC	608.3			See footnote 3, p. 43; See footnote 8.
	GC/MS	625.1	6410 B-00		
94. PCB-1260	GC	608.3			See footnote 3, p. 43; See footnote 8.
	GC/MS	625.1	6410 B-00		
95. 1,2,3,7,8-Pentachloro- dibenzofuran	GC/MS	1613B			
96. 2,3,4,7,8-Pentachloro- dibenzofuran	GC/MS	1613B			
97. 1,2,3,7,8,-Pentachloro-dibenzo- <i>p</i> -dioxin	GC/MS	1613B			
98. Pentachlorophenol	GC	604	6420 B-00		See footnote 3, p. 140.
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
99. Phenanthrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98).	
100. Phenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
101. Pyrene	GC	610			
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
	HPLC	610	6440 B-05	D4657-92 (98).	
102. 2,3,7,8-Tetrachloro- dibenzofuran	GC/MS	1613B ¹⁰			
103. 2,3,7,8-Tetrachloro-dibenzo- <i>p</i> - dioxin	GC/MS	613, 625.1 ^{5a} , 1613B			
104. 1,1,2,2-Tetrachloroethane	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³
105. Tetrachloroethene	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
106. Toluene	GC	602	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
107. 1,2,4-Trichlorobenzene	GC	612			See footnote 3, p. 130.
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27;

Parameter ¹	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
					O-4127-96 ¹³ , O-44436-16 ¹⁴
108. 1,1,1-Trichloroethane	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
109. 1,1,2-Trichloroethane	GC	601	6200 C-11		See footnote 3, p. 130.
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
110. Trichloroethene	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
111. Trichlorofluoromethane	GC	601	6200 C-11		
	GC/MS	624.1	6200 B-11		O-4127-96 ¹³
112. 2,4,6-Trichlorophenol	GC	604	6420 B-00		
	GC/MS	625.1, 1625B	6410 B-00		See footnote 9, p. 27
113. Vinyl chloride	GC	601	6200 C-11		
	GC/MS	624.1, 1624B	6200 B-11		O-4127-96 ¹³ , O-44436-16 ¹⁴
114. Nonylphenol	GC/MS			D7065-17	
115. Bisphenol A (BPA)	GC/MS			D7065-17	
116. p-tert-Octylphenol (OP)	GC/MS			D7065-17	
117. Nonylphenol Monoethoxylate (NP1EO)	GC/MS			D7065-17	
118. Nonylphenol Diethoxylate (NP2EO)	GC/MS			D7065-17	
119. Adsorbable Organic Halides (AOX)	Adsorption and Coulometric Titration	1650 ¹¹			
120. Chlorinated Phenolics	In Situ Acetylation and GC/MS	1653 ¹¹			

Notes

- All parameters are expressed in micrograms per liter (ug/L) except for Method 1613 in which the parameters are expressed in picograms per liter (pg/L).
- The full text of Methods 601-613, 1624B, and 1625B, are given at Appendix A. The full text of Method 1613B is incorporated by reference. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B. These methods are available at: <https://www.epa.gov/cwa-methods>
- Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978.
- 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.
- Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.
- 5a Method 625.1 screening only.
- Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the Fifteenth Edition of Standard Methods (1981).
- Each 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 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the

unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

8. Organochlorine Pesticides and PCBs in Wastewater Using Empore Disk, 3M Corporation Revised 10/28/94.
9. USGS Method O-3116-87 from Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory--Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments U.S. Geological Survey, Open File Report 93-125.
10. Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meets the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities. 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 40 CFR part 136 and at <https://www.epa.gov/cwa-methods/approved-cwa-methods-organic-compounds>.
11. Method 1650, Adsorbable Organic Halides by Adsorption and Coulometric Titration. Revision C, 1997. US EPA. Method 1653, Chlorinated Phenolics in Wastewater by In Situ Acetylation and GCMS. Revision A, 1997. US EPA.
12. The 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.
13. Method 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.
14. Method 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}	Standard Methods	ASTM	Other
1. Aldrin	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
2. Ametryn	GC	507, 619			Notes 3, 6, O-3106-93 ⁹
	GC/MS	525.2			O-1121-91 ¹⁴
3. Aminocarb	TLC				Notes 3, 6,
	HPLC	632			
4. Atraton	GC	619			Notes 3, 6
5. Atrazine	GC	507, 619			Notes 3, 6, O-3106-93 ⁹
	HPLC/MS				O-2060-01 ¹²
	GC/MS	525.1, 525.2			O-1126-95 ¹¹
6. Azinphos methyl	GC	614, 622, 1657			Notes 3, 6
	GC/MS				O-1126-95 ¹¹
7. Barban	TLC				Notes 3, 6,
	HPLC	632			
8.alpha-BHC	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, 3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		O-1126-95 ¹¹
9.beta-BHC	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		
10.delta-BHC	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		
11. gamma-BHC (Lindane)	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 ⁸ .
	GC/MS	625.1	6410 B-00		3M0222 ⁸ , O-1126-95 ¹¹
12. Captan	GC	617	6630 B- 07	D3086-90, D5812-96 (02)	Note 3
13. Carbaryl	TLC				Notes 3, 6
	HPLC	531.1, 632			
	HPLC/MS	553			O-2060-01 ¹²
	GC/MS				O-1126-95 ¹¹
14. Carbophenothion.	GC	617	6630 B-07		Notes 4, 6
15. Chlordane	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
16. Chloroprotham	TLC				Notes 3, 6,
	HPLC	632			
17. 2,4-D	GC	615	6640 B-06		Notes 3, O-3104-83 ⁴ ,
	HPLC/MS				O-2060-01 ¹²
18. 4,4'-DDD	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
19. 4,4'-DDE	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, 4, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
20. 4,4'-DDT	GC	608.3, 617	6630 B- & C-00	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
21. Demeton-O	GC	614, 622			Notes 3, 6
22. Demeton-S	GC	614, 622			Notes 3, 6.
23. Diazinon	GC	507, 614, 622, 1657			Notes 3, 6, O-3104-83 ⁴
	GC/MS	525.2			O-1126-95 ¹¹

Parameter	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
24. Dicamba	GC	615			Note 3
	HPLC/MS				O-2060-01 ¹²
25. Dichlofenthion	GC	622.1			Notes 4, 6
26. Dichloran	GC	608.2, 608.3, 617	6630 B-07		Note 3
27. Dicofol.	GC	617		D3086-90, D5812-96 (02)	O-3104-83 ⁴
28. Dieldrin	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		O-1126-95 ¹¹
29. Dioxathion	GC	614.1, 1657			Notes 4, 6
30. Disulfoton	GC	507, 614, 622, 1657			Notes 3, 6
	GC/MS	525.2			O-1126-95 ¹¹
31. Diuron	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS	553			O-2060-01 ¹²
32. Endosulfan I	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		O-2002-01 ¹³
33. Endosulfan II	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		O-2002-01 ¹³
34. Endosulfan Sulfate	GC	608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, 3M0222 ⁸
	GC/MS	625.1 ⁵	6410 B-00		
35. Endrin	GC	608.3, 617, 1656	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	525.2, 625.1 ⁵	6410 B-00		
36. Endrin aldehyde	GC	608.3, 617	6630 C-07	D3086-90, D5812-96 (02)	3M0222 ⁸
	GC/MS	625.1 ⁵			O-2002-01 ¹³
37. Ethion	GC	614, 614.1, 1657			Notes 4, 6
	GC/MS				O-2002-01 ¹³
38. Fenuron	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS	553			O-2060-01 ¹²
39. Fenuron-TCA.	TLC				Notes 3, 6
	HPLC	632			
40. Heptachlor	GC	505, 508, 608.3, 617, 1656	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	525.1, 525.2, 625.1	6410 B-00		
41. Heptachlor epoxide	GC	608.3, 617	6630 C-07	D3086-90, D5812-96 (02)	O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	625.1	6410 B-00		
42. Isodrin.	GC	617			Note 6, O-3104-83 ⁴
43. Linuron	GC				Notes 3, 6
	HPLC	632			
	HPLC/MS	553			O-2060-01 ¹²
	GC/MS				O-1126-95 ¹¹
44. Malathion	GC	614, 1657	6630 B-07		Notes 3, 6
	GC/MS				O-1126-95 ¹¹
45. Methiocarb	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS				O-2060-01 ¹²

Parameter	Method	EPA ^{2,7}	Standard Methods	ASTM	Other
46. Methoxychlor	GC	505, 508, 608.2, 608.3, 617, 1656	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, O-3104-83 ⁴ , 3M0222 ⁸
	GC/MS	525.1, 525.2			O-1126-95 ¹¹
47. Mexacarbate	TLC				Notes 3, 6
	HPLC	632			
48. Mirex	GC	617	6630 B- & C-07	D3086-90, D5812-96(02)	Note 3, O-3104-83 ⁴
49. Monuron	TLC				Notes 3, 6
	HPLC	632			
50. Monuron-TCA	TLC				Notes 3, 6
	HPLC	632			
51. Nuburon	TLC				Notes 3, 6.
	HPLC	632			
	HPLC/MS				O-2060-01 ¹²
52. Parathion methyl	GC	614, 622, 1657	6630 B-07		Notes 3, 4
	GC/MS				O-1126-95 ¹¹
53. Parathion ethyl	GC	614	6630 B-07		Note 3
	GC/MS				O-1126-95 ¹¹
54. PCNB	GC	608.1, 608.3, 617	6630 B- & C-07	D3086-90, D5812-96 (02)	Note 3
55. Perthane	GC	617		D3086-90, D5812-96(02)	O-3104-83 ⁴
56. Prometron	GC	507, 619			Notes 3, 6, O-3106-93 ⁹
	GC/MS	525.2			O-1126-95 ¹¹
57. Prometryn	GC	507, 619			Notes 3, 6, O-3106-93 ⁹
	GC/MS	525.1, 525.2			O-2002-01 ¹³
58. Propazine	GC	507, 619, 1656			Notes 3, 6, O-3106-93 ⁹
	GC/MS	525.1, 525.2			
59. Propham	TLC				Notes 3, 6, O-3106-93 ⁹
	HPLC	632			
	HPLC/MS				O-2060-01 ¹²
60. Propoxur	TLC				Notes 3, 6
	HPLC	632			
61. Sebumeton	TLC				Notes 3, 6
	GC	619			
62. Siduron	TLC				Notes 3, 6
	HPLC	632			
	HPLC/MS				O-2060-01 ¹²
63. Simazine	GC	505, 507, 619, 1656			Notes 3, 6, O-3106-93 ⁹
	GC/MS	525.1, 525.2			O-1126-95 ¹¹
64. Strobane	GC	608.1, 608.3, 617	6630 B- & C-07		Note 3
65. Swep	TLC				Notes 3, 6
	HPLC	632			
66. 2,4,5-T	GC	615	6640 B-06		Note 3, O-3104-83 ⁴
67. 2,4,5-TP (Silvex)	GC	615	6640 B-06		Note 3, O-3104-83 ⁴
68. Terbutylazine	GC	619, 1656			Notes 3, 6
	GC/MS				O-2002-01 ¹³
69. Toxaphene	GC	505, 508, 608.3, 617, 1656	6630 B- & C-07	D3086-90, D5812-96 (02)	Notes 3, 8, O-3105-83 ⁴
	GC/MS	525.1, 525.2, 625.1	6410 B-00		
70. Trifluralin	GC	627, 1656	6630 B-07		Notes 3, O-3106-93 ⁹
	GC/MS	525.2			O-1126-95 ¹¹

Notes:

1. Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.
2. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B.
3. Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.
4. Methods for Analysis of Organic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).
5. The method may be extended to include a-BHC, g-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
6. Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency.' Supplement to the Fifteenth Edition of Standard Methods (1981).
7. Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608.3 and 625.1 and in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608.3 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 and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.
8. 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..
9. EPA Methods 608.1, 608.2, 614, 614.1, 615, 617, 619, 622, 622.1, 627, and 632 are found in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, EPA 821-R-92-002, April 1992, US EPA. EPA Methods 505, 507, 508, 525.1, 531.1 and 553 are in Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, 1993, US EPA. EPA Method 525.2 is in Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.0, 1995, US EPA. EPA methods 1656 and 1657 are in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821–R–93–010A, 1993, US EPA. Methods 608.3 and 625.1 are available at:
10. <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 Radiological Test Procedures

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

Notes:

1. ``Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980), U.S. Environmental Protection Agency, August 1980.
2. Fishman, M.J. and Brown, Eugene, ``Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).
3. The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the ``total".

Table IF. List of Approved Methods for Pharmaceutical Pollutants

Pharmaceuticals pollutants	CAS 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	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)	1624C/1666/624.1

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

Table IG. Test Methods for Pesticide Active Ingredients

EPA Survey Code / Pesticide name	CAS No.	EPA Method No.(s)
8 Triadimefon	43121-43-3	507/633/525.1/1656/625.1
12 Dichlorvos	62-73-7 1	657/507/622/525.1/525.2/625.1
16 2,4-D; 2,4-D Salts and Esters [2,4-Dichlorophenoxyacetic acid]	94-75-7	1658/515.1/615/515.2/555
17 2,4-DB; 2,4-DB Salts and Esters [2,4-Dichlorophenoxybutyric acid]	94-82-6	1658/515.1/615/515.2/555
22 Mevinphos	7786-34-7	1657/507/622/525.1/525.2/625.1
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) benzothiazole]	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	507/633/525.1/1656/608.3/625.1
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
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	508/608.2/525.1/1656/608.3/625.1
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-tetrachloroterephthalate]	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	1656/505/508/608/617/525.1/525.2/608.3/625.1
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	507/633.1/525.1/1656/608.3/625.1
133 Fenthion	55-38-9	1657/622/625.1
138 Glyphosate [N-(Phosphonomethyl) glycine]	1071-83-6	547
140 Heptachlor	76-44-8	1656/505/508/608/617/525.1/525.2/608.3/625.1
144 Isopropalin	33820-53-0	1656/627/608.3

EPA Survey Code / Pesticide name	CAS No.	EPA Method No.(s)
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 1	1656/1627/608.3 See footnote 1
182 Fensulfothion	15-90-2	1657/622/625.1
183 Disulfoton	298-04-4	1657/507/614/622/525.2/608.3/625.1
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	625/1625/515.2/555/515.1/ 525.1/525.2
208 Permethrin	52645-53-1	608.2/508/525.1/525.2/1656/1660/608.3 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
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	507/633/525.1/1656/608.3/625.1
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	1656/505/508/608/617/525.1/525.2/608.3/625.1
263 Merphos [Tributyl phosphorotrithioate]	150-50-5	1657/507/525.1/525.2/622/625.1
264 Trifluralin	1582-09-8	1656/508/617/627/525.1/525.2/608.3/625.1
268 Ziram [Zinc dimethyldithiocarbamate]	137-30-4	630/630.1

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 Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume I, EPA 821-R-93-010A, Revision I, August 1993, US EPA. EPA Methods 200.9 and 505 through 555 are available in Methods For The Determination of Nonconventional Pesticides In Municipal and Industrial Wastewater, Volume II, EPA 821-R-93-010B, August 1993, US EPA. The full text of Methods 608.3, 625.1, and 1625 are provided at Appendix A of this Part 136. The full text of Method 200.7 is provided at Appendix C of this Part 136. 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 IH. List of Approved Biological Methods for Ambient Water

Parameter and units	Method ¹	EPA	Standard Methods Online	ASTM ¹⁰ AOAC ¹¹ USGS ⁵	Other
<i>Bacteria:</i>					
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-G E-14 9221 F-14 ³²		
	Membrane filter (MF) ² , single step	p. 124 ³	9222 D-15 ²⁶	B-0050-85 ⁴	
2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 132³	9221 C E-06		
	MF², single step⁵	p. 124³	9222 D-06 ²⁷		
2. Coliform (total), number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114 ³	9221 B-14		
	MF ² , single step or two step	p. 108 ³	9222 B-15 ²⁷	B-0025-85 ⁴	
	MF² with enrichment	p. 111³	9222 B-15 ²⁷		
3. E. coli, number per 100 mL	MPN ^{5,7,13} multiple tube		9221 B.3-14 9221 F14 ^{10,12, 32}		
	multiple tube/ multiple well		9223 B-16 ¹¹	991.15 ¹⁰	Colilert ^{11,15} Colilert-18 ^{11,14,15}
	MF ^{2,5,6,7} two step, or	1103.1 ¹⁸	9222 B-15 9222 G-15 ¹⁷ 9213 D-07	D5392-93 ⁹	
	single step	1603 ¹⁹ 1604 ²⁰			mColiBue 24 ¹⁷ KwikCount EC ^{28, 29}
4. Fecal streptococci, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 139 ³	9230 B-13		
	MF ² , or	p. 136 ³	9230 C-13 ³⁰	B-0055-85 ⁴	
	Plate count	p. 143 ³			
5. Enterococci, number per 100 mL	MPN ^{5,7} multiple tube/ multiple well, or		9230 D-13	D6503-99 ⁹	Enterolert ^{11,21}
	MF ^{2,5,6,7} two step, or	1106.1 ²²	9230 C-13 ³⁰		
	single step, or	1600 ²³	9230 C-13 ³⁰		
	Plate count	p. 143 ³			
Protozoa:					
6. <i>Cryptosporidium</i> ²⁸	Filtration/IMS/FA	1622 ²⁴ 1623 ²⁵ 1623.1 ^{25,31}			
7. <i>Giardia</i> ²⁸	Filtration/IMS/FA	1623 ²⁵ 1623.1 ^{25,31}			

Notes

- The method must be specified when results are reported.
- A 0.45 um membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.
- Microbiological Methods for Monitoring the Environment, Water, and Wastes. EPA/600/8-78/017. 1978. US EPA
- 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
- ~~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.~~
- 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 number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.
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.
9. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. 1995. AOAC.
10. The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.
11. These tests are collectively known as defined enzyme substrate tests, ~~where, for example, a substrate is used to detect the enzyme beta-glucuronidase produced by E. coli.~~
12. After prior enrichment in a presumptive medium for total coliform using 9221B.1-14, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h \pm 3 h of incubation shall be submitted to 9221F-14. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 ug/mL of MUG may be used.
13. Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® or **Quanti-Tray® 2000**, and the MPN calculated from the table provided by the manufacturer.
14. Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and E. coli that provides results within 18 h of incubation at 35 C rather than the 24 h required for the Colilert® test and is recommended for marine water samples.
15. Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, **and Quanti-Tray® 2000** may be obtained from IDEXX Laboratories, Inc.
16. Laboratories, Inc.
17. A description of the mColiBlue24® test is available from Hach Company.
18. Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.
19. Method 1103.1: Escherichia coli (E. coli) in Water by Membrane Filtration Using membrane-Thermotolerant Escherichia coli Agar (mTEC). EPA-821-R-10-002. March 2010. USEPA.
20. Method 1603: Escherichia coli (E. coli) in Water by Membrane Filtration Using Modified membrane-Thermotolerant Escherichia coli Agar (Modified mTEC). EPA-821-R-14-010. September 2014. USEPA.
21. ~~Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. New Medium for the Simultaneous Detection of Total Coliform and Escherichia coli in Water. Appl. Environ. Microbiol. 59:3534-3544 and in Method 1604: Total Coliforms and Escherichia coli (E. coli) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). DC EPA 821-R-02-024. September 2002. USEPA.~~
22. A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc.
23. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA). EPA-821-R-09-015. December 2009. USEPA.
24. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-beta-D-Glucoside Agar (mEI). EPA-821-R-14-011. September 2014. USEPA.
25. Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of Cryptosporidium. Method 1622: Cryptosporidium in Water by Filtration/IMS/FA. EPA-821-R-05-001. December 2005. USEPA.
26. Methods 1623 **and 1623.1** use filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of Cryptosporidium and Giardia oocysts and cysts.. Method 1623. Cryptosporidium and Giardia in Water by Filtration/IMS/FA. EPA-821-R-05-002. December 2005 USEPA. **Method 1623.1: Cryptosporidium and Giardia in Water by Filtration/IMS/FA. EPA 816-R-12-001. January 2012. US EPA.**
27. On a monthly basis, at least ten **sheen** colonies from the medium 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 \pm 0.5 °C for 48 \pm 3 h is optional. As per the**
31. **Errata to the 23rd Edition of Standard Methods for the Examination of Water and Wastewater "Growth on a BHI agar plate incubated at 10 \pm 0.5 °C for 48 \pm 3 h is further verification that the colony belongs to the genus Enterococcus."**

32. 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.
33. 9221 F.2-2014 This procedure allows for simultaneous detection of *E. coli* and thermotolerant coliforms by adding inverted vials to EC-MUG; the inverted vials collect gas produced by thermotolerant coliforms.

[References, Sources and Table Citations]

b) Certain material is incorporated by reference into this part 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 is available for inspection at EPA's Water Docket, EPA West, 1301 Constitution Avenue NW., Room 3334, Washington, DC 20004, 202-566-2426, and is available from the sources listed below. It is also available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: <https://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 Page 106 of 293 IA, Note 3; Table IH, Note 3.

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

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

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

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

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

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

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

(ii) [Reserved]

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

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

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

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

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- (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.
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- (F) Method 353.2, Determination of Nitrate-Nitrite Automated Colorimetry. Revision 2.0. Table IB, Note 52.
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- (H) Method 375.2, Determination of Sulfate by Automated Colorimetry. Revision 2.0. Table IB, Note 52.
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- (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.
- (v) OFR 76–177, Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters. 1976. Table IE, Note 2.
- (vi) 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.
- (vix) 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.
- (ix) 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.
- (x) 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.
- (xi) 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.
- (xii) 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.
- (xiii) OFR 98–165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-Water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. Table IB, Note 50.
- (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.

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

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

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

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

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IA-Bacteria Tests:			
1-4 Coliform, total, fecal and E. coli	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 Enterocci	PA, G	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
7 Salmonella	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, 4°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

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
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, FP, G	None required	Analyze immediately
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	HNO ₃ or H ₂ SO ₄ to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze immediately within 15 minutes
31, 43. Kjeldahl and organic nitrogen	P, FP, G	Cool, < 6°C ¹⁸ , H ₂ SO ₄ to pH<2	28 days
Table IB-Metals⁷			
18. Chromium VI	P, FP, G	Cool, < 6°C ¹⁸ , pH = 9.3-9.7 ²⁰	24 hours 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 5mL/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
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 or H ₂ SO ₄ or H ₃ PO ₄ , to pH<2	28 days
44. Orthophosphate	P, FP, G	Filter immediately, Cool, < 6°C	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately within 15 minutes
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G only	Cool, < 6°C 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	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
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

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
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.⁸			
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 pH2 ⁹	14 days.
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 pH2 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 ⁵ ; Adjust 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 ⁵	7 days until extraction, 40 days after extraction
7, 38. Benzidines ¹¹	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	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	7 days until extraction; 40 days after extraction
54, 55, 75, 79. Nitroaromatics and isophorone ¹¹	G, FP -lined cap	Cool, < 6°C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ store in dark	7 days until extraction; 40 days after extraction
1,2,5,8-12,32,33, 58,59,74,78,99,101. Polynuclear aromatic hydrocarbons 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
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, field and lab preservation	G, FP-lined cap	Cool, < 6°C ¹⁸ , pH,9, 0.008% Na ₂ S ₂ O ₃ ⁵	1 year
Solids, mixed phase and tissue: field preservation	G, FP-lined cap	Cool, < 6°C ¹⁸	7 days
Tissue: field preservation	G, FP-lined cap	Cool, < 6°C ¹⁸	24 hours
Solids, mixed phase and tissue: lab preservation	G, FP-lined cap	Freeze, < -10°C	1 year
114 -118. Alkylated phenols	G	Cool, < 6 °C, H2SO4 to pH < 2	28 days until extraction, 40 days after extraction
119. Adsorbable Organic Halides (AOX)	G	Cool, < 6 °C, 0.008% Na2S2O3 HNO3 to pH < 2	Hold <i>at least</i> 3 days, but not more than 6 months
120. Chlorinated Phenolics		Cool, < 6 °C, 0.008% Na2S2O3 H2SO4 to pH < 2	30 days until acetylation, 30 days after acetylation

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table ID-Pesticides Tests:			
1-70. Pesticides ¹¹	G, FP-lined cap	Cool, < 6°C ¹⁸ , pH 5-9 ¹⁵	7 days until extraction; 40 days after extraction.
Table IE-Radiological Tests:			
1-5. Alpha, beta and radium	P, FP, G	HNO ₃ to pH<2	6 months.
Table IH-Bacteria Tests:			
1-4. Coliform, total, fecal	PA, G	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ^{22,23}
5. E. coli	PA, G	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
6. Fecal streptococci	PA, G	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
7. Enterocci	PA, G	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ ⁵	8 hours ²²
Table IH--Protozoa Tests:			
8 Cryptosporidium	LDPE; field filtration	1-10 °C	96 hours ²¹
9 Giardia	LDPE; field filtration	1-10 °C	96 hours ²¹

Table II Notes

- 1 P is polyethylene; FP is fluoropolymer (polytetrafluoroethylene (PTFE), or other fluoropolymer, unless stated otherwise in this Table II; G is glass; PA is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); LDPE is low density polyethylene.
- 2 Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler), refrigerate the sample at <6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at <6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample ; otherwise, preserve the grab sample, composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see Method 1664A (and the procedures at 141.24).
- 3 When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions of concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- 4 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analyses and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under 136.3. For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 136.3 for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of

collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0–6 °C, with minimum head space.

- 5 ASTM D7365–09a specifies treatment options for samples containing oxidants (e.g., chlorine). Also, Section 9060A of *Standard Methods for the Examination of Water and Wastewater* (20th and 21st editions) 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 interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365–09a(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), 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. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (regarding the analysis of benzidine).
- 12 If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
- 13 Extracts may be stored up to 30 days at < 0 °C.
- 14 For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ 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% Na₂S₂O₃.
- 16 Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 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 Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
- 18 Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “≤ °C” is used in place of the “4 °C” and “< 4 °C” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the <6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
- 19 An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

- 20 To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in 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).

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

(a) A written application for review of an alternate test procedure (ATP) for nationwide use may be made by letter via email or by hard copy in triplicate to the National ATP Program Coordinator (National Coordinator), Office of Science and Technology (4303T), Office of Water, U.S. Environmental Protection Agency, 1200 Pennsylvania Ave., NW, Washington, DC 20460. Any application for an ATP under this paragraph shall:

- (1) Provide the name and address of the responsible person or firm making the application.
- (2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an ATP is being requested.
- (3) Provide a detailed description of the proposed ATP, together with references to published or other studies confirming the general applicability of the ATP for the analysis of the pollutant(s) or parameter(s) in wastewater discharges from representative and specified industrial or other categories.
- (4) Provide comparability data for the performance of the proposed ATP compared to the performance of the reference method.

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

(c) Approval for nationwide use

- (1) After a review of the application and any additional analyses requested from the applicant, the National Coordinator will notify the applicant, in writing, of acceptance or rejection of the alternate test procedure for nationwide use in CWA programs. If the application is not approved, the National Coordinator will specify what additional information might lead to a reconsideration of the application, and notify the Regional ATP Coordinators of such rejection. Based on the National Coordinator's rejection of a proposed ATP an assessment of any approvals for limited uses for the unapproved method, the Regional ATP Coordinator or permitting authority may decide to withdraw approval of the method for limited use in the Region.
- (2) Where the National Coordinator has recommended approval of an applicant's request for nationwide use of an ATP, the National Coordinator will notify the applicant. The National Coordinator will also notify the Regional ATP Coordinators that they may consider approval of this ATP for limited use in their Regions based on the information and data provided in the application until the alternate test procedure is approved by publication in a final rule in the Federal Register.
- (3) EPA will propose to amend Part 136 to include the ATP in 136.3. EPA shall make available for review all the factual bases for its proposal, including any performance data submitted by the applicant and any available EPA analysis of those data.
- (4) Following public comment, EPA shall publish in the Federal Register a final decision on whether to amend Part 136 to include the ATP as an approved analytical method.
- (5) Whenever the National Coordinator has recommended approval of an applicant's ATP request for nationwide use, any person may request an approval of the method for limited use under 136.5 from the EPA Region.

136.5 Approval of Alternate Test Procedures for Limited Use

(a) Any person may request the Regional ATP Coordinator to approve the use of an ATP in the Region.

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

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

- (1) Provide the name and address of the applicant and the applicable ID number of the existing or pending permit and issuing agency for which use of the ATP is requested, and the discharge serial number.
- (2) Identify the pollutant or parameter for which approval of an ATP is being requested.
- (3) Provide justification for using testing procedures other than those specified in Tables IA through IH, or in the NPDES permit.
- (4) Provide a detailed description of the proposed ATP, together with references to published studies of the applicability of the ATP to the effluents in question.
- (5) Provide comparability data for the performance of the proposed ATP compared to the performance of the reference method.

(d) Approval for limited use

- (1) The Regional ATP Coordinator will review the application and notify the applicant and the appropriate State agency of approval or rejection of the use of the ATP. The approval may be restricted to use only with respect to a specific discharge or facility (and its laboratory) or, at the discretion of the Regional ATP Coordinator or permitting authority, to all discharger or facilities (and their associated laboratories) specified in the approval for the Region. If the application is not approved, the Regional ATP Coordinator or permitting authority shall specify what additional information might lead to a reconsideration of the application.
- (2) The Regional ATP Coordinator will forward a copy of every approval and rejection notification to the National Alternate Test Procedure Coordinator.

136.6 Method Modifications and Analytical Requirements.

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

- (1) Analyst means the person or laboratory using a test procedure (analytical method).
- (2) Chemistry of the Method means the reagents and reactions used in a test procedure that allow determination of the analyte(s) of interest in an environmental sample.
- (3) Determinative Technique means the way in which an analyte is identified and quantified (e.g., colorimetry, mass spectrometry).
- (4) Equivalent Performance means that the modified method produces results that meet the QC acceptance criteria of the approved method at this part.
- (5) Method-defined Analyte means an analyte defined solely by the method used to determine the analyte. Such an analyte may be a physical parameter, a parameter that is not a specific chemical, or a parameter that may be comprised of a number of substances. Examples of such analytes include temperature, oil and grease, total suspended solids, total phenolics, turbidity, chemical oxygen demand, and biochemical oxygen demand.
- (6) QC means "quality control."

(b) *Method Modifications.*

- (1) If the underlying chemistry and determinative technique in a modified method are essentially the same as an approved Part 136 method, then the modified method is an equivalent and acceptable alternative to the approved method provided the requirements of this section are met. However, those who develop or

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

- (2) Requirements. The modified method must meet or exceed performance of the approved method(s) for the analyte(s) of interest, as documented by meeting the initial and ongoing quality control requirements in the method.
- (i) Requirements for establishing equivalent performance. If the approved method contains QC tests and QC acceptance criteria, the modified method must use these QC tests and the modified method must meet the QC acceptance criteria with the following conditions:
 - (A) The analyst may only rely on QC tests and QC acceptance criteria in a method if it includes wastewater matrix QC tests and QC acceptance criteria (*e.g.*, matrix spikes) and both initial (start-up) and ongoing QC tests and QC acceptance criteria.
 - (B) If the approved method does not contain QC tests and QC acceptance criteria or if the QC tests and QC acceptance criteria in the method do not meet the requirements of this section, then the analyst must employ QC tests published in the "equivalent" of a Part 136 method that has such QC, or the essential QC requirements specified at 136.7, as applicable. If the approved method is from a compendium or VCSB and the QA/QC requirements are published in other parts of that organization's compendium rather than within the Part 136 method then that part of the organization's compendium must be used for the QC tests.
 - (C) In addition, the analyst must perform ongoing QC tests, including assessment of performance of the modified method on the sample matrix (*e.g.*, analysis of a matrix spike/matrix spike duplicate pair for every twenty samples), and analysis of an ongoing precision and recovery sample (*e.g.*, laboratory fortified blank or blank spike) and a blank with each batch of 20 or fewer samples.
 - (D) If the performance of the modified method in the wastewater matrix or reagent water does not meet or exceed the QC acceptance criteria, the method modification may not be used.
 - (ii) Requirements for documentation. The modified method must be documented in a method write-up or an addendum that describes the modification(s) to the approved method prior to the use of the method for compliance purposes. The write-up or addendum must include a reference number (*e.g.*, method number), revision number, and revision date so that it may be referenced accurately. In addition, the organization that uses the modified method must document the results of QC tests and keep these records, along with a copy of the method write-up or addendum, for review by an

auditor.

- (3) Restrictions. An analyst may not modify an approved Clean Water Act analytical method for a method-defined analyte. In addition, an analyst may not modify an approved method if the modification would result in measurement of a different form or species of an analyte. Changes in method procedures are not allowed if such changes would alter the defined chemistry (i.e., method principle) of the unmodified method. For example, phenol method 420.1 or 420.4 defines phenolics as ferric iron oxidized compounds that react with 4-aminoantipyrine (4-AAP) at pH 10 after being distilled from acid solution. Because total phenolics represents a group of compounds that all react at different efficiencies with 4-AAP, changing test conditions likely would change the behavior of these different phenolic compounds. An analyst may not modify any sample collection, preservation, or holding time requirements of an approved method. Such modifications to sample collection, preservation, and holding time requirements do not fall within the scope of the flexibility allowed at Part 136.6. Method flexibility refers to modifications of the analytical procedures used for identification and measurement of the analyte only and does not apply to sample collection, preservation, or holding time procedures, which may only be modified as specified in 136.3 (e).
- (4) Allowable Changes. Except as under *Restrictions* of this section, an analyst may modify an approved test procedure (analytical method) provided that the chemistry of the method or the determinative technique is not changed, and provided that the requirements of this section are met. If equal or better performance can be obtained with an alternative reagent, then it is allowed. A laboratory wishing to use these modifications must demonstrate acceptable method performance by performing and documenting all applicable initial demonstration of capability and ongoing QC tests and meeting all applicable QC acceptance criteria as described in Part 136.7. Some examples of the allowed types of changes, provided the requirements of this section are met include:
- (i) Changes between manual method, flow analyzer, and discrete instrumentation.
 - (ii) Changes in chromatographic columns or temperature programs.
 - (iii) Changes between automated and manual sample preparation, such as digestions, distillations, and extractions; in-line sample preparation is an acceptable form of automated sample preparation for CWA methods.
 - (iv) In general, ICP-MS is a sensitive and selective detector for metal analysis; however isobaric interference can cause problems for quantitative determination, as well as identification based on the isotope pattern. Interference reduction technologies, such as collision cells or reaction cells, are designed to reduce the effect of spectroscopic interferences that may bias results for the element of interest. The use of interference reduction technologies is allowed, provided the method performance specifications relevant to ICP-MS measurements are met.
 - (v) The use of EPA Method 200.2 or the sample preparation steps from EPA Method 1638, including the use of closed-vessel digestion, is allowed for EPA Method 200.8, provided the method performance specifications relevant to the ICP-MS are met.
 - (vi) Changes in pH adjustment reagents. Changes in compounds used to adjust pH are acceptable as long as they do not produce interference. For example, using a different acid to adjust pH in colorimetric methods.
 - (vii) Changes in buffer reagents are acceptable provided that the changes do not produce interferences.
 - (viii) Changes in the order of reagent addition are acceptable provided that the change does not alter the chemistry and does not produce an interference. For example, using the same reagents, but adding them in different order, or preparing them in combined or separate solutions (so they can be added separately), is allowed, provided reagent stability or method performance is equivalent or improved.
 - (vix) Changes in calibration range (provided that the modified range covers any relevant regulatory limit and the method performance specifications for calibration are met).
 - (x) Changes in calibration model. (A) Linear calibration models do not adequately fit calibration data with one or two inflection points. For example, vendor-supplied data acquisition and processing software on some instruments may provide quadratic fitting functions to handle such situations. If the calibration data for a particular analytical method routinely display quadratic character, using quadratic fitting functions may be acceptable. In such cases, the minimum number of calibrators for second order fits should be six, and in no case should concentrations be extrapolated for

instrument responses that exceed that of the most concentrated calibrator. Examples of methods with nonlinear calibration functions include chloride by SM4500-Cl-E-1997, hardness by EPA Method 130.1, cyanide by ASTM D6888 or OIA1677, Kjeldahl nitrogen by PAI-DK03, and anions by EPA Method 300.0. (B) As an alternative to using the average response factor, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

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

where:

x'_i = Calculated concentration at level i

x_i = Actual concentration of the calibration level i

n = Number of calibration points

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

(C) Using the RSE as a metric has the added advantage of allowing the same numerical standard to be applied to the calibration model, regardless of the form of the model.

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

- (xi) Changes in equipment such as equipment from a vendor different from the one specified in the method.
- (xii) The use of micro or midi distillation apparatus in place of macro distillation apparatus.
- (xiii) The use of prepackaged reagents.
- (xiv) The use of digital titrators and methods where the underlying chemistry used for the determination is similar to that used in the approved method.
- (xv) Use of selected ion monitoring (SIM) mode for analytes that cannot be effectively analyzed in full-scan mode and reach the required sensitivity. False positives are more of a concern when using SIM analysis, so at a minimum, one quantitation and two qualifying ions must be monitored for each analyte (unless fewer than three ions with intensity greater than 15% of the base peak are available). The ratio of each of the two qualifying ions to the quantitation ion must be evaluated and should agree with the ratio observed in an authentic standard within ± 20 percent. Analyst judgment must be applied to the evaluation of ion ratios because the ratios can be affected by co-eluting compounds present in the sample matrix. The signal-to-noise ratio of the least sensitive ion should be at least 3:1. Retention time in the sample should match within 0.05 minute of an authentic standard analyzed under identical conditions. Matrix interferences can cause minor shifts in retention time and may be evident as shifts in the retention times of the internal standards. The total scan time should be such that a minimum of eight scans are obtained per chromatographic peak.
- (xvi) If the characteristics of a wastewater matrix prevent efficient recovery of organic pollutants and prevent the method from meeting QC requirements, the analyst may attempt to resolve the issue by adding salts to the sample, provided that such salts do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control

- samples, and spiked samples that also contain such salts), and that all requirements of paragraph (b)(2) of this section are met. Samples having residual chlorine or other halogen must be dechlorinated prior to the addition of such salts.
- (xvii) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevent the analyst from meeting QC requirements, the analyst may attempt to resolve the issue by adding a inert surfactant that does not affect the chemistry of the method, such as Brij-35 or sodium dodecyl sulfate (SDS), provided that such surfactant does not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such surfactant) and that all requirements of paragraph (b)(1) and (b)(2) of this section are met. Samples having residual chlorine or other halogen must be dechlorinated prior to the addition of such surfactant.
 - (xviii) The use of gas diffusion (using pH change to convert the analyte to gaseous form and/or heat to separate an analyte contained in steam from the sample matrix) across a hydrophobic semi-permeable membrane to separate the analyte of interest from the sample matrix may be used in place of manual or automated distillation in methods for analysis such as ammonia, total cyanide, total Kjeldahl nitrogen, and total phenols. These procedures do not replace the digestion procedures specified in the approved methods and must be used in conjunction with those procedures.
 - (xix) Changes in equipment operating parameters such as the monitoring wavelength of a colorimeter or the reaction time and temperature as needed to achieve the chemical reactions defined in the unmodified CWA method. For example, molybdenum blue phosphate methods have two absorbance maxima, one at about 660 nm and another at about 880 nm. The former is about 2.5 times less sensitive than the latter. Wavelength choice provides a cost-effective, dilution-free means to increase sensitivity of molybdenum blue phosphate methods.
 - (xx) Interchange of oxidants, such as the use of titanium oxide in UV-assisted automated digestion of TOC and total phosphorus, as long as complete oxidation can be demonstrated.
 - (xxi) Use of an axially viewed torch with Method 200.7.
 - (xvi) Changes are allowed in purge-and-trap sample volumes or operating conditions. Some examples are:
 - (A) Changes in purge time and purge-gas flow rate. A change in purge time and purge-gas flow rate is allowed provided that sufficient total purge volume is used to achieve the required minimum detectable concentration and calibration range for all compounds. In general, a purge rate in the range 20-200 mL/min and a total purge volume in the range 240-880 mL are recommended.
 - (B) Use of nitrogen or helium as a purge gas, provided that the required sensitivities for all compounds are met.
 - (C) Sample temperature during the purge state. Gentle heating of the sample during purging (e.g., 40 °C) increases purging efficiency of hydrophilic compounds and may improve sample-to-sample repeatability because all samples are purged under precisely the same conditions.
 - (D) Trap sorbent. Any trap design is acceptable, provided that the data acquired meet all QC criteria.
 - (E) Changes to the desorb time. Shortening the desorb time (e.g., from 4 minutes to 1 minute) may not affect compound recoveries, and can shorten overall cycle time and significantly reduce the amount of water introduced to the analytical system, thus improving the precision of analysis, especially for water-soluble analytes. A desorb time of four minutes is recommended, however a shorter desorb time may be used, provided that all QC specifications in the method are met.
 - (F) Use of water management techniques is allowed. Water is always collected on the trap along with the analytes and is a significant interference for analytical systems (GC and GC/MS). Modern water management techniques (e.g., dry purge or condensation points) can remove moisture from the sample stream and improve analytical performance.
 - (xvii) If the characteristics of a wastewater matrix prevent efficient recovery of organic pollutants and prevent the method from meeting QC requirements, the analyst may attempt to resolve the issue by adding salts to the sample, provided that such salts do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control

- samples, and spiked samples that also contain such salts), and that all requirements of paragraph (b)(2) of this section are met. Samples having residual chlorine or other halogen must be dechlorinated prior to the addition of such salts.
- (xviii) If the characteristics of a wastewater matrix result in poor sample dispersion or reagent deposition on equipment and prevent the analyst from meeting QC requirements, the analyst may attempt to resolve the issue by adding a inert surfactant that does not affect the chemistry of the method, such as Brij-35 or sodium dodecyl sulfate (SDS), provided that such surfactants do not react with or introduce the target pollutant into the sample (as evidenced by the analysis of method blanks, laboratory control samples, and spiked samples that also contain such surfactants) and that all requirements of paragraph (b)(1) and (b)(2) of this section are met. Samples having residual chlorine or other halogens must be dechlorinated prior to the addition of such surfactants.
 - (xix) The use of gas diffusion (using pH change to convert the analyte to gaseous form and/or heat to separate an analyte contained in steam from the sample matrix) across a hydrophobic semi-permeable membrane to separate the analyte of interest from the sample matrix may be used in place of manual or automated distillation in methods for analysis such as ammonia, total cyanide, total Kjeldahl nitrogen, and total phenols. These procedures do not replace the digestion procedures specified in the approved methods and must be used in conjunction with those procedures.
 - (xx) Changes in equipment operating parameters such as the monitoring wavelength of a colorimeter or the reaction time and temperature as needed to achieve the chemical reactions defined in the unmodified CWA method. For example, molybdenum blue phosphate methods have two absorbance maxima, one at about 660 nm and another at about 880 nm. The former is about 2.5 times less sensitive than the latter. Wavelength choice provides a cost-effective, dilution-free means to increase sensitivity of molybdenum blue phosphate methods.
 - (xxi) Interchange of oxidants, such as the use of titanium oxide in UV-assisted automated digestion of TOC and total phosphorus, as long as complete oxidation can be demonstrated.
 - (xxii) Use of an axially viewed torch with Method 200.7.
 - (xxiii) When analyzing metals by inductively coupled plasma-atomic emission spectroscopy, inductively coupled plasma-mass spectrometry, and stabilized temperature graphite furnace atomic absorption, closed-vessel microwave digestion of wastewater samples is allowed as an alternative heating source for EPA Method 200.2, "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements" for the following elements: aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, selenium, silver, sodium, thallium, tin, titanium, vanadium, zinc, provided the performance specifications in the relevant determinative method are met. (Note that this list does not include Mercury.) Each laboratory determining total recoverable metals is required to operate a formal quality control (QC) program. The minimum requirements include initial demonstration of capability, method detection limit (MDL), analysis of reagent blanks, fortified blanks, matrix spike samples, and blind proficiency testing samples, as continuing quality control checks on performance. The laboratory is required to maintain performance records on file that define the quality of the data generated.

(c) The permittee must notify their permitting authority of the intent to use a modified method. Such notification should be of the form "Method xxx has been modified within the flexibility allowed in 40 CFR 136.6." The permittee may indicate the specific paragraph of §136.6 allowing the method modification. Specific details of the modification need not be provided, but must be documented in the Standard Operating Procedure (SOP) and maintained by the analytical laboratory that performs the analysis.

136.7 Quality Assurance and Quality Control

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

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

- a. Refer to and follow the QA/QC published in the “equivalent” EPA method for that parameter that has such QA/QC procedures;
- b. Refer to the appropriate QA/QC section(s) of an approved Part 136 method from a consensus organization compendium;
- c. Incorporate the following twelve quality control elements, where applicable, into the laboratory’s documented standard operating procedure (SOP) for performing compliance analyses when using an approved Part 136 method when the method lacks such QA/QC procedures. One or more of the twelve QC elements may not apply to a given method and may be omitted if a written rationale is provided indicating why the element(s) is/are inappropriate for a specific method.
 - (1) Demonstration of Capability (DOC);
 - (2) Method Detection Limit (MDL);
 - (3) Laboratory reagent blank (LRB), also referred to as method blank (MB);
 - (4) Laboratory fortified blank (LFB), also referred to as a spiked blank, or laboratory control sample (LCS);
 - (5) Matrix spike (MS) and matrix spike duplicate (MSD), or laboratory fortified matrix (LFM) and LFM duplicate, may be used for suspected matrix interference problems to assess precision;
 - (6) Internal standards (for GC/MS analyses), surrogate standards (for organic analysis) or tracers (for radiochemistry);
 - (7) Calibration (initial and continuing), also referred to as initial calibration verification (ICV) and continuing calibration verification (CCV);
 - (8) Control charts (or other trend analyses of quality control results);
 - (9) Corrective action (root cause analysis);
 - (10) QC acceptance criteria;
 - (11) Definitions of preparation and analytical batches that may drive QC frequencies; and
 - (12) Minimum frequency for conducting all QC elements.

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