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Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

NR 219.01 Purpose.
NR 219.02 Applicability.
NR 219.03 Definitions.
NR 219.04 Identification of test procedures.
NR 219.05 Alternate test procedures.
NR 219.06 Laboratory certification or registration.

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 4° C where required. All samples requiring preservation at 4° 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 4° 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.

Note: Copies of the publications referenced in Tables A − F are available for inspection at the offices of the department of natural resources, the secretary of state 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.**

Table A
List of Approved Biological Test Procedures

Parameter and Units	Method[1]	EPA	Standard Methods 18th, 19th, 20th Ed.	USGS	Other
Bacteria:					
1. Coliform (fecal) number per 100 ml	Most Probable Number (MPN), 5 tube, 3 dilution; or,	p132[3]	9221C E[4]		
	membrane filter (MF)[2], single step	p124[3]	9222D[4]	B−0050−85[5]	
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or	p132[3]	9221C E[4]		
	MF, single step[6]	p124[3]	9222D[4]		
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or,	p114[3]	9221B[4]		
	MF[2] single step or two step	p108[3]	9222B[4]	B−0025−85[5]	
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, 3 dilution; or,	p114[3]	9221B[4]		
	MF[2] with enrichment.	p111[3]	9222(B+B.5c)[4]		
5. *E. coli*, number per 100 mL[28]	MPN[7,9,15], multiple tube;		9221B.1/9221F[4,12,14]		991.15[11]
	Multiple tube/multiple well;		9223B[4,13]		Colilert®[13,17]
					Colilert−18®[13,16,17]
					D5392−93[10]
	MF[2,6,7,8,9] two step, or	1103.1[20]	9222B/9222G[4,19]		
			9213D		MColiBlue24[18]
	Single step.	1603[21]			
		1604[22]			
6. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution;	p.139[3]	9230B[4]	B−0055−85[5]	
	MF[2], or	p.136[3]			
	Plate count	p.143[4]			
7. Enterococci, number per 100 mL.	MPN[7,9] multiple tube		9230B[4]		
	Multiple tube/multiple well				D6503−99[10]
					Enterolert®[13,23]
	MF[2,6,7,8,9] two step	1106.1[24]			D5259−92[10]
	Single step, or	1600[25]	9230C[4]		
	Plate count	p. 143			
Protozoa:					
8. Cryptosporidium[28]	Filtration/IMS/FA	1622[26]			
		1623[27]			
9. Giardia[28]	Filtration/IMS/FA	1623[27]			
Aquatic Toxicity:					
10. Toxicity, acute, fresh water organisms, percent effluent	Ceriodaphnia, 48−h static−renewal mortality.				Note 29
	Fathead minnow, 96−h static−renewal mortality, or 96−h flow−through mortality.				Note 29
10. Toxicity, chronic, fresh water organisms, percent effluent.	Fathead minnow larval survival and growth.				Note 29
	Ceriodaphnia survival and reproduction.				Note 29

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

[2] A 0.45 µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

[3] USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8–78/017.

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

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

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

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

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

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

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

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

[12] The multiple−tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false−positive rate and false−negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform−positive tubes on a seasonal basis.

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

[14] After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC–MUG media or EC media supplemented in the laboratory with 50 g/mL of MUG may be used.

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

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

[17] Descriptions of the Colilert, Colilert−18, Quanti−Tray, and Quanti−Tray/2000 may be obtained from IDEXX Laboratories, Inc., One IDEXX Drive, Westbrook, Maine 04092.

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

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

[20] USEPA. 2002. Method 1103.1: Escherichia coli (E. coli) In Water By Membrane Filtration Using membrane−Thermotolerant Escherichia coli Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington D.C. EPA−821−R−02−020.

[21] USEPA. 2002. Method 1603: Escherichia coli (E. coli) In Water By Membrane Filtration Using Modified membrane−Thermotolerant Escherichia coli Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington D.C. EPA−821−R−02−023.

[22] Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner et al. 1993. ''New Medium for the Simultaneous Detection of Total Coliform and Escherichia coli in Water.'' Appl. Environ. Microbiol. 59:3534−3544 and in USEPA. 2002. Method 1604: Total Coliforms and Escherichia coli (E. coli) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA 821−R−02−024.

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

[24] USEPA. 2002. Method 1106.1: Enterococci In Water By Membrane Filtration Using membrane−Enterococcus−Esculin Iron Agar (mE−EIA). U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA−821−R−02−021.

[25] USEPA. 2002. Method 1600: Enterococci in Water by Membrane Filtration Using membrane−Enterococcus Indoxyl−[beta]−D−Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC. EPA−821−R−02−022.

[26] Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of Cryptosporidium. SEPA. 2001. Method 1622: Cryptosporidium in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA−821−R−01−026.

[27] Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of Cryptosporidium and Giardia oocysts and cysts. USEPA. 2001. Method 1623. Cryptosporidium and Giardia in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA−821−R−01−025.

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

[29] 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, the Secretary of State and the Legislative Reference Bureau. Copies are available from: the Department of Natural Resources, Bureau of Integrated Science Services, P.O. Box 7921, Madison, WI 53707.

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
1. Acidity, as $CaCO_3$, mg/L, Electrometric end point or phenolphthalein end point	305.1		2310 B(4a) [18th, 19th, 20th]	D1067−92	I−1020−85	
2. Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric: Titration to pH 4.5, manual or automated	310.1 310.2		2320 B [18th, 19th, 20th]	D1067−92	I−1030−85 I−2030−85	973.43[3]
3. Aluminum, mg/L: Digestion[4] followed by:						
AA direct aspiration[4m,36],	202.1	7020	3111 D [18th, 19th]		I−305I−85	
AA furnace,	202.2 or 200.9[5]		3113 B [18th, 19th]			
Inductively coupled plasma (ICP)[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry (ICP−MS),	200.8[5]	6020A				
Direct current plasma (DCP)[4m,36], or				D4190−94		Note 34
Colorimetric (Eriochrome cyanine R)			3500−Al B [20th] or 3500−Al D [18th, 19th]			
4. Ammonia (as N), mg/L: Manual distillation[6] (at pH 9.5):	350.2		4500−NH_3 B [18th, 19th, 20th]			
Followed by Nesslerization,	350.2		4500−NH_3 C [18th]	D1426−89(A)	I−3520−85	973.49[3]
Titration,	350.2		4500−NH_3 C [19th, 20th] and 4500−NH_3 E [18th]			973.49[3]
Electrode,	350.3		4500−NH_3 D or E [19th, 20th] and 4500−NH_3 F or G [18th]	D1426−89(B)		
Automated phenate, or	350.1[1m]		4500−NH_3 G [19th, 20th] and 4500−NH_3 H [18th]		I−4523.85	
Automated electrode						Note 7
5. Antimony, ug/L: Digestion[4] followed by:						
AA direct aspiration[4m, 36],	204.1	7040	3111 B [18th, 19th]			
AA furnace,	200.9[5]	7041	3113 B [18th, 19th]			
AA (gaseous borohydride),		7062				
Inductively coupled plasma[4m, 36], or	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				

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Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
6. Arsenic, ug/L:						
Digestion[4] followed by	206.5					
AA (gaseous hydride),		7061A	3114 B[4g] [18th, 19th, 20th]	D2972−97(B)	I−3062.85	
AA (gaseous borohydride),		7062				
AA furnace,	206.2 or 200.9[5]	7060A	3113 B [18th, 19th]	D2972−97(C)	I−4063−98[49]	
Inductively coupled plasma[4m, 36,]	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry, or,	200.8[5]	6020A				
Colorimetric (SDDC)			3500−As B [20th] and 3500−As C [18th, 19th]	D2972−97(A)	I−3060−85	
7. Barium, mg/L:						
Digestion4 followed by:						
AA direct aspiration[4m,36],	208.1	7080A	3111 D [18th, 19th]		I−3084−85	
AA furnace,	208.2	7081	3113 B [18th, 19th]	D4382−95		
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry, or	200.8[5]	6020A				
Direct current plasma[4m,36]						Note 34
8. Beryllium, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	210.1	7090	3111 D [18th, 19th]	D3654−93(88)(A)	I−3095−85	
AA furnace,	210.2 or 200.9[5]	7091	3113 B [18th, 19th]	D3645−93(88)(B)		
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				Note 34
Direct current plasma, or				D4190−94		
Colorimetric (aluminon)			3500−Be D [18th, 19th, 20th]			
9. Biochemical oxygen demand (BOD_5), mg/L:						
Dissolved Oxygen Depletion			5210 B [18th, 19th, 20th]		I−1578−78[8]	973.443[3]
10. Boron[37], mg/L:						
Colorimetric (curcumin),	212.3		4500−B B [18th, 19th, 20th]		I−3112−85	
Inductively coupled plasma, or	200.7[5]	6010C	3120 B [18th, 19th, 20th]		1−4471−97[50]	
Direct current plasma				D4190−94		Note 34
11. Bromide, mg/L: Titrimetric	320.1			D1246−95(C)	I−1125−85	p.S44[10]
Ion Chromatography	300.0[1m]	9056				

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
12. Cadmium−Total[6], mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	213.1	7130	3111 B or C [18th, 19th]	D3557−95 (A or B)	I−3135−85 or I−3136−85	974.27[3]
AA furnace,	213.2 or 200.9[5]	7131A	3113 B [18th, 19th]	D3557−95(D)	I−1438−89[51]	
Inductively coupled plasma[4m,36]	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−1472−85 or I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				
Direct current plasma[4m,36],				D4190−94		Note 34
Voltametry[11], or				D3557−95(C)		
Colorimetric (Dithizone)			3500−Cd D [18th, 19th]			
13. Calcium, mg/L:						
Digestion[4] followed by:						
Atomic absorption,	215.1	7140	3111 B [18th, 19th]	D511−92(B)	I−3152−85	
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Direct current plasma, or						Note 34
EDTA titration	215.2		3500−Ca D [20th] and 3500−Ca D [18th, 19th]	D511−93(A)		
14. Carbonaceous Biochemical oxygen demand ($CBOD_5$), mg/L:						
with nitrification inhibitor[12]			5210 B [18th, 19th, 20th]			
15. Chemical oxygen demand (COD), mg/L:						
Closed reflux			5220 C [18th, 19th, 20th]			
Titrimetric	410.1 410.2 410.3		5220 B [18th, 19th, 20th]	D1252−95(A)	I−3560 or I−3562−85	973.46[3]
Automated and manual Spectrophotometric	410.4[1m]		5220 D [18th, 19th, 20th]	D1252−95(B)	I−3561−85	Notes 13 & 14
16. Chloride, mg/L:						
Titrimetric (silver nitrate) or		9253	4500−Cl− B [18th, 19th, 20th]	D512−89(B)	I−1183−85	
(Mercuric nitrate),	325.3	9252A	4500−Cl− C [18th, 19th, 20th]	D512−89(A)	I−1184−85	973.51[3]
Colorimetric (ferricyanide), manual					I−1187−85	
or automated, or	325.1 or 325.2	9250	4500−Cl− E [18th, 19th, 20th]		I−2187−85	
Ion chromatography	300.0[1m]	9056				

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Table B
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Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
17. Chlorine − Total residual, mg/L:						
amperometric,	330.1		4500−Cl D [18th, 19th, 20th]	D1253−86(92)		
Starch End point direct	330.3		4500−Cl B [18th, 19th, 20th]			
Back Titration either end point[15], or	330.2		4500−Cl C [18th, 19th, 20th]			
DPD−FAS,	330.4		4500−Cl F [18th, 19th, 20th]			
Spectrophotometric, DPD; or	330.5		4500−Cl G [18th, 19th, 20th]			
Electrode						Note 16
18. Chromium VI dissolved, ug/L: 0.45 micron filtration with:						
Extraction and atomic absorption,	218.4	7197	3111 C [18th, 19th]		I−1232−85	
Coprecipitation and atomic absorption,		7195				
Differential pulse polarography,		7198				
Colorimetric (Diphenylcarbazide), or		7196A	3500−Cr B [20th] and 3500−Cr D [18th, 19th]	D1687−92(A)	I−1230−85	
Ion Chromatography	218.6[5]					
19. Chromium, mg/L: Digestion[4] (optional extraction) followed by:						
AA direct aspiration[4m,36],	218.1	7190	3111 B [18th, 19th]	D1687−92(B)	I−3236−85	974.24[3]
AA chelation extraction,	218.3		3111 C [18th, 19th]			
AA furnace,	218.2 or 200.9[5]	7191	3113B [18th, 19th]	D1687−92(C)	I−3233−93[46]	
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
Direct current plasma[4m,36],				D4190−94		Note 34
or Colorimetric (diphenylcarbazide),			3500−Cr B [20th] and 3500−Cr D [18th, 19th]			
20. Cobalt, mg/L: Digestion[4] followed by:						
AA direct aspiration,	219.1	7200	3111 B [18th, 19th]	D3558−94 (A or B)	I−3239−85	
AA furnace, or	219.2 or 200.9[5]	7201	3113 B [18th, 19th]	D3558−94(C)	I−4243−89[51]	
Inductively coupled plasma, or	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				
Direct current plasma				D4190−94		Note 34

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
21. Color, Platinum Cobalt units or dominant wavelength hue,						
luminance, purity: Colorimetric, ADMI	110.1		2120 E [18th, 19th, 20th]			Note 18
Platinum cobalt; or	110.2		2120 B [18th, 19th, 20th]		I−1250−85	
Spectrophotometric	110.3		2120 C [18th, 19th, 20th]			
22. Copper, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	220.1	7210	3111 B or C [18th, 19th]	D1688−95 (A or B)	I−3271−85 or I−3270−85	974.27[3]
AA furnace,	220.2 or 200.9[5]	7211	3113 B [18th, 19th]	D1688−95(C)	I−4274−89[51]	
Inductively coupled plasma[4m,36]	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A		D4190−94		
Direct current plasma[4m,36],						Note 34
Colorimetric (Neocuproine), or			3500−Cu B [20th] and 3500−Cu D [18th, 19th]			
Bicinchoninate			3500−Cu C [20th]			Note 19
23. Cyanide − Total, ug/L:						
Manual distillation with $MgCl_2$			4500−CN−C [18th, 19th, 20th]	D2036−98(A)		
Followed by: titrimetric,			4500−CN−D [18th, 19th, 20th]			
Manual or	335.2[31]	9010C	4500−CN−E [18th, 19th, 20th]	D2036−98(A)	I−3300−85	
Automated[20] spectrophotometric, or	335.3[31]	9010C			I−4302−85	
Semi−automated colorimetry	335.4[1m]	9012B				
24. Cyanide, Available, ug/L:						
Cyanide amenable to chlorination (CATC), Manual distillation with $MgCl_2$ followed by titrimetric, manual or automated spectrophotometric	335.1	9010C	4500−CN−G [18th, 19th, 20th]	D2036−98(B)		
Flow injection and ligand exchange followed by amperometry						OIA−1677[44]
25. Fluoride − Total, mg/L:						
Manual distillation[8]			4500−F−B [18th, 19th, 20th]			
Followed by manual or	340.2		4500−F−C [18th, 19th, 20th]	D1179−93(B)		
automated electrode,					I−4327−85	
SPADNS,	340.1		4500−F−D [18th, 19th, 20th]	D1179−93(A)		
Ion chromatography,	300.0[1m]	9056				
Or automated complexone	340.3		4500−F−E [18th, 19th, 20th]			

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
26. Gold, mg/L:						
Digestion[4] followed by:						
AA direct aspiration	231.1		3111 B [18th, 19th]			
AA furnace,	231.2		3113 B [18th, 19th]			
Direct current plasma, or						Note 34
Inductively coupled plasma	200.7[5]	6010C				
27. Hardness − Total as $CaCO_3$, mg/L:						
Automated colorimetric,	130.1					
EDTA titration,	130.2		2340 B or C [18th, 19th, 20th]	D1126−86(92)	I−1338−85	973.52B[3]
or the sum of Ca and Mg as their respective carbonates (by ICP or AA direct aspiration) (See Parameters 13 and 33)						
28. Hydrogen ion (pH), pH units:						
Electrometric Measurements or	150.1	9040C	4500−H^+ B [18th, 19th, 20th]	D1293−84(90) (A or B)	I−1586−85	973.41[3]
Automated Electrode					I−2587−85	Note 21
29. Iridium, ug/L:						
Digestion[4] followed by:						
AA direct aspiration,	235.1		3111 B [18th, 19th]			
AA furnace, or	235.2					
Inductively coupled plasma	200.7[5]	6010C				
30. Iron, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	236.1	7380	3111 B or C [18th, 19th]	D1068−96 (A or B)	I−3381−85	974.27[3]
AA furnace,	236.2 or 200.9[5]	7381	3113 B [18th, 19th]	D1068−96(C)		
Inductively coupled plasma[4m,36],	200.7[5]	6010A	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Direct current plasma[4m,36], or				D4190−94		Note 34
Colorimetric (Phenanthroline)			3500−Fe B [20th] and 3500−Fe D [18th, 19th]	D1068−96(D)		Note 22

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
31. Kjeldahl nitrogen − Total (as N), mg/L:						
Digestion and distillation	351.3		4500−N_{org} B or C [18th, 19th, 20th] and 4500−NH_3 B [18th, 19th, 20th]	D3590−89(A)		
	351.3			D3590−89(A)		937.46[3]
Followed by titration	351.3		4500−NH_3 C [18th]	D3590−89(A)		
Nesslerization or	351.3		4500−NH_3 C [19th, 20th] and 4500−NH_3 E [18th]			
Electrode,						
	351.1		4500−NH_3 G [18th, 19th, 20th]		I−4551−78[8]	
Automated phenate,						
	351.2[1m]			D3590−89(B)	I−4515−91[45]	
Semi−automated block digester,	351.4			D3590−89(A)		
Or potentiometric						Note 39
Block digester, followed by Auto distallation and Titration, or						Note 40
Nesslerization, or						Note 41
Flow injection gas diffusion						
32. Lead, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	239.1	7420	3111 B or C [18th, 19th]	D3559−90 (A or B)	I−3399−90	974.27[3]
AA furnace,	239.2 or 200.9[5]	7421	3113 B [18th, 19th]	D3559−90(C)	I−4403−89[51]	
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				
Direct current plasma[4m,36],				D4190−94		Note 34
Voltametry[11] or				D3559−90(C)		
Colorimetric (Dithizone)			3500−Pb B [20th] and 3500−Pb D [18th, 19th]			
33. Magnesium, mg/L:						
Digestion[4] followed by:						
Atomic absorption,	242.1	7450	3111 B [18th, 19th]	D511−93(B)	I−3447−85	974.27[3]
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Direct current plasma, or						Note 34
Gravimetric			3500−Mg D [18th, 19th]			

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
34. Manganese, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	243.1	7460	3111 B [18th, 19th]	D858−90 (A or B)	I−3454−85	974.27[3]
AA furnace,	243.2 or 200.9[5]	7461	3113 B [18th, 19th]	D858−90 (C)		
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
Direct current plasma[4m,36],				D4190−94		Note 34
Colorimetric (Persulfate), or			3500−Mn D [20th] and 3500−Mn D [18th, 19th]			920.203[3]
Periodate						Note 23
35. Mercury − Total[6], ug/L:						
Cold vapor AA, manual or automated, or	245.1[5] 245.2	7470A	3112 B [18th, 19th, 20th]	D3223−91	I−3462−85	977.22[3]
Oxidation, purge and trap, cold vapor atomic fluorescence spectrometry (ng/l)[43m]	1631E[43]					
Oxidation, cold vapor atomic fluorescence spectrometry (ng/l)[43m]	245.7[5]					
35m. Mercury − Hg(II) and organomercurials, ug/L:						
HPLC with electrochemical detection	245.3[5]					
36. Molybdenum, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	246.1	7480	3111 D [18th, 19th]		I−3490−85	
AA furnace,	246.2	7481	3113 B [18th, 19th]		I−3492−96[47]	
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry, or	200.8[5]	6020A				
Direct current plasma						Note 34
37. Nickel, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	249.1	7520	3111 B or C [18th, 19th]	D1886−90 (A or B)	I−3499−85	
AA furnace,	249.2 or 200.9[5]		3113 B [18th, 19th]	D1886−90 (C)	I−4503−89[51]	
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
Direct current plasma[4m,36], or				D4190−94		Note 34
Colorimetric (Heptoxime)			3500−Ni D [17th]			
38. Nitrate (as N), mg/L:						
Brucine sulfate, or	352.1					973.50[3] 419D[17]
Nitrate−nitrite N minus Nitrite N (see parameters 39 and 40)						
Ion chromatography[25m]	300.0[1m]	9056				

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
39. Nitrate−nitrite (as N), mg/L:						
Cadmium reduction, manual	353.3		4500−NO_3 E [18th, 19th, 20th]	D3867−99(B)		
or automated, or	353.2[1m]		4500−NO_3 F [18th, 19th, 20th]	D3867−99(A)	I−4545−85	
automated hydrazine	353.1		4500−NO_3 H [18th, 19th, 20th]			
Ion chromatography[25m]	300.0[1m]	9056				
40. Nitrite (as N), mg/L:						
Spectrophotometric, manual or	354.1		4500−NO_2 B [18th, 19th, 20th]			Note 25
automated (Diazotization),					I−4540−85	
Automated (bypass cadmium reduction), or	353.2 Rev 2.0					
Ion chromatography[25m]	300.0[1m]	9056				
41. Oil and grease−Total recoverable, mg/L:						
Gravimetric (freon extraction)	413.1	9070	5520 B [18th, 19th, 20th][38]			
Oil and grease and non−polar material, mg/L:	1664A[42]		5520 B [18th, 19th, 20th][38]			
Hexane extractable material (HEM): n−hexane extraction and gravimetry.						
Silica gel treated HEM (SGT−HEM): Silica gel treatment and gravimetry.	1664A[42]					
42. Organic carbon − Total (TOC), mg/L:						
Combustion or oxidation,	415.1	9060A	5310 B C, or D [18th, 19th, 20th]	D2579−93 (A or B)	p. 14[24]	973.47[3]
Persulfate oxidation	415.2[1m]		5310 C [18th, 19th, 20th]			
Organic Halides, Adsorbable (AOX), ug/L						
Adsorption and coulometric titration	1650[42m]					
43. Organic nitrogen (as N), mg/L:						
Total Kjeldahl N (Parameter 31)						
minus ammonia N (Parameter 4)						
44. Orthophosphate (as P), mg/L:						
Ascorbic acid method, automated	365.1		4500−P F [18th, 19th, 20th]		I−4601−85	973.56[3]
Or manual single reagent or	365.2		4500−P E [18th, 19th, 20th]	D515−88(A)		973.55[3]
Manual two reagent, or	365.3					
Ion chromatography	300.0[1m]	9056				
45. Osmium, ug/L:						
Digestion[4] followed by:						
AA direct aspiration,	252.1	7550	3111 D [18th, 19th]			
AA furnace, or	252.2					
Inductively coupled plasma	200.7[5]	6010C				

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
46. Oxygen, dissolved, mg/L:						
Winkler (Azide modification)	360.2		4500−O C [18th, 19th, 20th]	D888−92(A)	I−1575−78[8]	973.45B[3]
Or electrode	360.1		4500−O G [18th, 19th, 20th]	D888−92(B)	I−1576−78[8]	
47. Palladium, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	253.1		3111 B [18th, 19th]			p. S27[10]
AA furnace,	253.2					p. S28[10]
Direct current plasma, or						Note 34
Inductively coupled plasma	200.7[5]	6010C				
48. Phenols, ug/L:						
Manual distillation[26]	420.1		5530 B [18th, 19th, 20th]			Note 27
Followed by manual	420.1	9065	5530 D [18th, 19th, 20th]			
Or automated[20] colorimetric (4AAP), or	420.2	9066				Note 27
Semi−automated colorimetric	420.4[1m]					
49. Phosphorus (elemental), mg/L:						
Gas−Liquid chromatography						Note 28
50. Phosphorus − Total, mg/L:						
Persulfate digestion	365.2		4500−P B,5 [18th, 19th, 20th]			973.55[3]
Followed by manual or	365.2 or 365.3		4500−P E [18th, 19th, 20th]	D515−88 (A)		
Automated ascorbic acid	365.1[1m]		4500−P F [18th, 19th, 20th]		I−4600−85	973.56[3]
Reduction, or semi−automated block digestor	365.4			D515−88 (B)	I−4610−91[48]	
51. Platinum, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	255.1		3111 B [18th, 19th]			
AA furnace,	255.2					
Direct current plasma, or						Note 34
Inductively coupled plasma	200.7[5]	6010C				
52. Potassium, mg/L:						
Digestion[4] followed by:						
Atomic absorption,	258.1	7610	3111 B [18th, 19th]		I−3620−85	973.53[3]
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Flame photometric, or			3500−K B [20th] and 3500−K D [18th, 19th]			
Colorimetric (cobalt nitrate)						317B[17]
53. Residue − total, (total solids), mg/L:						
Gravimetric 103−105°C	160.3		2540 B [18th, 19th, 20th]		I−3750−85	
54. Residue − filterable, (TDS), mg/L:						
Gravimetric, 180°C	160.1		2540 C [18th, 19th, 20th]		I−1750−85	

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
55. Residue − nonfilterable, (TSS), mg/L: Gravimetric, 103−105°C post washing of residue	160.2		2540 D [18th, 19th, 20th]		I−3765−85	
56. Residue − settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5		2540 F [18th, 19th, 20th]			
57. Residue − volatile mg/L: Gravimetric, 550°C	160.4				I−3753−85	
58. Rhodium, ug/L: Digestion[4] followed by:						
AA direct aspiration,	265.1		3111 B [18th, 19th]			
AA furnace, or	265.2					
Inductively coupled plasma	200.7[5]	6010C				
59. Ruthenium, ug/L: Digestion[4] followed by:						
AA direct aspiration,	267.1		3111 B [18th, 19th]			
AA furnace, or	267.2					
Inductively coupled plasma	200.7[5]	6010C				
60. Selenium, ug/L: Digestion[4] followed by:						
AA furnace,	270.2 or 200.9[9]	7740	3113 B [18th, 19th]	D3859−98(B)	I−4668−98[49]	
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
or AA (gaseous hydride)		7741A	3114 B[4g] [18th, 19th, 20th]	D3859−98(A)	I−3667−85	
61. Silica − Dissolved, mg/L: 0.45 micron filtration:						
Followed by manual or	370.1		4500−Si C [20th] and 4500−Si D [18th, 19th]	D859−94	I−1700−85	
automated colorimetric (Molybdosilicate), or					I−2700−85	
Inductively coupled plasma[6]	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
62. Silver[29], mg/L: Digestion[4] followed by:						
AA direct aspiration,		7760A	3111 B or C [18th, 19th]		I−3720−85	974.27[3]
AA furnace,	200.9[5]	7761	3113 B [18th, 19th]		I−4724−89[51]	
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
Or direct current plasma						Note 34

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
63. Sodium, mg/L:						
Digestion[4] followed by:						
Atomic absorption,	273.1	7770	3111 B [18th, 19th]		I−3735−85	973.54[3]
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Direct current plasma, or						Note 34
Flame photometric			3500−Na B [20th] and 3500−Na D [18th, 19th]			
64. Specific conductance, micromhos/cm at 25°C: Wheatstone bridge	120.1	9050A	2510 B [18th, 19th, 20th]	D1125−95(A)	I−2781−85	973.40[3]
65. Sulfate (as SO_4), mg/L:						
Automated colorimetric (barium chloroanilate),	375.1	9035				
Semi−automated colorimetric (methylthymol blue)	375.2[1m]	9036				
	375.3		4500−SO_4^2 C or D [18th, 19th, 20th]			925.54[3]
Gravimetric,	375.4	9038		D516−90		426C[30]
Turbidimetric, or	300.0[1m]	9056				
Ion chromatography						
66. Sulfide (as S), mg/L:						
Titrimetric (iodine) or	376.1		4500−S^{2-}F [19th, 20th] and 4500−S^{2-}E [18th]		I−3840−85	
Colorimetric (methylene blue)	376.2		4500−S^{2-}D [18th, 19th, 20th]			
67. Sulfite (as SO_3), mg/L:						
Titrimetric (iodine−iodate)	377.1		4500−$S0_3^{2-}$ B [18th, 19th, 20th]			
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1		5540 C [18th, 19th, 20th]	D2330−88		
69. Temperature, °C: Thermometric	170.1		2550 B [18th, 19th, 20th]			Note 32
70. Thallium, ug/L:						
Digestion[4] followed by:						
AA direct aspiration,	279.1	7840	3111 B [18th, 19th]			
AA furnace,	279.2 or 200.9[5]	7841	3113 B [18th, 19th]			
Inductively coupled plasma, or	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry	200.8[5]	6020A				
71. Tin, ug/L:						
Digestion[4] followed by:						
AA direct aspiration,	282.1	7870	3111 B [18th, 19th]		I−3850−78[8]	
AA furnace, or	282.2 or 200.9[5]		3113 B [18th, 19th]			
Inductively coupled plasma	200.7[5]	6010C				

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW−846[3c,3i]	Standard Methods[3p]	ASTM[3w]	USGS[2]	Other
72. Titanium, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	283.1		3111 D [18th, 19th]			
AA furnace,	283.2		3113 B [18th, 19th]			
Direct current plasma, or						Note 34
Inductively coupled plasma	200.7[5]	6010C				
73. Turbidity, NTU: Nephelometric	180.1[1m]		2130 B [18th, 19th, 20th]	D1889−94(A)	I−3860−85	
74. Vanadium, mg/L:						
Digestion[4] followed by:						
AA direct aspiration,	286.1	7910	3111 D [18th, 19th]			
AA furnace,	286.2	7911	3113 B [18th, 19th]	D3373−93		
Inductively coupled plasma,	200.7[5]	6010C	3120 B [18th, 19th, 20th]		I−4471−97[50]	
Inductively coupled plasma−mass spectrometry	200.8[5]					
Direct current plasma, or				D4190−94		Note 34
Colorimetric (Gallic acid)			3500−V B [20th] and 3500−V D [18th, 19th]			
75. Zinc, mg/L:						
Digestion[4] followed by:						
AA direct aspiration[4m,36],	289.1	7950	3111 B or C [18th, 19th]		I−3900−85	974.27[3]
AA furnace,	289.2 or 200.9[5]	7951	3113 B [18th, 19th]			
Inductively coupled plasma[4m,36],	200.7[5]	6010C	3120 B [18th, 19th, 20th]			
Inductively coupled plasma−mass spectrometry,	200.8[5]	6020A				
Direct current plasma[4m,36],				D4190−94		Note 34
Colorimetric (Dithizone), or			3500−Zn E [18th, 19th]			
Colorimetric (Zincon)			3500−Zn B [20] and 3500−Zn F [18th, 19th]			Note 33

[1] "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory−−Cincinnati (EMSL−CI), EPA−600/4−79−020, Revised March 1983 and 1979 where applicable. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487−4650.

[1m] "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA−600/R−93−100, Environmental Protection Agency, August 1993, Office of Research and Development, Washington D.C. 20460, August 1993. Available from: NTIS, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487−4650.

[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. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[3] "Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 15th ed. (1990). Available from: The Association of Official Analytical Chemists, 1111 N. 19th Street, Suite 210, Arlington, VA 22209.

[3c] "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.

[3i] SW−846 series 7000 methods include SW−846 methods 7000B and 7010, the general AA method descriptions.

[3p] "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.

[3w] "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1994, 1996, and 1999. Available from: the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

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[4] A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. The required digestion procedure(s) for a particular metals analysis is listed in Table BM, Metals Digestion Procedures. Use of the graphite furnace AA technique, inductively coupled plasma, direct current plasma, as well as determination for certain elements such as arsenic, mercury, selenium, silver, and titanium require a modified digestion procedure. In all cases, the analytical method should be consulted for specific instructions and cautions.

If a digestion procedure is given in the determinative method for any of the metals in table B, and this digestion is not listed in table BM, the procedure given in the analytical method should be used; however, if the digestion included in one of the approved non−EPA references (e.g. "Standard Methods for the Examination of Water and Wastewater") is significantly different from one of the EPA procedures listed in table BM, then the EPA procedure from table BM should be used.

Sample digestion may be omitted for AA (direct aspiration or graphite furnace), direct current plasma, and inductively coupled plasma analyses provided the sample solution to be analyzed meets the following criteria:
(a) has a low COD (<20),
(b) is visibly transparent with a turbidity measurement of 1 NTU or less,
(c) is colorless with no perceptible odor, and
(d) is of one liquid phase and free of particulate or suspended matter following acidification.

[4g] Use the digestion given in the method.

[4m] "Test Methods for Evaluating Solid Waste", SW−846 method 3015A. United States EPA SW−846, 3rd Edition and updates. Footnote 3c lists the complete reference.

[5] "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.

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

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

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

[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). Available on inter−library loan.

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

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

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

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

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

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

[17] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976. Available on inter−library loan.

[18] "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.

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

[20] After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re−sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

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

[22] Iron, 1,10−Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, PO Box 389, Loveland, CO 80537. Available from: Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[23] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2−113 and 2−117, Hach Chemical Company, Loveland, CO 80537. Available from: Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[24] Wershaw, R.L., et al, "Methods for Analysis of Organic Substances in Water," Techniques of Water−Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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

[25m] Nitrate−nitrite determinations by ion chromatography must be analyzed within 48 hours.

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

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

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

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

[30] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition. Available on inter−library loan.

[31] EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.

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

[33] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2−231 and 2−333, Hach Chemical Company, Loveland, CO 80537. Available from: Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

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

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

[36] "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, PO Box 200, Matthews, NC 28106−0200, April 16, 1992. Available from: the CEM Corporation.

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

[38] Only use Trichlorotrifluorethane (1,1,2−trichloro−1,2,2−trifluoroethane; CFC−113) extraction solvent when determining Total Recoverable Oil and Grease (analogous to EPA Method 413.1). Only use n−hexane extraction solvent when determining Hexane Extractable Material (analogous to EPA Method 1664A). Use of other extraction solvents is strictly prohibited.

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

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

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

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

[42m] 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.

[43] USEPA. 2002. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry". September 2002. Office of Water, U.S. Environmental Protection Agency (EPA−821−R−02−019). The application of clean techniques described in EPA's draft Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels (EPA−821−R−96−011) are recommended to preclude contamination at low−level, trace metal determinations. Available at NTIS, PB−121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.

[43m] 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.

[44] Available Cyanide, Method OIA−1677 (Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry), ALPKEM, A Division of OI Analytical, PO Box 9010, College Station, TX 77842− 9010.

[45] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method", Open File Report (OFR) 00−170. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[46] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry", Open File Report (OFR) 93−449. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[47] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry", Open File Report (OFR) 97−198. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[48] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis" Open File Report (OFR) 92−146. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[49] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace−Atomic Absorption Spectrometry" Open File Report (OFR) 98−639. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[50] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Elements in Whole−water Digests Using Inductively Coupled Plasma−Optical Emission Spectrometry and Inductively Coupled Plasma−Mass Spectrometry", Open File Report (OFR) 98−165. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[51] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment", Open File Report (OFR) 93−125. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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]	EPA Method Number[2,7] GC	EPA Method Number[2,7] GC/MS	Standard Methods[2f]	EPA Method Number[2m] GC	EPA Method Number[2m] GC/MS	Other
I. VOLATILES		**624[4]**		**8021B**	**8260B**	
A. Halogenated volatiles	**601**	**1624B**	**6230 B[20th], 6210B[18th,19th], 6200 B[20th], 6200 C [20th]**	———	———	
Bromodichloromethane						
Bromoform						

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

Bromomethane						
Carbon tetrachloride			not 6210 B, not 6200 B			Note 3, p.130
Chloroethane						
2−Chloroethylvinyl ether						
Chloroform						Note 3, p.130
Chloromethane						
Dibromochloromethane						
Dichlorodifluoromethane		———	not 6210 B, not 6200 B			
1,1−Dichloroethane						
1,2−Dichloroethane						
1,1−Dichloroethene						
trans−1,2−Dichloroethene						
1,2−Dichloropropane						
cis−1,3 Dichloropropene						
trans−1,3−Dichloropropene						
Methylene chloride			not 6210 B, not 6200 B			Note 3, p.130
1,1,2,2−Tetrachloroethane						Note 3, p.130
Tetrachloroethene						Note 3, p.130
1,1,1−Trichloroethane						
1,1,2−Trichloroethane						Note 3, p.130
Trichloroethene						
Trichlorofluoromethane		———				
Vinyl chloride						
B. Aromatic volatiles	**602**		**6220 B [18th,19th], 6200 B [20th], 6200 C [20th]**	———	———	
Benzene		1624B	6210 B [18th,19th]			
Chlorobenzene	601	1624	6210 B [18th,19th], 6230 B [18th,19th]			Note 3, p.130
1,2−Dichlorobenzene	601, 612	625, 1625B	6230 B [18th,19th], 6410 B [18th,19th,20th]			
1,3−Dichlorobenzene	601, 612	625, 1625B	6230 B[18th,19th], 6410 B [18th,19th,20th]			
1,4−Dichlorobenzene	601, 612	625, 1625B	6230 B[18th,19th], 6410 B [18th,19th,20th]			
Ethylbenzene		1624B	6210 B [18th,19th]			
Toluene		1624B	6210 B [18th,19th]			
C. Other volatiles	**603**	**1624B,624[4]**		**8030A**	**8260B**	
Acrolein						8315A[2m]
Acrylonitrile				8031		8316[2m]
II. PHENOLS	**604**	**625, 1625B**	**6410 B, 6420 B [18th, 19th, 20th]**	**8041A**	**8270D**	**Note 9, p. 27**
4−Chloro−3−methylphenol						
2−Chlorophenol						
2,4−Dichlorophenol						
2,4−Dimethlyphenol						
2,3−Dinitrophenol			Not 6410 B			
2,4−Dinitrophenol						
2,6−Dinitrophenol			Not 6410 B			
2−Methyl−4,6−dinitrophenol						
2−Nitrophenol						
4−Nitrophenol						
Pentachlorophenol		1653[9s]				Note 3, p.140
Phenol						
Trichlorosyringol	Not 604	1653[9s], not 625 or 1625B				

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

3,4,5−Trichlorocatechol	Not 604	1653[9s], not 625 or 1625B				
3,4,6−Trichlorocatechol	Not 604	1653[9s], not 625 or 1625B				
3,4,5−Trichloroguaiacol	Not 604	1653[9s], not 625 or 1625B				
3,4,6−Trichloroguaiacol	Not 604	1653[9s], not 625 or 1625B				
4,5,6−Trichloroguaiacol	Not 604	1653[9s], not 625 or 1625B				
2,4,5−Trichlorophenol	Not 604	1653[9s], not 625 or 1625B				
2,4,6−Trichlorophenol		1653[9s]				
Tetrachlorocatechol	Not 604	1653[9s], not 625 or 1625B				
Tetrachloroguaiacol	Not 604	1653[9s], not 625 or 1625B				
2,3,4,6−Tetrachlorophenol	Not 604	1653[9s], not 625 or 1625B				
III. PHTHALATE ESTERS	**606**	**625, 1625B**	**6410 B [18th,19th,20th]**	**8061A**	**8270D**	**Note 9, p. 27**
Benzyl butyl phthalate						
Bis(2−ethylhexyl)phthalate						
Diethyl phthalate						
Dimethyl phthalate						
Di−n−butyl phthalate						
Di−n−octyl phthalate						
IV. NITROSAMINES	**607**	**625, 1625B**	**6410 B [18th,19th,20th]**	**——**	**8270D**	**Note 9, p. 27**
N−Nitrosodimethylamine		Note 5				
N−Nitrosodi−n−propylamine						
N−Nitrosodiphenylamine		Note 5				
V. POLYCHLORINATED BIPHENYLS	**608**	**625**	**6410 B [18th,19th,20th]**	**8081B**	**8270D**	**Note 3, p.43**
PCB−1016						
PCB−1221						
PCB−1232						
PCB−1242						
PCB−1248						
PCB−1254						
PCB−1260						
VI. NITROAROMATICS &CYCLIC KETONES	**609**	**625, 1625B**	**6410 B [18th,19th,20th]**	**8091**	**8270D**	**Note 9, p. 27**
2,3−Dinitrotoluene						
2,4−Dinitrotoluene						
2,6−Dinitrotoluene						
Isophorone						
Nitrobenzene						Note 9m
VII. POLYNUCLEAR AROMATIC HYDROCARBONS	**610/FID**	**625, 1625B**	**6410 B, 6440 B [18th,19th,20th]**	**——**	**8270D**	**Note 9, p. 27, Note 9m, 610[2], 8310[2m]**
Acenaphthene						
Acenaphthylene						
Anthracene						
Benzo(a)anthracene						
Benzo(a)pyrene						

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

Benzo(b)fluoranthene						
Benzo(g,h,i)perylene						
Benzo(k)fluoranthene						
Chrysene						
Dibenzo(a,h)anthracene						
Fluoranthene						
Fluorene						
Ideno (1,2−3−cd)pyrene						
Naphthalene				8021B		
Phenanthrene						
Pyrene						
VIII. HALOETHERS	**611**	**625, 1625B**	**6410 B [18th,19th,20th]**	**8111**	**8270D**	**Note 9, p. 27**
Bis(2−chloroethoxy) methane						
Bis(2−chloroethyl)ether						
4−Bromophenylphenyl ether						
4−Chlorophenylphenyl ether						
2,2−Oxybis (2−chloropropane)						
IX. CHLORINATED HYDROCARBONS	**612**	**625, 1625B**	**6410 B [18th,19th,20th]**	**8121**	**8270D 8260B**	
Benzidine	———	Note 5	———	———	not 8260B	LC: 605[2]
Benzyl chloride	———	———	———		not 8270D	Note 3, p.130; Note 6, p.S102
2−Chloronaphthalene					not 8260B	8410[2m], Note 9, p. 27
3,3−Dichlorobenzidine	———			———	not 8260B	LC: 605[2]
Epichlorohydrin	———	———	———		not 8270D	Note 3, p.130; Note 6, p.S102
Hexachlorobenzene				8081B	not 8260B	8410[2m], Note 9, p. 27
Hexachlorobutadiene				8021B		8410[2m], Note 9, p. 27
Hexachlorocyclopentadiene		Note 5		8081B	not 8260B	8410[2m], Note 9, p. 27
Hexachloroethane	Not 612, 616					8410[2m], Note 9, p. 27
1,2,4−Trichlorobenzene				8021B		Note 3, p.130, Note 9, p. 27
X. POLYCHLORINATED DIBENZO−P−DIOXINS AND FURANS		**1613 B[9f]**		**———**	**8280B, 8290A**	
1,2,3,4,6,7,8−Heptachlorodibenzo−p−dioxin						
1,2,3,4,6,7,8−Heptachlorodibenzofuran						
1,2,3,4,7,8,9−Heptachlorodibenzofuran						
1,2,3,4,7,8−Hexachlorodibenzo−p−dioxin						
1,2,3,6,7,8−Hexachlorodibenzo−p−dioxin						
1,2,3,7,8,9−Hexachlorodibenzo−p−dioxin						
1,2,3,4,7,8−Hexachlorodibenzofuran						
1,2,3,6,7,8−Hexachlorodibenzofuran						
1,2,3,7,8,9−Hexachlorodibenzofuran						
2,3,4,6,7,8−Hexachlorodibenzofuran						
Octachlorodibenzo−p−dioxin						
Octachlorodibenzofuran						
1,2,3,7,8−Pentachlorodibenzo−p−dioxin						
1,2,3,7,8−Pentachlorodibenzofuran						
2,3,4,7,8−Tetrachlorodibenzo−p−dioxin						
2,3,7,8−Tetrachlorodibenzo−p−dioxin		613[5m]				
2,3,7,8−Tetrachlorodibenzofuran						

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

[2] The full text of Methods 601−613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants", of 40 CFR 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 40 CFR Part 136. Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[2f] "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.

[2m] "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.

[3] "Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater", U.S. Environmental Protection Agency, September, 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

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

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N−nitrosodimethylamine, and N−nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.

[7] Each Analyst must make an initial, one−time demonstration of their ability to generate acceptable precision and accuracy with Methods 601−603, 624, 625, 1624B, and 1625B (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on−going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624B and 1625B) of all samples to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Note: These warning limits are promulgated as an "interim final action with a request for comments."

[8] "Organochlorine Pesticides and PCBs in Wastewater Using Empore TM Disk" 3M Corporation, Revised 10/28/94. Method available from: 3M Corporation, 3M Center Building 220–9E–10, St. Paul, MN 55144–1000.

[9] USGS Method 0−3116−87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory−−Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments", U.S. Geological Survey, Open File Report 93−125. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[9f] Method 1613: Tetra− through Octa− Chlorinated Dioxins and Furans by Isotope Dilution, HRGC/HRMS, Environmental Protection Agency, Federal Register, Office of Water, September 1994, Revision B, EPA 821−B−94−005. Available from: The Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.

[9m] Method D4657−92, "Annual Book of Standards− Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from: the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[9s] The full text of Method 1653 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.

TABLE D

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

Parameter		Method	EPA[2,7]	SW−846[A]	Standard Methods[B]	ASTM[C]	Other
1.	Aldrin	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
2.	Ametryn	GC					Note 3, p.83; Note 6, p.S68
3.	Aminocarb	HPLC					Note 3m
4.	Atraton	GC					Note 3, p.83; Note 6, p.S68
5.	Atrazine	GC		8141B			Note 3, p.83; Note 6, p.S68
6.	Azinphos methyl	GC		8141B			Note 3, p.25; Note 6, p.S51
		GC/MS		8270D			
7.	Barban	HPLC					Note 3m
		GC/MS		8270D			
8.	α−BHC	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 8.
		GC/MS	625[5]	8270D	6410 B		
9.	β−BHC	GC	608	8081B	6630 C	D3086−90	Note 8.
		GC/MS	625	8270D	6410 B		
10.	δ−BHC	GC	608	8081B	6630 C	D3086−90	Note 8.
		GC/MS	625[5]	8270D	6410 B		
11.	γ−BHC (Lindane)	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		

TABLE D (Continued)

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

12.	Captan	GC			6630 B	D3086−90	Note 3, p.7.
		GC/MS		8270D			
13.	Carbaryl	HPLC					Note 3m
		GC/MS		8270D			
14.	Carbophenothion	GC		8141B			Note 4, p.27; Note 8; Note 6, p.S73.
		GC/MS		8270D			
15.	Chlordane	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p. 27; Note 8
		GC/MS	625	8270D	6410 B		
16.	Chloropropham	HPLC					Note 3m
17.	2,4−D	GC		8151A	6640 B		Note 3, p.115; Note 4, p.40.
18.	4,4'−DDD	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
19.	4,4'−DDE	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
20.	4,4'−DDT	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
21.	Demeton−O	GC		8141B			Note 3, p.25; Note 6, p.S51.
		GC/MS		8270D			
22.	Demeton−S	GC		8141B			Note 3, p.25; Note 6, p.S51.
		GC/MS		8270D			
23.	Diazinon	GC		8141			Note 3, p.25; Note 4, p.27; Note 8; Note 6, p.S51
24.	Dicamba	GC		8151A			Note 3, p.115
25.	Dichlofenthion	GC		8141			Note 4, p.27; Note 8.; Note 6, p.S73
26.	Dichloran	GC			6630 B & C		Note 3, p.7
27.	Dicofol	GC				D3086−90	
28.	Dieldrin	GC	608	8081B	6630 B & C		Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
29.	Dioxathion	GC		8141B			Note 4, p.27; Note 8; Note 6, p.S73
		GC/MS		8270D			
30.	Disulfoton	GC		8141B			Note 3, p.25; Note 6, p.S51
		GC/MS		8270D			
31.	Diuron	HPLC					Note 3m
32.	Endosulfan I	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p. 27; Note 8.
		GC/MS	625[5]	8270D	6410 B		
33.	Endosulfan II	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 8
		GC/MS	625[5]	8270D	6410 B		
34.	Endosulfan sulfate	GC	608	8081B	6630 C		Note 8
		GC/MS	625	8270D	6410 B		
35.	Endrin	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625[5]	8270D	6410 B		
36.	Endrin aldehyde	GC	608	8081B		D3086−90	Note 8.
		GC/MS	625	8270D	6410 B		
37.	Ethion	GC		8141B			Note 4, p.27; Note 8; Note 6, p.S73
		GC/MS		8270D			
38.	Fenuron	HPLC					Note 3, p.104; Note 6, p.S64
39.	Fenuron−TCA	HPLC					Note 3m
40.	Heptachlor	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
41.	Heptachlor epoxide	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8; Note 6 p.S73
		GC/MS	625	8270D	6410 B		
42.	Isodrin	GC		8081B			Note 4, p.27; Note 8; Note 6, p.S73
		GC/MS		8270D			
43.	Linuron	HPLC					Note 3m
44.	Malathion	GC		8141B	6630 C		Note 3, p.25; Note 4, p.27; Note 8; Note 6, p.S51
		GC/MS		8270D			
45.	Methiocarb	HPLC					Note10
46.	Methoxychlor	GC		8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS		8270D			
47.	Mexacarbate	HPLC					Note 3m
		GC/MS		8270D			
48.	Mirex	GC		8081B	6630 B & C		Note 3, p.7; Note 4, p. 27.
		GC/MS		8270D			
49.	Monuron	HPLC					Note 3m
50.	Monuron−TCA	HPLC					Note 3m
51.	Neburon	HPLC					Note 3m
52.	Parathion methyl	GC		8141B	6630 C		Note 3, p.25; Note 4, p.27.

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TABLE D (Continued)

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

		GC/MS		8270D			
53.	Parathion ethyl	GC		8141B	6630 C		Note 3, p.25
		GC/MS		8270D			
54.	PCNB	GC		8081B	6630 B & C		Note 3, p.7
		GC/MS		8270B			
55.	Perthane	GC		8081B		D3086−90	Note 4, p. 27.
56.	Prometon	GC					Note 3, p.83; Note 6, p.S68; Note 9.
57.	Prometryn	GC					Note 3, p.83; Note 6, p.S68; Note 9.
58.	Propazine	GC					Note 3, p.83; Note 6, p.S68; Note 9.
59.	Propham	HPLC					Note 3m
60.	Propoxur	HPLC					Note 3m
61.	Secbumeton	HPLC					Note 3m
62.	Siduron	HPLC					Note 3m
63.	Simazine	GC		8141B			Note 3, p.83; Note 6, p.S68; Note 9.
64.	Strobane	GC		8081B	6630 B & C		Note 3, p.7
65.	Swep	HPLC					Note 3m
66.	2,4,5−T	GC		8151A	6640 B		Note 3, p.115; Note 4, p.40.
67.	2,4,5−TP (Silvex)	GC		8151A	6640 B		Note 3, p.115; Note 4, p. 40.
68.	Terbuthylazine	GC					Note 3, p.83; Note 6, p.S68
69.	Toxaphene	GC	608	8081B	6630 B & C	D3086−90	Note 3, p.7; Note 4, p.27; Note 8.
		GC/MS	625	8270D	6410 B		
70.	Trifluralin	GC		8081B	6630 B		Note 3, p.7; Note 9.
		GC/MS		8270D			

[A] "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.

[B] "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.

[C] "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1994, 1996, and 1999. Available from: The American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

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

[2] The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants", of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit", of this Part 136. Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September 1978. This EPA publication includes thin−layer chromatography (TLC) methods. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[3m] 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.

[4] "Methods for Analysis of Organic Substances in Water and Fluvial Sediments", Techniques of Water−Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] The method may be extended to include [alpha]−BHC, [gamma]−BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency". Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981). Available from: American Public Health Association, 1015 15th St., N.W., Washington, D.C. 20005.

[7] Each analyst must make an initial, one−time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on− going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited. Note: These warning limits are promulgated as an "Interim final action with a request for comments." Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[8] "Organochlorine Pesticides and PCBs in Wastewater Using Empore ™ Disk", 3M Corporation, Revised 10/28/94. Method available from: 3M Corporation, 3M Center Building 220−9E−10, St. Paul, MN 55144−1000.

[9] USGS Method 0−3106−93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory−−Determination of Triazine and Other Nitrogen−containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94−37. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

Table E
List of Approved Radiological Test Procedures For Wastewater

Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]
1. Alph−Total, pCi per liter	Proportional or Scintillation Counter	900.0	7110 B	D1943−90	pp. 75 and 78[5]
2. Alpha−Counting error, pCi per liter	Proportional or Scintillation Counter	Appendix B	7110 B	D1943−90	p. 79
3. Beta−Total, pCi per liter	Proportional Counter	900.0	7110 B	D1943−90	pp. 75 and 78[5]
4. Beta−Counting error, pCi	Proportional Counter	Appendix B	7110 B	D1943−90	p. 79
5. (a) Radium−Total	Proportional Counter	903.0	7500Ra B	D2460−90	
(b) ^{226}Ra, pCi per liter	Scintillation Counter	903.1	7500Ra C	D3454−90	p. 81

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

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

[3] "1991 Annual Book of Standards, Water" Section 11, American Society for Testing and Materials, 1980. Available from: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open−File Report 76−177 (1976)

[5] 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
Approved Analytical Methods For Sludge

Parameter	Sample Preparation	Method	Method Number
Metals[1]			
Arsenic	3050B	Inductively Coupled Plasma Emission	6010C
Arsenic	7061A	Gaseous Hydride[2]	7061A
Arsenic	3050B	Graphite Furnace	7010
Beryllium	3050B	Inductively Coupled Plasma Emission	6010C
Beryllium	3050B	Flame Atomic Absorption	7000B
Beryllium	3050B	Graphite Furnace	7010
Cadmium	3050B	Inductively Coupled Plasma Emission	6010C
Cadmium	3050B	Flame Atomic Absorption	7000B
Cadmium	3050B	Graphite Furnace	7010
Chromium	3050B	Inductively Coupled Plasma Emission	6010C
Chromium	3050B	Flame Atomic Absorption	7000B
Chromium	3050B	Graphite Furnace	7010
Copper	3050B	Inductively Coupled Plasma Emission	6010C
Copper	3050B	Flame Atomic Absorption	7000B
Lead	3050B	Inductively Coupled Plasma Emission	6010C
Lead	3050B	Flame Atomic Absorption	7000B
Lead	3050B	Graphite Furnace[3]	7010
Mercury	7471B	Cold Vapor	7471B
Molybdenum	3050B	Inductively Coupled Plasma Emission	6010C
Molybdenum	3050B	Graphite Furnace	7010
Nickel	3050B	Inductively Coupled Plasma Emission	6010C
Nickel	3050B	Flame Atomic Absorption	7000B
Selenium	3050B	Inductively Coupled Plasma Emission	6010C
Selenium	7741A	Gaseous Hydride[2]	7741A

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Table EM Approved Analytical Methods For Sludge			
Selenium	3050B	Graphite Furnace	7010
Zinc	3050B	Inductively Coupled Plasma Emission	6010C
Zinc	3050B	Flame Atomic Absorption	7000B
Organic[1]			
PCB (Aroclor or Congeners)	3540C or 3545A	Gas Chromatography	8082A[11]
PCB (Congeners)	1668A	Gas Chromatography/Mass Spectrometry	1668A[10,12]
Biological			
Enteric viruses	NA	Centrifuge Concentration	D 4994−89[4] Appendix H[8]
Fecal coliform	NA	Most Probable Number Membrane Filter	9221 E or 9222 D[5] or Appendix F[8]
Helminth ova	NA	Density Gradient Flotation	[6] or Appendix I[8]
Specific Oxygen Uptake Rate	NA	Respirometer	2710 B[5] or Appendix D.2.[8]
Salmonella	NA	Most Probable Number Selective Media Culture	9260 D.1[5] or Appendix G[8]
Physical			
Solids	NA	Gravimetric	2540 G[5]
Percent Volatiles Solids Reduction	NA	Calculation	Appendix D.1. & 3.[8]

[1] "Test Methods for Evaluating Solid Waste", Third Edition, SW–846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, September 1992, August 1993, September 1994, January 1995, December 1996, January 1998, and November 2000 updates, Washington, DC 20460. Available from: The Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250–7954, (202) 783–3238. Available online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

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

[3] Concentrations of lead in municipal sludge may exceed the working range of Graphite Furnace.

[4] "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.

[5] "Standard Methods for the Examination of Water and Wastewater", 18th, 19th, or 20th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1992, 1995, or 1998. Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[6] "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, (703) 487–4650.

[8] "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, (703) 487–4650.

[9] If an alternative digestion procedure is specified in the analytical method, the digestion in the method 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.

[10] EPA Method 1668 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.

[11] 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 lab 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

[12] "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, (703) 487–4650.

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.
triethlyamine	121−44−8	1666/1671.
xylenes	(Note 1)	1624C/1666.

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http://docs.legis.wisconsin.gov/code/admin_code

[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 Times for Wastewater

Parameter No./Name		Container[1]	Preservation[2,3]	Maximum holding time[4]
TABLE A − Bacterial Tests:				
1−7.	Bacteria	P,G	Cool, <10°C, 0.0008%, $Na_2S_2O_3$[5]	6 hours
8−9.	Protozoa	LDPE	0−8°C	96 hours[17a]
9−12.	Acute & chronic toxicity	P,G	Cool, ≤6°C[16]	36 hours
TABLE B − Inorganic Tests:				
1.	Acidity	P,G	Cool, ≤6°C	14 days
2.	Alkalinity	P,G	Cool, ≤6°C	14 days
4.	Ammonia	P,G	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
9.	Biochemical oxygen demand	P,G	Cool, ≤6°C	48 hours
11.	Bromide	P,G	None required	28 days
14.	Biochemical oxygen demand, carbonaceous	P,G	Cool, ≤6°C	48 hours
15.	Chemical oxygen demand	P,G	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
16.	Chloride	P,G	None required	28 days
17.	Chlorine, total residual	P,G	None required	Analyze immediately
21.	Color	P,G	Cool, ≤6°C	48 hours
23−24.	Cyanide: total, amenable to chlorination, and available	P,G	Cool, ≤6°C, NaOH to pH>12, 0.6g ascorbic acid[5]	14 days[6]
25.	Fluoride	P	None required	28 days
27.	Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
28.	Hydrogen ion (pH)	P,G	None required	Analyze immediately
31, 43.	Kjeldahl and organic nitrogen	P,G	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
38.	Nitrate	P,G	Cool, ≤6°C	48 hours
39.	Nitrate−nitrite	P,G	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
40.	Nitrite	P,G	Cool, ≤6°C	48 hours
41.	Oil and grease	G	Cool, ≤6°C, HCl or H_2SO_4 to pH<2	28 days
42.	Organic carbon	G	Cool, ≤6°C, HCl or H_2SO_4 or H_3PO_4 to pH<2	28 days
44.	Orthophosphate	P,G	Filter immediately, Cool, ≤6°C	48 hours
46.	Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately
47.	Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48.	Phenols	G only	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
49.	Phosphorus (elemental)	G	Cool, ≤6°C	48 hours
50.	Phosphorus, total	P,G	Cool, ≤6°C, H_2SO_4 to pH<2	28 days
53.	Residue, total	P,G	Cool, ≤6°C	7 days
54.	Residue, Filterable	P,G	Cool, ≤6°C	7 days
55.	Residue, Nonfilterable (TSS)	P,G	Cool, ≤6°C	7 days
56.	Residue, Settleable	P,G	Cool, ≤6°C	48 hours
57.	Residue, Volatile	P,G	Cool, ≤6°C	7 days
61.	Silica	P, or Quartz	Cool, ≤6°C	28 days
64.	Specific conductance	P,G	Cool, ≤6°C	28 days
65.	Sulfate	P,G	Cool, ≤6°C	28 days
66.	Sulfide	P,G	Cool, ≤6°C, add zinc acetate plus NaOH to pH >9	7 days
67.	Sulfite	P,G	None required	Analyze immediately

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

Parameter No./Name		Container[1]	Preservation[2,3]	Maximum holding time[4]
68.	Surfactants	P,G	Cool, ≤6°C	48 hours
69.	Temperature	P,G	None required	Analyze immediately
73.	Turbidity	P,G	Cool, ≤6°C	48 hours
TABLE B − Metals[7]:				
10.	Boron	P, or Quartz	HNO_3 to pH<2	6 months
18.	Chromium VI[7]	P,G	Cool, ≤6°C	24 hours
35.	Mercury[17b]	P,G	HNO_3 to pH<2	28 days
35m.	Mercury (II) & Organomercurials	Amber G	Cool, ≤6°C	7 days
71.	Tin	P	HCl or HNO_3 to pH<2	6 months
3, 5−8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32−34, 36, 37, 45, 47, 51, 52, 58−60, 62, 63, 70−72, 74, 75.	Metals: (except Cr VI, Sn, Hg[7], & B)	P,G	HNO_3 to pH<2	6 months
TABLE C − Organic Tests[8]:				
IA.	Purgeable halocarbons	G, Teflon−lined septum	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5]	14 days
IB.	Purgeable aromatics	G, Teflon−lined septum	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5], HCl to pH<2	14 days
IC.	Acrolein and acrylonitrile	G, Teflon−lined septum	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5] Adjust pH to 4−5[10]	14 days
II.	Phenols[11]	G, Teflon−lined cap	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
IX.	Benzidines (Benzidine and 3,3−Dichlorobenzidine)[11]	G, Teflon−lined cap	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5]	7 days after extraction[13]
III.	Phthlate esters[11]	G, Teflon−lined cap	Cool, ≤6°C	7 days until extraction; 40 days after extraction
IV.	Nitrosamines[11, 14]	G, Teflon−lined cap	Cool, ≤6°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
V.	PCBs[11]	G, Teflon−lined cap	Cool, ≤6°C	7 days until extraction; 40 days after extraction
VI.	Nitroaromatics, cyclic ketones and isophorone[11]	G, Teflon−lined cap	Cool, ≤6°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
VII.	Polynuclear aromatic hydrocarbons[11]	G, Teflon−lined cap	Cool, ≤6°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
VIII.	Haloethers[11]	G, Teflon−lined cap	Cool, ≤6°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

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

Parameter No./Name		Container[1]	Preservation[2,3]	Maximum holding time[4]
IX.	Chlorinated hydrocarbons[11]	G, Teflon−lined cap	Cool, ≤6°C	7 days until extraction; 40 days after extraction
X.	Chorinated Dioxans and Furans	G, Teflon−lined cap	Cool, ≤6°C, pH<9, 0.008% $Na_2S_2O_3$[5]	1 year
TABLE E − Pesticide Tests:				
1−70.	Pesticides[11]	G, Teflon−lined cap	Cool, ≤6°C, pH 5−9[15]	7 days until extraction; 40 days after extraction
TABLE F − Radiological Tests:				
1−5.	Alpha, beta, and radium	P,G	HNO_3 to pH<2	6 months

[1]Polyethylene (P), or glass (G). For bacteria, plastic sample containers must be made of sterilizable materials (polypropylene [PP] or other autoclavable plastic). For protozoa, plastic sample containers must be made of low−density polyethylene (LDPE). For samples collected for trace−level mercury see note 17b.

[2]All samples requiring preservation at ≤6°C must be cooled immediately after collection, but not frozen. 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. If sampling teams use "blue ice" packs, it is necessary to pre−chill all sample containers to at least 6 degrees celsius with ice or refrigeration prior to shipping. 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. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤6°C until compositing and sample splitting are completed. For low−level mercury see note 17b.

[3]When any sample is to be shipped by common carrier or sent through the United States mail, 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 F, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4]Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. For other composite samples, the holding time commences immediately after the samples are removed from the composite sampler. The time the sample spends in the sampler during collection does not count towards the maximum holding time. Samples for biological or chemical analysis may be held for longer periods than specified in this table only if the permittee or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator (s. NR 219.05). Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See sec. 40 CFR 136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

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

[6]Immediately after collection, preserve the sample using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to 12 by addition of sodium hydroxide and refrigeration as specified:

(1) Sulfide: The maximum holding time for an untreated sample is 24 hours when sulfide is present. Optionally, the sample may be treated and the maximum holding time extended to 14 days. Generally, the laboratory should test the sample with lead acetate test paper to determine the presence or absence of sulfide ion. However, for cyanide methods using amperometric detection systems (*e.g.*, OIA–1677 for available cyanide), sulfide levels below those detectable with lead acetate paper (approximately 5 ppm) may produce a false positive signal for cyanide. If there is reason to suspect sulfide levels below the detectable level of lead acetate paper when using an amperometric method, test the sample using a more sensitive sulfide method to determine if the treatment (described below) is required. If sulfide ion is present, treat the sample immediately (within 15 minutes of collection) with sufficient solid lead carbonate to remove sulfide (as evidenced by a lead acetate test paper), and immediately filter into another sample bottle to remove precipitated lead sulfide. If sulfide ion is suspected to be present, but its presence is not detected by the lead acetate paper test, two samples must be collected. One is treated for the presence of sulfide and immediately filtered, while the second is not treated for sulfide. Analyze both samples and report the lower of the two results.

(2) Sulfide and particulate matter: If the sample contains sulfide and particulate matter that would be removed by filtration, filter the sample prior to treatment with lead carbonate to assure that cyanides associated with the particulate matter are included in the measurement. Save the particulate matter and treat the filtrate using the sulfide removal procedure above. Combine and homogenize the collected particulate and treated filtrate prior to shipment to the laboratory for analysis.

[7]Samples should be filtered immediately on−site before adding preservative for dissolved metals, except for samples collected for trace−level mercury (see note 17b).

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

[9]Samples receiving no pH adjustment must be analyzed within 7 days of sampling.

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

[11]When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within 2 or more chemical categories, the sample may be preserved by cooling to $\leq 6°C$, 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 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

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

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

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

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

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

[17a]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.

[17b]Samples collected for the determination of trace level mercury (100 ng/L) using EPA Method 1631 must be collected in tightly−capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

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 149.12 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.

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.