Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

Note: A number of the references cited in this chapter are no longer in print. Copies of references which are out−of−print are available at any public library by inter−library loan.

NR 219.01 Purpose. The purpose of this chapter is to establish analytical test methods, preservation procedures, requirements for laboratories, and procedures applicable to effluent limitations for discharges from point sources as authorized by ss. 299.11 and 283.55 (1), Stats.

History: Cr. Register, August, 1976, No. 248, eff. 9−1−76; am. Register, April, 1986, No. 364, eff. 8−28−86; am. Register, June, 1986, No. 366, eff. 7−1−86; am. Register, April, 1988, No. 388, eff. 5−1−88; corrections made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.

NR 219.02 Applicability. (1) The procedures prescribed herein shall, except as provided in s. NR 219.06, be used in the determination of concentrations and quantities of pollutant parameters as required for:

(a) An application submitted to the department for a permit under ch. 283, Stats.

(b) Reports required to be submitted by dischargers in accordance with the conditions of issued permits.

(2) Section NR 219.06 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149.

History: Cr. Register, August, 1976, No. 248, eff. 9−1−76; am. Register, April, 1986, No. 364, eff. 8−28−86; am. (1) (intro.), Register, June, 1986, No. 366, eff. 7−1−86**;** correction in (1) (a) made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491; correction in (2) made under s. 13.93 (2m) (b) 7., Stats., Register October 2002 No. 562; correction in (2) made under s. 13.93 (2m) (b) 7., Stats., Register November 2004 No. 587.

NR 219.03 Definitions. As used in this chapter:

(1) "EPA" means the U.S. environmental protection agency.

(2) "Department" means the department of natural resources.

(3) "Sludge" is defined in ss. NR 204.03 (55) and 214.03 (34).

History: Cr. Register, August, 1976, No. 248, eff. 9−1−76; am. (1), (2), (3) and (4m), Register, January, 1978, No. 265, eff. 2−1−78; r. and recr. Register, June, 1986, No. 366, eff. 7−1−86; r. and recr. (1), r. (3) and (4), Register, November, 1992, No. 443, eff. 12−1−92: CR 04−033: cr. (3) Register November 2004 No. 587, eff. 12−1−04.

NR 219.04 Identification of test procedures. (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which wastewater analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. Parameters or pollutants, for which sludge analytical methods are approved, are listed together with test procedure descriptions and references in table EM. Metals samples digestion procedures and references are listed in table BM. The discharge values for the listed parameters shall be determined by one of the standard analytical test procedures identified in a table under this subsection or by an alternate test procedure established under ss. NR 219.05 and 149.12.

(2) SAMPLE PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to E are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to less than or equal to 6° C where required. All samples requiring thermal preservation at less than or equal to 6° C shall be cooled immediately after collection, and the required temperature maintained during shipping. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

(3) TEMPERATURE REPORTING PROCEDURES. Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to less than or equal to 6° C prior to shipping, and a temperature blank shall be submitted with the samples. Samples cooled during shipping with ice packs may not be recorded as received on ice. Samples may be recorded as received on ice only if solid ice is present in the cooler at the time the samples are received. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

(a) The temperature of an actual sample.

(b) The temperature of a temperature blank shipped with the samples.

(c) The temperature of the melt water in the shipping container.

(4) INCORPORATION BY REFERENCE. The materials in this section are incorporated by reference for the purposes of the permit program under ch. 283, Stats.

Note: Copies of the publications referenced in Tables A−F are available for inspection at the offices of the department of natural resources and the legislative reference bureau. Many of these materials are also available through inter−library loan.

History: Cr. Register, June, 1986, No. 366, eff. 7−1−86; r. and recr. Tables B and E, Register, April, 1988, No. 388, eff. 5−1−88; am.; r. and recr. Tables A to F, Register, November, 1992, No. 443, eff. 12−1−92; am. (1), am. Tables A to F, Register, April, 1994, No. 460, eff. 5−1−94; am. (1) and (2), Tables A to F, cr. (3), Register, February, 1996, No. 482, eff. 3−1−96; CR 02−019: am. Table B Register October 2002 No. 562, eff. 11−1−02; CR 04−033: r. and recr. Table A, Table B, Table BM, Table C, Table D, Table E, Table EM, and Table F, cr. Table ES Register November 2004 No. 587, eff. 12−1−04; CR 04−101: am. Table A Note 29 Register May 2005 No. 593, eff. 6−1−05; CR 08−076: am. (2) and (3) (intro.), cr. (4), r. and recr. Table A, B, C to EM and F Register May 2009 No. 641, eff. 6−1−09; **correction to Table B Parameter No. 41 made under s. 13.92 (4) (b) 7., Stats., Register September 2009 No. 645.**

Table A
List of Approved Biological Test Procedures

Parameter and Units	Analytical Technology[1]	EPA	Standard Methods[4]	Standard Methods Online[12]	AOAC, ASTM, USGS	Other
Bacteria:						
1. Coliform (fecal), number per 100 mL or number per gram dry weight	Most Probable Number (MPN), 5 tube 3 dilution, or	p. 132[3], 1680[12,14], 1681[12,19]	9221 C or E [18th, 19th, 20th, 21st]	9221 C–99 or 9221 E–99		
	Membrane filter (MF)[2], single step	p. 124[3]	9222 D [18th, 19th, 20th, 21st]	9222 D–97	B–0050–85[5]	
	MPN, multiple tube/multiple well		9223 B [18th, 19th, 20th, 21st]	9223 B–97	991.15[11]	Colilert® [13,17], Colilert–18® [13,16,17]
2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 132[3]	9221 C or E [18th, 19th, 20th, 21st]	9221 C–99 or 9221 E–99		
	MPN, multiple tube/multiple well, or		9223 B [18th, 19th, 20th, 21st]	9223 B–97	991.15[11]	Colilert® [13,17], Colilert–18® [13,16,17]
	MF[2], single step	p. 124[3]	9222 D [18th, 19th, 20th, 21st]	9222 D–97		
3. Coliform (total), in number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B [18th, 19th, 20th, 21st]	9221 B–99		
	MF[2], single step or two step	p. 108[3]	9222 B [18th, 19th, 20th, 21st]	9222 B–97	B–0025–85[5]	
4. Coliform (total), in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B [18th, 19th, 20th, 21st]	9221 B–99		
	MF[2] with enrichment	p. 111[3]	9222 (B+B. 5c) [18th, 19th, 20th, 21st]	9222 (B+B. 5c)–97		
5. *E. coli*, number per 100 mL[20]	MPN[7,9,15] multiple tube		9221 B.1 or 9221F [18th, 19th, 20th][22,25]	9221 B.1 or F–99[22,25]		
	MPN, multiple tube/multiple well		9223 B [18th, 19th, 20th, 21st][13]	9223 B–97[13]	991.15[11]	Colilert® [13,17], Colilert–18® [13,16,17]
	MF[2,6,7,8,9] two step, or	1103.1[27]	9222 B [18th, 19th, 20th, 21st][26], 9222 G [18th, 19th, 20th][26], 9213 D [18th, 19th, 20th]	9222 B–97[26] or G–97[26]	D5392–93[10]	
	MF[2, 6, 7, 8, 9] single step	1603[21], 1604[28]				mColiBlue–24® [18]
6. Fecal streptococci, number per 100 mL	MPN, 5 tube, 3 dilution,	p. 139[3]	9230 B [18th, 19th, 20th, 21st]	9230 B–93		
	MF[2], or	P. 136[3]	9230 C [18th, 19th, 20th, 21st]	9230 C–93	B–0055–85[5]	
	Plate count	p. 143[3]				

Table A (Continued)
List of Approved Biological Test Procedures

Parameter and Units	Analytical Technology[1]	EPA	Standard Methods[4]	Standard Methods Online[12]	AOAC, ASTM, USGS	Other
7. Enterococci, number per 100 mL[20]	MPN[7,9], multiple tube,		9230 B [18th, 19th, 20th, 21st]	9230 B–93		
	MPN, multiple tube/multiple well,				D6503–99[10]	Enterolert® [13, 23]
	MF[2], two step,	1106.1[29]	9230 C [18th, 19th, 20th, 21st]	9230 C–93	D5259–92[10]	
	MF[2,6,7,8,9] single step, or	1600[24]				
	Plate count	p. 143[3]				
Protozoa:						
8. Cryptosporidium	Filtration/IMS/FA	1622[30], 1623[31]				
9. Giardia	Filtration/IMS/FA	1623[31]				
Aquatic Toxicity:						
10. Toxicity, acute, fresh water organisms, percent effluent	Ceriodaphnia, 48–h static–renewal mortality					Note 32
	Fathead minnow, 96–h static renewal mortality, or 96–h flow–through mortality					Note 32
11. Toxicity, chronic, fresh water organisms, percent effluent	Ceriodaphnia survival and reproduction					Note 32
	Fathead minnow larval survival and growth					Note 32

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

[2] A 0.45 ìm 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] U.S. EPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH, EPA/600/8–78/017.

[4] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[5] USGS. 1989. U.S. Geological Survey Techniques of Water–Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, VA.

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

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

[8] When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple–tube technique to demonstrate applicability and comparability of results.

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

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

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

[12] "Standard Methods for the Examination of Water and Wastewater On–Line", Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006. Subscription service available at: http:/www.standardmethods.org.

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

[14] U.S. EPA. July 2006. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple–Tube Fermentation Using Lauryl–Tryptose Broth (LTB) and EC Medium. US Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–012.

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

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

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

[18] A description of the mColiBlue24® test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.
[19] U.S. EPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple−Tube Fermentation using A–1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–013.
[20] Recommended for enumeration of target organism in wastewater effluent.
[21] U.S. EPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane−Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–011.
[22] 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.
[23] A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.
[24] U.S. EPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane−Enterococcus Indoxyl−b−D−Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–009.
[25] After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 ìg/mL of MUG may be used.
[26] Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA–MUG media.
[27] U.S. EPA. July 2006. Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane−Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–010.
[28] U.S. EPA, September 2002, Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA 821–R–02–024.
[29] U.S. EPA. July 2006. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane−Enterococcus−Esculin Iron Agar (mE–EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–008.
[30] 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*. U.S. EPA. 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–026.
[31] Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. U.S. EPA. 2001. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–01–025.
[32] Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 2nd Edition," Wisconsin Department of Natural Resources, 2004. This publication is available for inspection at the offices of the Department of Natural Resources and the Legislative Reference Bureau. Copies are available from the Department of Natural Resources, Bureau of Science Services, P.O. Box 7921, Madison, WI 53707.

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
1. Acidity, as $CaCO_3$, mg/L:	Electrometric or phenolphthalein endpoint			2310 B(4a) [18th, 19th, 20th, 21st]	2310 B(4a)–97	D1067–92, 02, 06	I–1020–85[2]	
2. Alkalinity, as $CaCO_3$, mg/L:	Electrometric or colorimetric titration to pH 4.5, manual, or			2320 B [18th, 19th, 20th, 21st]	2320 B–97	D1067–92, 02, 06	I–1030–85[2]	973.43[3]
	automatic	310.2, (Rev. 1974)[1]					I–2030–85[2]	
3. Aluminum–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99		I–3051–85[2]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99			
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120B [21st]	3120 B–99		I–4471–97[2]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP), or					D4190–94, 99, 03		Note 43
	Colorimetric (Eriochrome cyanine R)			3500–Al D [18th, 19th], 3500 Al B [20th, 21st]	3500–Al B–01			
4. Ammonia (as N), mg/L:	Manual distillation (at pH 9.5) followed by:	350.1, Rev. 2.0 (1993)		4500– NH_3 B [18th, 19th, 20th, 21st]	4500–NH_3 B–97			973.49[3]
	Titration			4500– NH_3 E [18th], 4500–NH_3 C [19th, 20th, 21st]	4500–NH_3–C–97			
	Electrode			4500– NH_3 F or G [18th], 4500–NH_3 D or E [19th, 20th, 21st]	4500–NH_3_D or E–97	D1426–98, 03 (B)		
	Automated phenate	350.1, Rev. 2.0, (1993)[70]		4500– NH_3 H [18th], 4500–NH_3 G [19th, 20th, 21st]	4500–NH_3 G–97		I–4523–85[2]	
	Automated electrode, or							Note 15
	Ion chromatography					D6919–03		

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
5. Antimony–Total[9], ug/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99			
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
6. Arsenic–Total[9], ug/L:	Digestion[9,11,45] followed by:							
	AA gaseous hydride		7061A	3114 B[10] [18th, 19th, 21st]	3114 B 4.d.–97	D2972–97, 03 (B)	I–3062–85[2]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D2972–97, 03 (C)	I–4063–98[59]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Colorimetric (SDCC)			3500–As C [18th, 19th], 3500–As B [20th, 21st]	3500–As B–97	D2972–97, 03(A)	I–3060–85[2]	
7. Barium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99		I–3084–85[2]	
	AA graphite furnace (GFAA)		7010	3113 B [18th, 19th, 21st]	3113 B–99	D4382–95, 02		
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)							Note 43

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
8. Beryllium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99	D3645–93 (88), 03 (A)	I–3095–85[2]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D3645–93 (88), 03 (B)		
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP), or					D4190–94, 99, 03		Note 43
	Colorimetric (Aluminon)			3500–Be D [18th, 19th]				
9. Biochemical Oxygen Demand (BOD_5), mg/L:	Dissolved oxygen depletion			5210 B [18th, 19th, 20th, 21st]	5210 B–01		I–1578–78[16]	973.443[3], p17[16]
10. Boron[46], mg/L:	Colorimetric (Curcumin)			4500–B B [18th, 19th, 20th, 21st]	4500–B B–00		I–3112–85[2]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)[11,45]	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)[11,45]					D4190–94, 99, 03		Note 43
11. Bromide, mg/L:	Titrimetric					D1246–95, 99 (C)	I–1125–85[2]	p. S44[18]
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev 2[64]

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
12. Cadmium–Total[9] mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D3557–95, 02 (A or B)	I–3135–85[2] or I–3136–85[2]	974.27[3], p. 37[17]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D3557–95, 02 (D)	I–4138–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–1472–85[2] or I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
	Voltammetry[19], or					D3557–95, 02 (C)		
	Colorimetric (Dithizone)			3500–Cd D [18th, 19th]				
13. Calcium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99	D511–93, 03 (B)	I–3152–85[2]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
	Titrimetric (EDTA), or			3500–Ca D [18th, 19th], 3500–Ca B [20th, 21st]	3500–Ca B–97	D511–93, 03 (A)		
	Ion chromatography					D6919–03		
14. Carbonaceous Biochemical Oxygen Demand ($CBOD_5$)[20], mg/L:	Dissolved oxygen depletion with nitrification inhibitor			5210 B [18th, 19th, 20th, 21st]	5210 B–01			
15. Chemical Oxygen Demand (COD), mg/L:	Titrimetric, or	410.3, (Rev. 1978)[1]		5220 C [18th, 19th, 20th, 21st]	5220 C–97	D–1252–95, 00, 06 (A)	I–3560–85[2]	973.46[3] and p.17[17]
	Spectrophotometric, manual or automatic	410.4, Rev 2.0 (1993)		5520 D [18th, 19th, 20th, 21st]	5220 D–97	D1252–95, 00, 06 (B)	I–3561–85[2]	Notes 21, 22

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
16. Chloride, mg/L:	Titrimetric (Silver nitrate) or		9253	4500–Cl^- B [18th, 19th, 20th, 21st]	4500–Cl^- B–97	D512–89 (99), 04 (B)	I–1183–85[2]	
	Colorimetric; manual or,						I–1187–85[2]	
	automated (Ferricyanide)		9250	4500–Cl^- E [18th, 19th, 20th, 21st]	4500–Cl^- E–97		I–2187–85[2]	
	Potentiometric titration			4500–Cl^- D [18th, 19th, 20th, 21st]	4500–Cl^- D–97			
	Ion selective electrode					D512–89 (99), 04 (C)		
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev. 2[64]
17. Chlorine, Total Residual, mg/L:	Amperometric direct			4500–Cl D [18th, 19th, 20th, 21st]	4500–Cl D–00	D1253–86 (96), 03		
	Amperometric direct (low level)			4500–Cl E [18th, 19th, 20th, 21st]	4500–Cl E–00			
	Iodometric direct			4500–Cl B [18th, 19th, 20th, 21st]	4500–Cl B–00			
	Back titration either end–point[23]			4500–Cl C [18th, 19th, 20th, 21st]	4500–Cl C–00			
	DPD–FAS			4500–Cl F [18th, 19th, 20th, 21st]	4500–Cl F–00			
	Spectrophotometric, DPD, or			4500–Cl G [18th, 19th, 20th, 21st]	4500–Cl G–00			
	Ion selective electrode							Note 24
18. Chromium VI, dissolved, ug/L:	0.45 micron filtration followed by:							
	AA chelation–extraction		7197	3111 C [18th, 19th, 21st]	3111 C–99		I–1232–85[2]	
	Ion chromatography, or	218.6, Rev. 3.3 (1994)	7199	3500–Cr E [18th, 19th], 3500–Cr C [20th, 21st]	3500–Cr C–01	D5257–97, 03		993.23[3]
	Colorimetric (Diphenylcarbazide)		7196* 7196A*	3500–Cr D [18th, 19th], 3500–Cr B [20th, 21st]	3500–Cr B–01	D1687–92, 02 (A)	I–1230–85[2]	

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
19. Chromium–Total[9], mg/L:	Digestion[9, 11, 45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99	D1687–92, 02 (B)	I–3236–85[2]	974.27[3]
	AA chelation–extraction			3111 C [18th, 19th, 21st]	3111 C–99			
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D1687–92, 02 (C)	I–3233–93[56]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP), or					D4190–94, 99, 03		Note 43
	Colorimetric (Diphenylcarbazide)			3500–Cr D [18th, 19th], 3500–Cr B [20th, 21st]	3500–Cr B–01			
20. Cobalt–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D3558–94, 03 (A or B)	I–3239–85[2]	p 37[17]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D3558–94, 03 (C)	I–4243–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
21. Color, platinum cobalt units or dominant wavelength, hue, luminance, purity:	Colorimetric (ADMI)			2120 E [18th, 19th, 20th, 21st]				Note 26
	(Platinum cobalt), or			2120 B [18th, 19th, 20th, 21st]	2120 B–01		I–1250–85[2]	
	Spectrophotometric			2120 C [18th, 19th, 20th, 21st]				

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
22. Copper–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D1688–95, 02 (A or B)	I–3270–85[2] or I–3271–85[2]	974.37[3], p 37[17]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D1688–95, 02 (C)	I–4274–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
	Colorimetric (Neocuproine), or			3500–Cu D [18th, 19th], 3500–Cu B [20th, 21st]	3500–Cu B–99			
	(Bicinchoninate)			3500–Cu E [18th, 19th], 3500–Cu C [20th, 21st]	3500–Cu C–99			Note 27
23. Cyanide, Total, ug/L:	Automated Distillation and Colorimetry							Kelada–01[65]
	Manual distillation with $MgCl_2$ followed by:	335.4, Rev. 1.0 (1993)[67]	9010B, 9010C	4500–CN^- C [18th, 19th, 20th, 21st]		D2036–98, 06(A)		10–204–00–1–X[66]
	Titrimetric		9014	4500–CN^- D [18th, 19th, 20th, 21st]	4500–CN^- D–99			p.22[17]
	Spectrophotometric, manual		9014	4500–CN^- E [18th, 19th, 20th, 21st]	4500–CN^- E–99	D2036–98, 06 (A)	I–3300–85[2]	
	Automated[28], or	335.4, Rev. 1.0 (1993)[67]	9012A, 9012B				I–4302–85[2]	10–204–00–1–X[66]
	Ion selective electrode			4500–CN^- F [18th, 19th, 20th, 21st]	4500–CN^- F–99	D2036–98, 06 (A)		

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
24. Cyanide, Available, ug/L:	Cyanide Amenable to Chlorination (CATC); Manual distillation with $MgCl_2$ followed by Titrimetric or Spectro-photometric		9010B, 9010C, 9014	4500–CN^- G [18th, 19th, 20th, 21st]	4500–CN^- G–99	D2036–98, 06 (B)		
	Flow injection and ligand exchange, followed by amperometry[71], or					D6888–04		OIA–1677[54]
	Automated distillation and colorimetry		9012A					Kelada–01[65]
25. Fluoride–Total, mg/L:	Manual distillation[14] followed by:			4500–F^- B [18th, 19th, 20th, 21st]	4500–F^- B–97			
	Electrode, manual			4500–F^- C [18th, 19th, 20th, 21st]	4500–F^- C–97	D1179–93, 99, 04 (B)		
	Automated						I–4327–85[2]	
	Colorimetric (SPADNS)			4500–F^- D [18th, 19th, 20th, 21st]	4500–F^- D–97	D1179–93, 99, 04 (A)		
	Automated complexone			4500–F^- E [18th, 19th, 20th, 21st]	4500–F^- E–97			
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev. 2[64]
26. Gold–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	231.2, Rev. 1978[1]						
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
27. Hardness, Total as $CaCO_3$, mg/L:	Automated colorimetric	130.1, (Issued 1971)[1]						
	Titrimetric (EDTA), or			2340 B or C [18th, 19th, 20th, 21st]	2340 B or C–97	D1126–86 (92), 02	I–1338–85[2]	973.52 B[3]
	Ca plus Mg as their carbonates by inductively coupled plasma–atomic emission spectrometry (ICP) or AA direct aspiration (See Parameters 13 and 33)							
28. Hydrogen ion (pH), pH units:	Electrometric measurement or		9040C	4500–H^+ B [18th, 19th, 20th, 21st]	4500–H^+ B–00	D1293–84 (90), 99 (05) (A or B)	I–1586–85[2]	973.41[3]
	Automated electrode	150.2, (Dec. 1982)[1]					I–2587–85[2]	Note 29
29. Iridium–Total[9], ug/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	235.2, (Issued 1978)[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
30. Iron– Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D1068–96, 03, 05 (A or B)	I–3381–85[2]	974.27[3]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D1068–96, 03, 05 (C)		
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP), or					D4190–94, 99, 03		Note 43
	Colorimetric (Phenanthroline)			3500–Fe D [18th, 19th], 3500–Fe B [20th, 21st]	3500–Fe B–97	D1068–96, 03, 05 (D)		Note 30
31. Kjeldahl Nitrogen–Total[12], (as N), mg/L:	Digestion and distillation[28] followed by:			4500–N_{org} B or C [18th, 19th, 20th, 21st] and 4500–NH_3 B [18th, 19th, 20th, 21st]	4500–N_{org} B or C–97 and 4500–NH_3 B–97	D3590–89, 02 (06) (A)		
	Titration			4500– NH_3 E [18th], 4500–NH_3 C [19th, 20th, 21st]	4500–NH_3 C–97	D3590–89, 02 (06) (A)		973.48[3]
	Electrode			4500– NH_3 F or G [18th], 4500–NH_3 D or E [19th, 20th, 21st]	4500–NH_3 D or E–97			
	Automated phenate colorimetric	351.1, (Rev. 1978)[1]					I–4551–78[16]	
	Semi–automated block digestor colorimetric	351.2, Rev. 2.0 (1993)				D3590–89, 02 (06) (B)	I–4515–91[55]	
	Manual or block digestor potentiometric					D3590–89, 02 (06) (A)		
	Block digestor, followed by auto distillation and titration, or							Note 48
	Flow injection gas diffusion							Note 49

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
32. Lead–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D3559–96, 03 (A or B)	I–3399–90[2]	974.27[3]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D3559–96, 03 (D)	I–4403–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
	Voltammetry, or					D3559–96, 03 (C)		
	Colorimetric (Dithizone)			3500–Pb D [18th, 19th], 3500–Pb B [20th, 21st]	3500–Pb B–97			
33. Magnesium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99	D511–93, 03 (B)	I–3447–85[2]	974.27[3]
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
	Gravimetric, or			3500–Mg D [18th, 19th]				
	Ion chromatography					D6919–03		

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
34. Manganese–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99	D858–95, 02 (A or B)	I–3454–85[2]	974.27[3]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D858–95, 02 (C)		
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
	Colorimetric (Persulfate), or			3500–Mn D [18th, 19th], 3500–Mn B [20th, 21st]	3500–Mn B–99			920.203[3]
	(Periodate)							Note 31
35. Mercury–Total[9], ug/L:	Cold vapor, manual or	245.1	7470A	3112 B [18th, 19th, 21st]	3112 B–99	D3223–97, 02	I–3462–85[2]	977.22[3]
	Automated	245.2						
	Purge and trap cold vapor atomic fluorescence spectrometry[53] (CVAFS), or	1631E[52]						
	Cold vapor atomic fluorescence spectrometry (CVAFS)[53]	245.7[69]						
36. Molybdenum– Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99		I–3490–85[2]	
	AA graphite furnace (GFAA)		7010	3113 B [18th, 19th, 21st]	3113 B–99		I–3492–96[57]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)							Note 43

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
37. Nickel–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D1886–90, 94 (98), 03 (A or B)	I–3499–85[2]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99	D1886–90, 94 (98), 03 (C)	I–4503–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
38. Nitrate (as N), mg/L:	Ion chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)		4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	Ion selective electrode, or			4500–NO_3 D [18th, 19th, 20th, 21st]	4500–NO_3 D–00			
	CIE/UV, or					D6508–00 (05)		D6508, Rev 2[64]
	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 [18th, 19th, 20th, 21st]	4500–NO_3 E–00	D3867–99, 04(B)		
	Automated	353.2, Rev. 2.0 (1993)		4500–NO_3 F [18th, 19th, 20th, 21st]	4500–NO_3 F–00	D3867–99, 04(A)	I–4545–85[2]	
	Automated hydrazine			4500–NO_3 H [18th, 19th, 20th, 21st]	4500–NO_3 H–00			
	Ion chromatography[34], or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev 2[64]

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
40. Nitrite (as N), mg/L:	Spectrophotometric, Manual			4500–NO_2 B [18th, 19th, 20th, 21st]	4500–NO_2 B–00			Note 33
	Automated (Diazotization)						I–4540–85[2]	
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)		4500–NO_3 F [18th, 19th, 20th, 21st]	4500–NO_3 F–00	D3867–99, 04(A)	I–4545–85[2]	
	Manual (*bypass cadmium reduction)			4500–NO_3 E [18th, 19th, 20th, 21st]	4500–NO_3 E–00	D3867–99, 04(B)		
	Ion chromatography[34], or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev 2[64]
41. Oil and Grease– Total recoverable[47], mg/L:	Hexane extractable material (HEM), or	1664A[50]			5520 B–01			
	Silica–gel treated HEM (SGT–HEM); Silica gel treatment and gravimetry	1664A[50]						
42. Organic Carbon, Total (TOC), mg/L:	Combustion or oxidation			5310 B, C or D [18th, 19th, 20th, 21st]	5310 B, C or D–00			973.47[3], p.14[32]
43. Organic Halides, Adsorbable (AOX), ug/L:	Adsorption and coulometric titration	1650[51]						
44. Organic Nitrogen (as N), mg/L:	Kjeldahl nitrogen, total (Parameter 31) minus Ammonia nitrogen (Parameter 4)							

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
45. Orthophosphate (as P), mg/L:	Ascorbic acid method:							
	Automated	365.1, Rev. 2.0 (1993)[1]		4500–P^{-} F [18th, 19th, 20th, 21st]			I–4601–85[2]	973.56[3]
	Manual single reagent			4500–P^{-} E [18th, 19th, 20th, 21st]		D515–88 (A)		973.55[3]
	Manual two reagent	365.3, (Issued 1978)						
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev. 2[64]
46. Osmium–Total[9], ug/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99			
	AA furnace	252.2, (Issued 1978)[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]		3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
47. Oxygen, Dissolved, mg/L:	Winkler (azide modification)			4500–O C [18th, 19th, 20th, 21st]	4500–O C–01	D888–92, 03, 05 (A)	I–1575–78[16]	973.45B[3]
	Electrode, or			4500–O G [18th, 19th, 20th, 21st]	4500–O G–01	D888–92, 03, 05 (B)	I–1576–78[16]	
	Luminescence					D888–05 (C)		Note 72

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
48. Palladium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 B [18th, 19th, 21st]	3111 B–99			p. S27[18]
	AA graphite furnace (GFAA)	253.2, (Issued 1978)[1]	7010					p. S28[18]
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]		3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
49. Phenols, ug/L:	Manual distillation[35] followed by:	420.1, (Rev. 1978)[1]						Note 36
	Colorimetric (4AAP) manual, or	420.1, (Rev. 1978)[1]	9065					Note 36
	Automated	420.4, Rev. 1.0 (1993)	9066					
50. Phosphorus (elemental) mg/L:	Gas–liquid chromatography							Note 37
51. Phosphorus–Total, mg/L:	Persulfate digestion[28] followed by:			4500–P B.5 [18th, 19th, 20th, 21st]				973.55[3]
	Manual	365.3, (Issued 1978)[1]		4500–P F [18th, 19th, 20th, 21st]		D515–88 (A)	I–4600–85[2]	973.56[3]
	Automated ascorbic acid reduction, or	365.1, Rev. 2.0, (1993)		4500–P E [18th, 19th, 20th, 21st]				
	Semi–automated block digestor	365.4, (Issued 1974)[1]				D515–88 (B)	I–4610–91[58]	

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
52. Platinum–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	255.2[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]		3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
53. Potassium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99		I–3630–85[2]	973.53[3]
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
	Flame photometric			3500–K D [18th, 19th], 3500–K B [20th, 21st]	3500–K B–99			
	Colorimetric, or							317B[25]
	Ion chromatography					D6919–03		
54. Residue, total, mg/L:	Gravimetric, 103–105°C			2540 B [18th, 19th, 20th, 21st]	2540 B–97		I–3750–85[2]	
55. Residue, filterable, mg/L:	Gravimetric, 180°C			2540 C [18th, 19th, 20th, 21st]	2540 C–97		I–1750–85[2]	
56. Residue, non–filterable, mg/L:	Gravimetric, 103–105°C post–washing of residue			2540 D [18th, 19th, 20th, 21st]	2540 D–97		I–3765–85[2]	
57. Residue, settleable, mg/L:	Volumetric (Imhoff cone), or gravimetric			2540 F [18th, 19th, 20th, 21st]	2540 F–97			
58. Residue, volatile mg/L:	Gravimetric, 550° C	160.4[1]					I–3753–85[2]	

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
59. Rhodium–total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	265.2[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
60. Ruthenium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	267.2[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
61. Selenium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA gaseous hydride		7741A	3114 B[10] [18th, 19th, 21st]	3114 B–97[10]	D3859–98, 03 (A)	I–3667–85[2]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]		3113 B [18th, 19th, 21st]	3113 B–99	D3859–98, 03 (B)	I–4668–98[59]	
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
62. Silica–Dissolved[46], mg/L:	0.45 micron filtration followed by:							
	Colorimetric, Manual			4500–Si D [18th, 19th], 4500–SiO_2 C [20th, 21st]	4500–SiO_2 C–97	D859–94, 00, 05	I–1700–85[2]	
	Automated (Molybdosilicate)						I–2700–85[2]	
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
63. Silver–Total[9,38,40], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99		I–3720–85[2]	974.27[3]
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99		I–4724–89[61]	
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[2]	
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)							Note 43
64. Sodium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99		I–3735–85[2]	973.54[3]
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [18th, 19th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
	Flame photometric, or			3500–Na D [18th, 19th], 3500–Na B [20th, 21st]	3500–Na B–97			
	Ion chromatography					D6919–03		

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA [44,62]	SW–846 [4,5]	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
65. Specific conductance, micromohs/cm at 25° C:	Wheatstone bridge	120.1[1]	9050A	2510 B [18th, 19th, 20th, 21st]	2510 B–97	D1125–95 (99), (05) (A)	I–2781–85[2]	973.40[3]
66. Sulfate (as SO_4^{2-}), mg/L:	Automated colorimetric	375.2, Rev. 2.0 (1993)	9035					
	Gravimetric			4500–SO_4^{2-} C or D [18th, 19th, 20th, 21st]				925.54[3]
	Turbidimetric					D516–90, 02		426C[39]
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20th, 21st]	4110 B–00	D4327–97, 03		993.30[3]
	CIE/UV					D6508–00 (05)		D6508, Rev 2[64]
67. Sulfide (as S), mg/L:	Titrimetric (iodine)			4500–S^{2-} E [18th], 4500–S^{2-} F [19th, 20th, 21st]	4500–S^{2-} F–00		I–3840–85[2]	
	Colorimetric (methylene blue), or			4500–S^{2-} D [18th, 19th, 20th, 21st]	4500–S^{2-} D–00			
	Ion selective electrode			4500–S^{2-} G [18th, 19th, 20th, 21st]	4500–S^{2-} G–00	D4658–03		
68. Sulfite (as SO_3), mg/L:	Titrimetric (iodine–iodate)			4500–SO_3^- B [18th, 19th, 20th, 21st]	4500–SO_3^- B–00			
69. Surfactants, mg/L:	Colorimetric (methylene blue)			5540 C [18th, 19th, 20th, 21st]	5540 C–00	D2330–88, 02		
70. Temperature, °C:	Thermometric			2550 B [18th, 19th, 20th, 21st]	2550 B–00			Note 41
71. Thallium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99			
	AA graphite furnace (GFAA)	279.2 (Issued 1978)[1], 200.9, Rev. 2.2 (1994)[13]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
72. Tin– Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18th, 19th, 21st]	3111 B–99		I–3850–78[16]	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994)[13]	7010	3113 B [18th, 19th, 21st]	3113 B–99			
	Inductively coupled plasma–atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C					
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A					
73. Titanium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)			3111 D [18th, 19th, 21st]	3111 D–99			
	AA graphite furnace (GFAA)	283.2	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99			
	Inductively coupled plasma–mass spectrometry (ICP–MS), or	200.8, Rev. 5.4 (1994)[13]	6020A					
	Direct current plasma (DCP)							Note 43
74. Turbidity[63], NTU:	Nephelometric	180.1		2130 B [18th, 19th, 20th, 21st]	2130 B–01	D1889–94, 00	I–3860–85[2]	
75. Vanadium–Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18th, 19th, 21st]	3111 D–99			
	AA graphite furnace (GFAA)		7010			D3373–93, 03		
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP), or					D4190–94, 99, 03		Note 43
	Colorimetric (Gallic acid)			3500–V D [18th, 19th], 3500–V B [20th, 21st]	3500–V B–97			

Table B (Continued)
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units	Analytical Technology[68]	EPA 44,62	SW–846 4,5	Standard Methods [Editions][6]	Standard Methods–Online[7]	ASTM[8]	USGS	Other
76. Zinc– Total[9], mg/L:	Digestion[9,11,45] followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18th, 19th, 21st]	3111 B or C–99	D1691–95, 02 (A or B)	I–3900–85[2]	974.27[3], p 37[17]
	AA graphite furnace (GFAA)	289.2 (Issued 1978)[1]	7010					
	Inductively coupled plasma–atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994)[13]	6010B, 6010C	3120 B [20th, 21st]	3120 B–99		I–4471–97[60]	
	Inductively coupled plasma–mass spectrometry (ICP–MS)	200.8, Rev. 5.4 (1994)[13]	6020A			D5673–03, 05		993.14[3]
	Direct current plasma (DCP)					D4190–94, 99, 03		Note 43
	Colorimetric (Dithizone), or			3500–Zn E [18th, 19th]				
	Colorimetric (Zincon)			3500–Zn F [18th, 19th], 3500–Zn B [20th, 21st]	3500–Zn B–97			Note 42

[1] "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI), EPA–600/4–79–020 (NTIS PB 84–128677), Revised March 1983 and 1979 where applicable. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

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

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

[4] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW–846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512–1800 (Publication Number: 955–001–00000–1). Also, available on–line at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

[5] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW–846, Update IV includes methods 7000B, Flame Atomic Absorption Spectrophotometry and 7010, Graphite Furnace Atomic Absorption Spectrophotometry, general method descriptions.

[6] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[7] "Standard Methods for the Examination of Water and Wastewater On–Line", Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006. Subscription service available at: http:/www.standardmethods.org.

[8] "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1994, 1996, 1999, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959. Also available online at www.astm.org.

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

[10] Use the digestion given in the method.
[11] "Test Methods for Evaluating Solid Waste", SW−846 method 3015A. United States EPA SW−846, 3rd Edition and updates. Footnote 4 lists the complete reference.
[12] Copper sulfate may be used in place of mercuric sulfate.
[13] "Methods for the Determination of Metals in Environmental Samples", EPA−600/4−91−010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.
[14] 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.
[15] Ammonia, Automated Electrode Method, Industrial Method Number 379–75 WE, February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
[16] 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).
[17] American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd St., New York, NY 10036.
[18] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).
[19] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
[20] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data using a nitrification inhibitor.
[21] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.
[22] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.
[23] The back titration method will be used to resolve controversy.
[24] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977, Thermo Scientific, 81 Wyman Street, Waltham, MA 02454. 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.
[25] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976. Available on interlibrary loan.
[26] "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color", NCASI Technical Bulletin No. 253. December, 1971. Available from: National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016.
[27] Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.
[28] When using a method with block digestion, this treatment is not required.
[29] Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
[30] Iron, 1,10−Phenanthroline Method, Method 8008, 1980, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.
[31] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, Hach Chemical Company, Loveland, CO 80537. Available on−line at http:/www.hach.com.
[32] Wershaw, R.L., et al, "Methods for Analysis of Organic Substances in Water," Techniques of Water− Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
[33] Nitrogen, Nitrite, Method 8507, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.
[34] Nitrate + nitrite determinations by ion chromatography must be analyzed within 48 hours.
[35] Just prior to distillation, adjust the sulfuric−acid−preserved sample to pH 4 with 1 + 9 NaOH.
[36] The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ±0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrometric procedure. Available on interlibrary loan.
[37] R.F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas–Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421–426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from: Elsevier/North−Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.
[38] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
[39] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition. Available on interlibrary loan.
[40] 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.

[41] Stevens, H.H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water−Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[42] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, Hach Company, Loveland, CO 80537. Available on−line at http:/www.hach.com.

[43] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991. Available from: Thermo Scientific, 81 Wyman Street, Waltham, MA 02454.

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

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

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

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

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

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

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

[51] The full text of Method 1650 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[52] US EPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–024). The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA–821–R–96–011) are recommended to preclude contamination at low−level, trace metal determinations. Available at NTIS, PB−121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.

[53] Quality control requirements for low level mercury are found in s. NR 106.145 (9) and (10), Wis. Adm. Code. Low−level mercury methods are performance based so some method modifications are allowable, provided quality control requirements are met. If an atomic absorption detector is substituted for atomic fluorescence detector, the appropriate method citation is 245.1 (manual) or 245.2 (automated). If method 1631E is modified to eliminate the purge and trap step, the appropriate method citation is 245.7.

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

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

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

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

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

[59] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace−Atomic Absorption Spectrometry," Open File Report (OFR) 98–639.

[60] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole−water Digests Using Inductively Coupled Plasma−Optical Emission Spectrometry and Inductively Coupled Plasma−Mass Spectrometry," Open File Report (OFR) 98−165.

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

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

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

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

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

[66] QuikChem Method 10–204–00–1–X, "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis" is available from Hach Company, P.O. Box 389, Loveland, CO 80537.

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

[68] 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.

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

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

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

[72] Oxygen, Dissolved, Luminescence, Hach Method 10360− Luminescence Measurement of Dissolved Oxygen (LDO®) in Water and Wastewater, Revision 1.1, January 2006, Hach Chemical Company, Loveland, CO 80537. Available from: Hach Company, P.O. Box 389, Loveland, CO 80537. Available on−line at http:/www.hach.com.

Table BM
Metals Digestion Procedures

Analysis	SW−846[1]	EPA[2]	EPA[3]
Dissolved Metals[4]	3005A,3040A[10]		4.1.1
Suspended Metals[5]	3005A		4.1.2
Total Metals[6]	3010A, 3020A[11], 3050B[10], 3051A[10]		4.1.3
Total Recoverable Metals[7]	3005A	200.2	4.1.4
Acid Soluble Metals[8]		200.1[12]	
Available Metals[9]	3015A[13]		

[1] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW–846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986 (third edition), including July 1992 (update I), September 1994 (update II), January 1995 (update IIB), December 1996 (update III), January 1998 (update IVA), November 2000 (update IVB), August 2002 (update IIIB) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512−1800 (Publication Number: 955−001−00000−1). Also, available on−line at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

[2] "Methods for the Determination of Metals in Environmental Samples", EPA−600/4−91−010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91−231498, 5258 Port Royal Road, Springfield, Virginia 22161, (703) 487−4650.

[3] "Methods for Chemical Analysis of water and Wastes", EPA−600/4−79−020, United States Environmental Protection Agency, Revised March 1983 and 1979 where applicable. Available from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487−4650.

[4] "Dissolved metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter prior to sample acidification.

[5] "Suspended metals" means the concentration of metals determined in the portion of a sample retained by a 0.45 micron membrane filter prior to acidification.

[6] "Total metals" means the concentration of metals determined on a solid sample or unfiltered aqueous sample following a vigorous digestion, or alternatively the sum of the metals determined in both the dissolved and suspended fractions.

[7] "Total recoverable metals" means the concentration of metals determined on an unfiltered sample following treatment with hot dilute mineral acid.

[8] "Acid soluble metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter after the sample has been adjusted to pH 1.75 and held for 16 hours. This method is applicable to arsenic, cadmium, chromium, copper, and lead.

[9] "Available metals" are equivalent to "total metals". SW−846 lists method 3015 as a preparation for available metals.

[10] "These methods are for total metals analysis of sediment, sludge, and soil samples and do not apply to wastewater. The required analytical methodology for metals in wastewater sludge is given in Table EM.

[11] Method 3020 is applicable for analysis by GFAA. Method 3010 requires sample acidification with HCl.

[12] Method 200.1 is only applicable for As, Cd, Cr, Cu and Pb.

[13] This method is a microwave−assisted acid leachate digestion.

Table C
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
1. Acenaphthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th, 21st]		
2. Acenaphthylene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
3. Acrolein	GC	603				
	GC/MS	624[7], 1624B				
	HPLC		8316			
4. Acrylonitrile	GC	603	8031			
	GC/MS	624[7], 1624B				
	HPLC		8315A, 8316			
5. Anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
6. Benzene	GC	602	8021B	6220 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
7. Benzidine	GC		8121			Note 6, p.1
	GC/MS	625[8], 1625B	8270C, 8270D			
	HPLC	605				
8. Benzo(a)anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
9. Benzo(a)pyrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
10. Benzo(b)fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
11. Benzo(g,h,i) perylene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
12. Benzo(k) fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
13. Benzyl chloride	GC		8021B,* 8121			Note 6, p.130, Note 10, p.S102
	GC/MS		8260B, 8270C,* 8270D*			
14. Benzyl butyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
15. Bis(2–chloroethoxy) methane	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
16. Bis(2–chloroethyl) ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
17. Bis(2–ethylhexyl) phthalate	GC	606				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
18. Bromodichloromethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
19. Bromoform	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
20. Bromomethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
21. 4–Bromophenyl phenyl ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
22. Carbon tetrachloride	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
23. 4–Chloro–3–methylphenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
24. Chlorobenzene	GC	601, 602	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
25. Chloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
26. 2–Chloroethylvinyl ether	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
27. Chloroform	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
28. Chloromethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
29. 2–Chloronaphthalene	GC	612	8021B,* 8121			Note 13, p.27
	GC/MS	625, 1625B	8260B,* 8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
30. 2–Chlorophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
31. 4–Chlorophenyl phenyl ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
32. Chrysene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
33. Dibenzo(a,h)anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
34. Dibromochloromethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
35. 1,2–Dichlorobenzene	GC	601, 602	8021B,* 8121*	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 13, p.27
	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
36. 1,3–Dichlorobenzene	GC	601, 602	8021B,* 8121*	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 13, p.27
	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
37. 1,4–Dichlorobenzene	GC	601, 602	8021B,* 8121*	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 13, p.27
	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
38. 3,3–Dichlorobenzidine	GC		8121			
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	605				
39. Dichlorodifluoromethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
40. 1,1–Dichloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
41. 1,2–Dichloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
42. 1,1–Dichloroethene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
43. *trans*–1,2–Dichloroethene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
44. 2,4–Dichlorophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
45. 1,2–Dichloropropane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
46. *cis*–1,3–Dichloropropene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
47. *trans*–1,3–Dichloropropene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
48. Diethyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
49. 2,4–Dimethylphenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
50. Dimethyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
51. Di–n–butyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
52. Di–n–octyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
53. 2,3–Dinitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
54. 2,4–Dinitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
55. 2,6–Dinitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
56. 2,3–Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
57. 2,4–Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
58. 2,6–Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
59. Epichlorohydrin	GC		8121			Note 6, p.130, Note 10, p.S102
	GC/MS		8260B			
60. Ethylbenzene	GC	602	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
61. Fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
62. Fluorene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
63. 1,2,3,4,6,7,8–Heptachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
64. 1,2,3,4,7,8,9–Heptachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
65. 1,2,3,4,6,7,8–Heptachlorodibenzo–p–dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
66. Hexachlorobenzene	GC	612	8081A, 8081B, 8121			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
67. Hexachlorobutadiene	GC	612	8021B,* 8121			Note 13, p.27
	GC/MS	625, 1625B	8260B, 8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
68. Hexachlorocyclopenta-diene	GC	612	8081A, 8081B, 8121			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
69. 1,2,3,4,7,8–Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
70. 1,2,3,6,7,8–Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
71. 1,2,3,7,8,9–Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
72. 2,3,4,6,7,8–Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
73. 1,2,3,4,7,8–Hexachlorodibenzo–p–dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
74. 1,2,3,6,7,8–Hexachlorodibenzo–p–dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
75. 1,2,3,7,8,9–Hexachlorodibenzo–p–dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
76. Hexachloroethane	GC	612	8021B,* 8121			Note 13, p.27
	GC/MS	625, 1625B	8260B, 8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
77. Indeno(1,2,3–cd) pyrene	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	Note 13, p.27
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
78. Isophorone	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
79. Methylene chloride	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
80. 2–Methyl–4,6–dinitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
81. Naphthalene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
82. Nitrobenzene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
83. 2–Nitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
84. 4–Nitrophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
85. N–Nitrosodimethylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
86. N–Nitrosodi–n–propylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
87. N–Nitrosodiphenylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
88. Octachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
89. Octachlorodibenzo–p–dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
90. 2,2'–Oxybis (2–chloropropane)	GC	611	8111			
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
91. PCB–1016 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/HRMS	1668A[18]				
92. PCB–1221 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/HRMS	1668A[18]				

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
93. PCB–1232 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/ HRMS	1668A[18]				
94. PCB–1242 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/ HRMS	1668A[18]				
95. PCB–1248 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/ HRMS	1668A[18]				
96. PCB–1254 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/ HRMS	1668A[18]				
97. PCB–1260 (Aroclor or congeners)[16,17]	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HRGC/ HRMS	1668A[18]				
98. 1,2,3,7,8–Pentachlorodibenzofuran	HRGC/ LRMS		8280B			
	HRGC/ HRMS	1613B[14]	8290A[14]			
99. 2,3,4,7,8–Pentachlorodibenzofuran	HRGC/ LRMS		8280B			
	HRGC/ HRMS	1613B[14]	8290A[14]			
100. 1,2,3,7,8–Pentachlorodibenzo–p–dioxin	HRGC/ LRMS		8280B			
	HRGC/ HRMS	1613B[14]	8290A[14]			
101. Pentachlorophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
102. Phenanthrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th, 21st]		

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
103. Phenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
104. Pyrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	
	HPLC	610	8310	6440 B [18th, 19th, 20th]		
105. 2,3,7,8–Tetrachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
106. 2,3,7,8–Tetrachlorodibenzo–p–dioxin	GC/MS	625[9]				
	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B[14]	8290A[14]			
107. 1,1,2,2–Tetrachloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
108. Tetrachlorocatechol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
109. Tetrachloroethene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
110. Tetrachloroguaicol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
111. 2,3,4,6–Tetrachlorophenol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
112. Toluene	GC	602	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
113. 1,2,4–Trichlorobenzene	GC	612	8021B			Note 6, p.130, Note 13, p.27
	GC/MS	625, 1624B	8260B, 8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	

Table C (Continued)
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
114. 3,4,5–Trichlorocatechol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
115. 3,4,6–Trichlorocatechol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
116. 1,1,1–Trichloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
117. 1,1,2–Trichloroethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
118. Trichloroethene	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
119. Trichlorofluoromethane	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C–97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B–97	
120. 3,4,5–Trichloroguaicol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
121. 3,4,6–Trichloroguaicol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
122. 4,5,6–Trichloroguaicol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
123. 2,4,5–Trichlorophenol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B–00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20th, 21st]	6410 B–00	
124. 2,4,6–Trichlorophenol	GC	604	8041A	6420 B [18th, 19th, 20th, 21st]	6420 B–00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00	

Table C (Continued)
List of Approved Test Procedures for Non−Pesticide Organic Compounds in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW−846[3]	Standard Methods [Edition(s)][4]	Standard Methods Online[5]	Other
125. Trichlorosyringol	GC			6420 B [18th, 19th, 20th, 21st]	6420 B−00	
	GC/MS	1653[15]		6410 B [18th, 19th, 20t, 21st]	6410 B−00	
126. Vinyl chloride	GC	601	8021B	6230 B [18th, 19th], 6200 C [20th, 21st]	6200 C−97	
	GC/MS	624, 1624B	8260B	6210 B [18th, 19th], 6200 B [20th, 21st]	6200 B−97	

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

[2] The full text of Methods 601–613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B is incorporated by reference into this Part 136 and is available from the National Technical Information Services as stock number PB95–104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW–846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512−1800 (Publication Number: 955−001−00000−1). Also, available on−line at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

[4] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[5] "Standard Methods for the Examination of Water and Wastewater Online", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. Subscription service available at http:/www.standardmethods.org.

[6] "Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

[7] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624B.

[8] 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.

[9] 5a 625, screening only.

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

[11] Each analyst must make an initial, one−time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624B, and 1625B (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these methods. Additionally, each laboratory, on an ongoing basis must spike and analyze 10% (5% for methods 624 and 625 and 100% for methods 1624B and 1625B) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

[12] "Organochlorine Pesticides and PCBs in Wastewater Using EmporeTM Disk" 3M Corporation Revised 10/28/94.

[13] USGS Method 0–3116–87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments," U.S. Geological Survey, Open File Report 93–125.

[14] Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

[15] The full text of Method 1653 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Also available on−line at http:/www.gpoaccess.gov/.

[16] EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non−detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration reported. It is recognized a number of congeners will co−elute with others, so there will not be 209 results to sum.

[17] EPA Method 8082A shall be used for PCB−Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:

3620C – Florisil
3640A – Gel Permeation

3630C – Silica Gel
3611B – Alumina
3660B – Sulfur Clean Up
3665A – Sulfuric Acid Clean Up.

[18] "Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS", EPA–821–R–00–002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Table D
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
1. Aldrin	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
2. Ametryn	GC						Note 7, p. 83; Note 10, p. S68
3. Aminocarb	HPLC						Note 7, p. 94; Note 10, p. S16; Note 14
	LC/MS		8321A				
4. Atraton	GC						Note 7, p. 83; Note 10, p. S68
5. Atrazine	GC		8141A,* 8141B*				Note 7, p. 83; Note 10, p. S68; Note 13
6. Azinphos methyl	GC						Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
7. Barban	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
	LC/MS		8321A				
8. á–BHC	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 12
	GC/MS	625[5]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
9. â–BHC	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 12
	GC/MS	625[5]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
10. ä–BHC	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 12
	GC/MS	625[5]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
11. ã–BHC (Lindane)	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625[5]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
12. Captan	GC			6630 B [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table D (Continued)
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
13. Carbaryl	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
	LC/MS		8321A, 8325				
14. Carbophenthion	GC		8141B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
15. Chlordane	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625[5]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
16. Chloropropham	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A				
17. 2,4–D	GC		8151A*	6640 B [18th, 19th, 20th]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
18. 4,4'–DDD	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
19. 4,4'–DDE	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
20. 4,4'–DDT	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
21. Demeton–O	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
22. Demeton–S	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
23. Diazinon	GC		8141A, 8141B				Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
24. Dicamba	GC		8151A*				Note 7, p. 115
	LC/MS		8321A				

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table D (Continued)
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
25. Dichlofenthion	GC		8141A, 8141B				Note 8, p. 27; Note 10, p. S73
26. Dichloran	GC			6630 B and C [18th, 19th, 20th]			Note 7, p. 7
27. Dicofol	GC					D5812– 96 (02)	
28. Dieldrin	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
29. Dioxathion	GC		8141A, 8141B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
30. Disulfoton	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
31. Diuron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
32. Endosulfan I	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625[9]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
33. Endosulfan II	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
34. Endosulfan sulfate	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
35. Endrin	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625[9]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
36. Endrin Aldehyde	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
37. Ethion	GC						Note 8, p. 27; Note 10, p. S73
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
38. Fenuron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A				

Table D (Continued)
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
39. Fenuron–TCA	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
40. Heptachlor	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625[9]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
41. Heptachlor epoxide	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 10, p. S73; Note 12
	GC/MS	625[9]	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
42. Isodrin	GC		8081A, 8081B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
43. Linuron	HPLC						Note 7, p.104; Note 10 p. S64; Note 14
	LC/MS		8321A, 8325				
44. Malathion	GC		8141A,* 8141B*	6630 C [18th, 19th, 20th]			Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
45. Methiocarb	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	LC/MS		8321A, 8325				
46. Methoxychlor			8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
47. Mexacarbate	HPLC						Note 7, p.94; Note 10, p. S60; Note 14
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
48. Mirex	GC		8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 7, p. 7; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
49. Monuron	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
50. Monuron–TCA	HPLC						Note 7, p.104; Note 10, p. S64; Note 14

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table D (Continued)
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
51. Neburon	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A				
52. Parathion methyl	GC		8141A, 8141B	6630 C [18th, 19th, 20th]			Note 7, p. 25; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
	LC/MS		8321A				
53. Parathion ethyl	GC		8141A, 8141B	6630 C [18th, 19th, 20th]			Note 7, p. 25; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
54. PCNB	GC		8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 7, p. 7
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
55. Perthane	GC		8081A, 8081B			D5812– 96 (02)	Note 8, p. 27
56. Prometon	GC						Note 7, p. 83; Note 10, p. S68; Note 13
57. Prometryn	GC						Note 7, p. 83; Note 10, p. S68; Note 13
58. Propazine	GC						Note 7, p. 83; Note 10, p. S68; Note 13
59. Propham	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A				
60. Propoxur	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	LC/MS		8321A				
61. Secbumeton	HPLC						Note 7, p. 83; Note 10, p. S68; Note 14
62. Siduron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
63. Simazine	GC		8141A, 8141B				Note 7, p. 83; Note 10, p. S68; Note 13
64. Strobane	GC		8081A, 8081B	6630 B and C [18th, 19th, 20th]			Note 7, p. 7
65. Swep	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14

Table D (Continued)
List of Approved Test Procedures for Pesticides[1] in Wastewater

Parameter[1]	Analytical Technology	EPA[2,11]	SW–846[3]	Standard Methods[4]	Standard Methods Online[5]	ASTM[6]	Other
66. 2,4,5–T	GC		8151A	6640 B [18th, 19th, 20th]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
67. 2,4,5–TP (Silvex)	GC		8151A	6640 B [18th, 19th, 20th]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
68. Terbuthylazine	GC						Note 7, p. 83; Note 10, p. S68
69. Toxaphene	GC	608	8081A, 8081B	6630 B and C [18th, 19th, 20th]		D5812– 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		
70. Trifluralin	GC		8081A, 8081B	6630 B [18th, 19th, 20th]			Note 7, p. 7; Note 13
	GC/MS		8270C, 8270D	6410 B [18th, 19th, 20th, 21st]	6410 B–00		

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

[2] The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," 40 CFR, Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," 40 CFR, Part 136.

[3] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW–846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512–1800 (Publication Number: 955–001–00000–1). Also, available on–line at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

[4] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2001), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[5] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On–line subscription service available at http:/www.standardmethods.org.

[6] "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959. Also available online at www.astm.org.

[7] "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 (HPLC) methods.

[8] "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).

[9] 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.

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

[11] Each analyst must make an initial, one–time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A, 40 CFR, Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on–going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

[12] "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation, Revised 10/28/94.

[13] USGS Method 0–3106–93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen–containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94–37.

[14] HPLC method 623 from "Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater", EPA 440/1–83/079–C, United States Environmental Protection Agency. Available from National Technical Information Service, 5258 Port Royal Road, Springfield, Virginia, 22161 (703) 487–4650.

Table E
List of Approved Radiological Analytical Methods for Wastewater

Parameter and Units	Analytical Technology	EPA[1]	Standard Methods[2]	Standard Methods Online[3]	ASTM4	USGS5
1. Alpha–Total, pCi per liter	Proportional or scintillation counter	900.0	7110 B	7110 B–00	D1943–05	pp. 75 and 78[6]
2. Alpha–Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	7110 B–00	D1943–05	p. 79
3. Beta–Total, pCi per liter.	Proportional counter	900.0	7110 B	7110 B–00	D1890–05	pp. 75 and 78[6]
4. Beta–Counting error, pCi	Proportional counter	Appendix B	7110 B	7110 B–00	D1890–05	p. 79
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500–Ra B	7500–Ra B–01	D2460–05	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500–Ra C	7500–Ra C–01	D3454–05	p. 81

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", EPA–600/–4–80–032, U.S. Environmental Protection Agency.
[2] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
[3] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On–line subscription service available at http:/www.standardmethods.org.
[4] "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959. Also available online at www.astm.org.
[5] 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).
[6] 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 EM
List of Approved Analytical Methods for Sludge

Parameter	Analytical Technology	Sample Preparation		Method			
		SW–846[1]	EPA[4]	SW–846[1]	EPA[2,3]	Standard Methods[8,9]	Other
Metals							
Arsenic	Gaseous Hydride[5]	7061A		7061A			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Beryllium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 D [18th, 19th, 21st], 3111 D–99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Cadmium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Chromium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B [18th, 19th, 21st], 3111 B–99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Copper	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18th, 19th, 21st], 3111 B–99 or C–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Lead	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace[6]	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Mercury	Cold Vapor Atomic Absorption	7471A, 7471B		7471A, 7471B			
	Cold vapor atomic fluorescence spectrometry	7474					

Table EM (Continued)
List of Approved Analytical Methods for Sludge

Parameter	Analytical Technology	SW–846[1]	EPA[4]	SW–846[1]	EPA[2,3]	Standard Methods[8,9]	Other
Molybdenum	Graphite Furnace[6]	3050B, 3051A	200.2	7010	200.9	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Nickel	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18th, 19th, 21st], 3111 B–99 or C–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Selenium	Gaseous Hydride[5]	7741A		7741A			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9 *	3113 B [18th, 19th, 21st], 3113 B–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Zinc	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18th, 19th, 21st], 3111 B–99 or C–99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20th, 21st], 3120 B–99	
	Inductively Coupled Plasma/ Mass Spectrometry	3050B, 3051A		6020A	200.8		
Organics							
Dioxins and Furans	Gas Chromatography/Mass Spectrometry	8290A[11]	1613B[11]	8290A	1613B		
PCB (Aroclor or Congeners)	Gas Chromatography	3540B, 3540C, 3545A		8082, 8082A[12]			
PCB (Congeners)	Gas Chromatography/Mass Spectrometry	1668A[13, 14,15]			1668-A[13, 14,15]		
Biological							
Enteric Viruses	Centrifuge Concentration						D 4994–89 (02)[7], or Appendix H[10]
Fecal Coliform	Most Probable Number Membrane Filter					9221 E [18th, 19th, 20th, 21st], 9221 E–99, 9222 D, 9222 D–97	Appendix F[10]
Helminth ova	Density Gradient Flotation						Note 9 or Appendix I[9]
Specific Oxygen Uptake Rate	Respirometer					2710 B [18th, 19th, 20th, 21st], 2710 B–04	Appendix D.2.[10]
Salmonella	Most Probable Number Selective Media Culture						9260 D.1[8] or Appendix G[10]

***Note:** The department of natural resources reports that these numbers should have been included in the creation of this table by CR 08–076 and that they will be added to the table in subsequent rulemaking.

Table EM (Continued)
List of Approved Analytical Methods for Sludge

Parameter	Analytical Technology	SW–846[1]	EPA[4]	SW–846[1]	EPA[2,3]	Standard Methods[8,9]	Other
Physical							
Solids	Gravimetric					2540 G [18th, 19th, 20th, 21st], 2540 G–97	
Percent Volatiles Solids Reduction	Calculation						Appendix D.1. and 3[10]

[1] "Test Methods for Evaluating Solid Waste", Physical/Chemical Methods," SW–846, Environmental Protection Agency, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: The Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250–7954. Available online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

[2] If an alternative digestion procedure is specified in the analytical method, the digestion in this table shall be used. In all cases, consult the analytical method for special requirements and cautions. SW–846 method 3051A is an acceptable alternate digestion procedure to SW–846 method 3050B.

[3] "Methods for the Determination of Metals in Environmental Samples", EPA–600/4–91–010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

[4] "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements", Method 200.2, Revision 2.8, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, 1994. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

[5] High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult Method 3114, of "Standard Methods for the Examination of Water and Wastewater", 18th, 19th, 20th, or 21st edition, for more information.

[6] Concentrations of lead in municipal sludge may exceed the working range of graphite furnace.

[7] 1993 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993, 1916 Race Street, Philadelphia, PA 19103. Available from: the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[8] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21st Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[9] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On–line subscription service available at http:/www.standardmethods.org.

[10] "Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1–87–014, Environmental Protection Agency, 1987. Available from: the National Technical Information Service, order # PB 88–154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161.

[11] "Environmental Regulations and Technology – Control of Pathogens and Vectors Attraction in Sewage Sludge", EPA–625/R–92/013, Revised October 1999, Environmental Protection Agency, Cincinnati, OH, 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

[12] Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

[13] EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non–detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration by dry weight reported. It is recognized that a number of the congeners will co–elute with others, so there will not be 209 results to sum.

[14] EPA Method 8082A shall be used for PCB–Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. For either type of analysis, the sample shall be extracted using Soxhlet extraction Method 3540C or Pressurized Fluid Extraction Method 3545A. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and achieve as close to a limit of detection of 0.11 mg/kg as possible. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and to achieve as close to a limit of detection of 0.003 mg/kg as possible for each congener. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:

3620C – Florisil
3640A – Gel Permeation
3630C – Silica Gel
3611B – Alumina
3660B – Sulfur Clean Up
3665A – Sulfuric Acid Clean Up.

[15] "Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS", EPA–821–R–00–002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Table ES
List of Approved Methods for Pharmaceutical Pollutants [1a]

Pharmaceuticals pollutants	CAS registry No.	Analytical method number[1m]
acetonitrile	75–05–8	1666/1671/D3371/D3695.
n–amyl acetate	628–63–7	1666/D3695.
n–amyl alcohol	71–41–0	1666/D3695
benzene	71–43–2	D4763/D3695/502.2/524.2.
n–butyl–acetate	123–86–4	1666/D3695.
tert–butyl alcohol	75–65–0	1666
chlorobenzene	108–90–7	502.2/524.2.
chloroform	67–66–3	502.2/524.2/551.
o–dichlorobenzene	95–50–1	1625C/502.2/524.2.
1,2–dichloroethane	107–06–2	D3695/502.2/524.2.
diethylamine	109–89–7	1666/1671.
dimethyl sulfoxide	67–68–5	1666/1671.
ethanol	64–17–5	1666/1671/D3695.
ethyl acetate	141–78–6	1666/D3695.
n–heptane	142–82–5	1666/D3695.
n–hexane	110–54–3	1666/D3695.
isobutyraldehyde	78–84–2	1666/1667.
isopropanol	67–63–0	1666/D3695.
isopropyl acetate	108–21–4	1666/D3695.
isopropyl ether	108–20–3	1666/D3695.
methanol	67–56–1	1666/1671/D3695.
Methyl Cellosolve [Delta]	109–86–4	1666/1671
methylene chloride	75–09–2	502.2/524.2
methyl formate	107–31–3	1666
4–methyl–2–pentanone (MIBK)	108–10–1	1624C/1666/D3695/D4763/524.2.
phenol	108–95–2	D4763.
n–propanol	71–23–8	1666/1671/D3695.
2–propanone (acetone)	67–64–1	D3695/D4763/524.2.
tetrahydrofuran	109–99–9	1666/524.2.
toluene	108–88–3	D3695/D4763/502.2/524.2.
triethylamine	121–44–8	1666/1671.
xylenes	(Note 1)	1624C/1666.

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

[1a] Test methods listed in Table C may be used for the parameters listed in this table.

[1m] EPA Methods 1666, 1667, and 1671 listed in the table above are published in the compendium titled Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters (EPA 821–B–98–016). EPA Methods 502.2 and 524.2 have been incorporated by reference into 40 CFR 141.24 and are in Methods for the Determination of Organic Compounds in Drinking Water, EPA–600/4–88–039, December 1988, Revised, July 1991, and Methods for the Determination of Organic Compounds in Drinking Water–Supplement II, EPA–600/R–92–129, August 1992, respectively. These EPA test method compendia are available from the National Technical Information Service, NTIS PB91–231480 and PB92–207703, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll–free number is 800–553–6847. ASTM test methods D3371, D3695, and D4763 are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.

Table F
Required Containers, Preservation Techniques, and Holding Time for Wastewater

Parameter Number/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
Table A — Bacterial Tests			
1–5. Coliform, total, fecal and *E. coli*	PA, G	Cool, <10°C, 0.0008% Na_2SO_3[5]	6 hours
6. Fecal streptococci	PA, G	Cool, <10°C, 0.0008% Na_2SO_3[5]	6 hours
7. Enterococci	PA, G	Cool, <10°C, 0.0008% Na_2SO_3[5]	6 hours
Table A — Protozoan Tests			
8. Cryptosporidium	LDPE, field filtration	0–8°C	96 hours[21]
9. Giardia	LDPE, field filtration	0–8°C	96 hours[21]
Table A — Aquatic Toxicity Tests			
10–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6°C[16]	36 hours
Table B — Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6°C[18]	14 days
2. Alkalinity	P, FP, G	Cool, ≤6°C[18]	14 days
4. Ammonia	P, FP, G	Cool, ≤6°C[18], H_2SO_4 to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6°C[18]	48 hours
11. Bromide	P, FP, G	None required	28 days
14. Carbonaceous biochemical oxygen demand	P, FP, G	Cool, ≤6°C[18]	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6°C[18], H_2SO_4 to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6°C[18]	48 hours
23–24. Cyanide, total or available (CATC)	P, FP, G	Cool, ≤6°C [18], NaOH to pH>12[6], reducing agent[5]	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 44. Kjeldahl and organic N	P, FP, G	Cool, ≤6°C [18], H_2SO_4 to pH<2	28 days
38. Nitrate	P, FP, G	Cool, ≤6°C[18]	48 hours
39. Nitrate + nitrite	P, FP, G	Cool, ≤6°C [18], H_2SO_4 to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6°C[18]	48 hours
41. Oil and grease	G	Cool, ≤6°C [18], HCl or H_2SO_4 to pH<2	28 days
42. Organic carbon	P, FP, G	Cool, ≤6°C [18], HCl, H_2SO_4 or H_3PO_4 to pH<2	28 days
45. Orthophosphate	P, FP, G	Cool, ≤6°C[18]	Analyze within 48 hours; filter, if needed, within 15 minutes[22]
47. Oxygen, dissolved (Probe or Luminescence)	G, Bottle and top	None required	Analyze within 15 minutes
47. Oxygen, dissolved (Winkler)	G, Bottle and top	Fix on site and store in dark	8 hours
49. Phenols	G	Cool, ≤6°C [18], H_2SO_4 to pH<2	28 days
50. Phosphorus (elemental)	G	Cool, ≤6°C[18]	48 hours
51. Phosphorus, total	P, FP, G	Cool, ≤6°C [18], H_2SO_4 to pH<2	28 days
54. Residue, total	P, FP, G	Cool, ≤6°C[18]	7 days
55. Residue, filterable	P, FP, G	Cool, ≤6°C[18]	7 days
56. Residue, nonfilterable (TSS)	P, FP, G	Cool, ≤6°C[18]	7 days
57. Residue, settleable	P, FP, G	Cool, ≤6°C[18]	48 hours

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Time for Wastewater

Parameter Number/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
58. Residue, volatile	P, FP, G	Cool, ≤6°C[18]	7 days
62. Silica	P or Quartz	Cool, ≤6°C[18]	28 days
65. Specific conductance	P, FP, G	Cool, ≤6°C[18]	28 days
66. Sulfate	P, FP, G	Cool, ≤6°C[18]	28 days
67. Sulfide	P, FP, G	Cool, ≤6°C[18], add zinc acetate plus sodium hydroxide to pH>9	7 days
68. Sulfite	P, FP, G	None required	Analyze within 15 minutes
69. Surfactants	P, FP, G	Cool, ≤6°C[18]	48 hours
70. Temperature	P, FP, G	None required	Analyze within 15 minutes
74. Turbidity	P, FP, G	Cool, ≤6°C[18]	48 hours
Table B — Metals[7]			
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 46, 48, 52, 53, 59–61, 63, 64, 71–73, 75, 76. 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
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months
18. Chromium IV	P, FP, G	Cool, ≤6°C [18], pH = 9.3 – 9.7[20]	28 days
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days
35. Mercury (CVAFS)	FP, G; and FP–lined cap[17]	5 mL/L 12N HCl or 5 mL/L BrCl[17]	90 days[17]
Table C — Organic Tests[8]			
3, 4. Acrolein and acrylonitrile	G, FP–lined septum	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5]	14 days[10]
7, 38. Benzidines[11, 12]	G, FP–lined cap	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5]	7 days until extraction[13]
29, 35–37, 66–68, 76, 113. Chlorinated hydrocarbons[11]	G, FP–lined cap	Cool, ≤6°C[18]	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 90. Haloethers	G, FP–lined cap	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5]	7 days until extraction, 40 days after extraction
56–58, 78, 82. Nitroaromatics and Isophorone[11]	G, FP–lined cap	Cool, ≤6°C[18], store in dark, 0.0008% Na_2SO_3[5]	7 days until extraction, 40 days after extraction
85–87. Nitrosamines[11, 14]	G, FP–lined cap	Cool, ≤6°C[18], store in dark, 0.0008% Na_2SO_3[5]	7 days until extraction, 40 days after extraction
91–97. PCBs[11]	G, FP–lined cap	Cool, ≤6°C[18]	1 year until extraction, 1 year after extraction
63–65, 69–75, 88, 89, 98–100, 105, 106. PCDDs/PCDFs[11]			
Aqueous Samples: Field and Laboratory Preservation	G	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5], pH<9	1 year
Solids and Mixed Phase Samples: Field Preservation	G	Cool, ≤6°C[18]	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6°C[18]	24 hours
Solids, Mixed–Phase, and Tissue Samples: Laboratory Preservation	G	Freeze, ≤–10°C	1 year
23, 30, 44, 49, 53–55, 80, 83, 84, 101, 103, 108, 110, 111, 114, 115, 120–125. Phenols[11]	G, FP–lined cap	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5]	7 days until extraction, 40 days after extraction
14, 17, 48, 50–52. Phthalate esters[11]	G, FP–lined cap	Cool, ≤6°C[18], 0.0008% Na_2SO_3[18]	7 days until extraction, 40 days after extraction[13]
1, 2, 5, 8–12, 32, 33, 61, 62, 77, 81, 102, 104. Polynuclear aromatic hydrocarbons[11]	G, FP–lined cap	Cool, ≤6°C[18], store in dark, 0.0008% Na_2SO_3[5]	7 days until extraction, 40 days after extraction

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Time for Wastewater

Parameter Number/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 59, 79, 107, 109, 113, 116–119, 126. Purgeable halocarbons.	G, FP–lined septum	Cool, ≤6°C[18], 0.0008% Na_2SO_3[5]	14 days
6, 60, 112. Purgeable aromatic hydrocarbons	G, FP–lined septum	Cool, ≤6°C[18], HCl to pH<2, 0.0008% Na_2SO_3[5]	14 days[9]
Table D — 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 E — Radiological Tests			
1–5. Alpha, beta and radium	P, FP, G	HNO_3 to pH<2	6 months

[1] "P" is polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table F; "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 F each grab sample shall be preserved within 15 minutes of collection. For a composite sample collected with an automated sampler, refrigerate the sample at ≤6°C during collection unless specified elsewhere in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and analysis, maintain the sample at ≤6°C, unless specified elsewhere in this table 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 the sample. Preserve (e.g., addition of acid, base or other chemical) the grab sample, composite sample, or aliquot split from the composite sample within 15 minutes of collection. The temperature of the samples shall be documented upon receipt at the laboratory. If the samples are shipped in crushed or cube ice (not "blue ice" packs) and solid ice is still present in the cooler, the lab may simply report the samples as "received on ice". If the ice has melted, the lab must report the either the temperature of the meltwater or of a temperature blank. A temperature blank is defined as an aliquot of deionized water, in an appropriate sample container, which is transported along with the samples. Since shipping simply with "blue ice" packs does not insure that samples are maintained at the appropriate temperatures, the sample collector must submit a temperature blank when using these ice packs for shipping.

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator (s. NR 219.05). 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.

[5] Add a reducing agent only in the presence of residual chlorine.

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

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

(1) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well–ventilated area, transfer 0.75 liter of sample to a 4.4–L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH <2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to >12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume

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

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

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

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

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

[7] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives.

[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 samples 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 three days of sampling.

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

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

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

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

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

[16] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that ≤6°C has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on−site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

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

[18] Aqueous samples must be preserved at ≤6°C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. The specification of "≤6°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 must be analyzed within 15 minutes.

[19] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately

(see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

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

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

[22] Filtration is only required when reporting dissolved orthophosphate, dissolved hydrolyzable phosphorus or dissolved organic phosphorus as described in EPA Method 365.1 (1993). Filtration must be completed within 15 minutes of collection using a 0.45µm filter, sample shall be maintained at 6°C and analyzed within 48 hours.

NR 219.05 Alternate test procedures. Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. The department may approve the use of an alternate test procedure on a case−by−case basis if the criteria for approval of the alternate procedure established in s. NR 149.42 are met. If the department or the EPA approves an alternate test procedure, it shall be considered equivalent to the approved method.

Note: The federal requirements for alternate test procedure approval are given in 40 CFR 136.5.

History: Cr. Register, August, 1976, No. 248, eff. 9−1−76; r. and recr. January, 1978, No. 265, eff. 2−1−78; renum. from NR 219.04 and am. Register, June, 1986, No. 366, eff. 7−1−86; r. and recr. Register, November, 1992, No. 443, eff. 12−1−92; am. Register, February, 1996, No. 482, eff. 3−1−96; correction made under s. 13.92 (4) (b) 7., Stats., Register May 2009 No. 641, eff. 6−1−09.

NR 219.06 Laboratory certification or registration. Bacteriological analyses of groundwater samples, and all radiological analyses shall be performed by the state laboratory of hygiene or at a laboratory certified or approved by the department of agriculture, trade and consumer protection. Other laboratory test results, including effluent toxicity, submitted to the department under a WPDES permit shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

(1) Temperature,

(2) Turbidity,

(3) Bacteria tests in wastewater effluent and sludges,

(4) pH,

(5) Chlorine residual,

(6) Specific conductance,

(7) Physical properties of soils and sludges,

(8) Nutrient tests of soils and sludges,

(9) Flow measurements.

History: Cr. Register, April, 1986, No. 364, eff. 8−28−86; renum. from NR 219.07 and am. (intro.) Register, November, 1992, No. 443, eff. 7−1−93; am. Register February, 1996, No. 482, eff. 3−1−96; correction in (intro.) made under s. 13.93 (2m) (b) 6., Stats., Register November 2004 No. 587.