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Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

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

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

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

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

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

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

(2) Section NR 219.06 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149, HFS 157 or HSS 165.

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.**

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.

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.

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 revisor of statutes. 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**.

Table A
List of Approved Biological Test Procedures For Wastewater

Parameter and Units	Method[1]	EPA	Standard Methods 18th Ed.	USGS	WDNR
Bacteria:					
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF)[2], single step.	p132[3] p124[3]	9221E 9222D	B–0050–85[4]	
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or MF, single step[5]	p132[3] p124[3]	9221E 9222D		

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Parameter and Units	Method[1]	EPA	Standard Methods 18th Ed.	USGS	WDNR
Bacteria:					
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF[2] single step or two step	p114[3] p108[3]	9221B 9222B	B−0025−85[4]	
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF[2] with enrichment.	p114[3] p111[3]	9221B 9222B+B.5c		
5. Fecal strepto−cocci, number per 100 ml	MPN, 5 tube, 3 dilution; MF[2], or Plate count	p136[3] p136[3] p143[3]	9230B 9230C	B−0055−85[4]	
Enteroviruses:					
6. Enteroviruses in water, plaque forming units per liter.	Absorption, elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 6[6] Ch. 9[6] Ch. 10[6] Ch. 12[6]	9510B,C,D,E 9510G 9510G 9510G		
7. Enteroviruses in sludge, plaque forming units per liter.	Beef extract elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 7[6] Ch. 9[6] Ch. 10[6] Ch. 12[6]	9510F 9510G 9510G 9510G		
Mutagenicity:					
8. Mutagenicity (revertants per liter)	Ames test, test strains TA97, TA98, TA100, and TA102.	Note 7			
Acute and Chronic Toxicity:					
9. Toxicity, acute, fresh water organisms, percent effluent[10]	Ceriodaphnia, 48−h static−renewal mortality. Fathead minnow, 96−h static−renewal mortality, or 96−h flow−through mortality.				8 8
10. Toxicity, chronic, fresh water organisms, percent effluent.[10]	Fathead minnow larval survival and growth. Ceriodaphnia survival and reproduction.				8 8

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

[2] A 0.45 µm membrane filter (MF) or other port 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] Bordner, R.H., and J.A. Winter, eds. "Microbiological Methods for Monitoring the Environment, Water and Wastes", United States Environmental Protection Agency, EPA−600/8−78−017, 1978. Available from ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. Martin Luther King Drive, Cincinnati, Ohio 45268.

[4] Britton, L.J., and P.E. Greeson, eds. "1988 Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples", edited by et al., U.S. Geological Survey, Techniques of Water−Resources Investigation (USGS TWRI), Book 5 chapter A4, Laboratory analysis, 1977. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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

[6] Berg, G., R.S. Safferman, D.R. Dahling, D. Berman, and C.J. Hurst, 1984. USEPA Manual of Methods for Virology. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4−84/013. (Chapter 9 revised January 1987; Chapter 10 revised December 1987; Chapter 12 revised May 1988; Chapter 7 revised September 1989).

[7] Williams, L.R., and J.E. Preston, eds. 1983. Interim Procedures for Conducting the Salmonella/Microsomal Mutagenicity Assay (Ames Test). Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Las Vegas, Nevada. EPA/600/4−82/068.

[8] Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 1st Edition," Wisconsin Department of Natural Resources, 1996. This publication is available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and the Revisor of Statutes. Copies are available from the Department of Natural Resource, Bureau of Integrated Science Services, P.O. Box 7921, Madison,WI 53707.

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
1. Acidity, as $CaCO_3$, mg/L, Electrometric end point or phenolphthalein end point	305.1		2310 B(4a)	D1067–92		
2. Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric: Titration to pH 4.5, manual Or automated	310.1 310.2		2320 B	D1067–92	I–1030–85	973.43[5]
3. Aluminum, mg/L: Digestion[6] followed by:						
AA direct aspiration[6m],	202.1	7020	3111 D		I–305I–85	
AA furnace,	202.2 or 200.9[1g]		3113 B			
Inductively coupled plasma (ICP)[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry (ICP–MS),	200.8[1g]	6020				
Direct current plasma (DCP)[6m], or				D4190–82(88)		Note 36
Colorimetric (Eriochrome cyanine R)			3500–Al D			
4. Ammonia (as N), mg/L: Manual distillation[8] (at pH 9.5): Followed by	350.2		4500–NH_3 B			973.49[5]
Nesslerization,	350.2		4500–NH_3 C	D1426–89(A)	I–3520–85	973.46[5]
Titration,	350.2		4500–NH_3 E			
Electrode,	350.3		4500–NH_3F&G	D1426–89(B)		
Automated phenate, or	350.1[1m]		4500–NH_3 H		I–4523.85	
Automated electrode						Note 9
5. Antimony, ug/L: Digestion[6] followed by:						
AA direct aspiration[6m],	204.1	7040	3111 B			
AA furnace,	200.9[1g]	7041	3113 B			
AA (gaseous borohydride),		7062				
Inductively coupled plasma[6m], or	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
6. Arsenic, ug/L: Digestion[6] followed by	206.5					
AA (gaseous hydride),		7061A	3114 B[37]	D2972–88(B)	I–3062.85	
AA (gaseous borohydride),		7062				
AA furnace,	206.2 or 200.9[1g]	7060A	3113 B	D2972–88(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Or, colorimetric (SDDC)			3500–As C	D2972–88(A)	I–3060–85	
7. Barium, mg/L: Digestion[6] followed by:						
AA direct aspiration[6m],	208.1	7080A	3111 D		I–3084–85	
AA furnace,	208.2	7081	3113 B	D4382–91		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry, or	200.8[1g]	6020				
Direct current plasma[6m]						Note 36

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Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
8. Beryllium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	210.1	7090	3111 D	D3654–(88)(A)	I–3095–85	
AA furnace,	210.2 or 200.9[1g]	7091	3113 B	D3645(88)(B)		
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
Direct current plasma, or				D4190–82(88)		Note 36
Colorimetric (aluminon)			3500–Be D			
9. Biochemical oxygen demand (BOD_5), mg/L:						
Dissolved Oxygen Depletion			5210 B		I–1578–78[10]	973.443[5]
10. Boron, mg/L:						
Colorimetric (curcumin),	212.3		4500–B B		I–3112–85	
Inductively coupled plasma, or	200.7[1g]	6010A	3120 B			
Direct current plasma				D4190–82(88)		Note 36
11. Bromide, mg/L: Titrimetric	320.1			D1246–82(88)(C)	I–1125–85	p.S44[12]
Ion Chromatography	300.0[1m]	9056				
12. Cadmium–Total[6], mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	213.1	7130	3111 B or C	D3557–90 (A or B)	I–3135–85 or I–3136–85	974.27[5]
AA furnace,	213.2 or 200.9[1g]	7131A	3113 B	D3557–90(D)		
Inductively coupled plasma[6m]	200.7[1g]	6010A	3120 B		I–1472–85	
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
Direct current plasma[6m],				D4190–82(88)		Note 36
Voltametry[13], or				D3557–90(C)		
Colorimetric (Dithizone)			3500–Cd D			
13. Calcium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	215.1	7140	3111 B	D511–92(B)	I–3152–85	
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Direct current plasma, or						Note 36
EDTA titration	215.2		3500–Ca D	D511–92(A)		
14. Carbonaceous Biochemical oxygen demand ($CBOD_5$), mg/L: with nitrification inhibitor[14]			5210 B			
15. Chemical oxygen demand (COD), mg/L:						
Closed reflux			5220 C or D			Notes 15&16
Titrimetric						973.46[5]
	410.1		5220 B	D1252–88(A)	I–3560 or	
	410.2				I–3562–85	
	410.3					
Automated and manual	410.4[1m]				I–3561–85	
Spectrophotometric				D1252–88(B)		

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Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
16. Chloride, mg/L:						
Titrimetric (silver nitrate) or		9253	4500–Cl– B	D512–89(B)	I–1183–85	
(Mercuric nitrate),	325.3	9252A	4500–Cl– C	D512–89(A)	I–1184–85	973.51[5]
Colorimetric (ferricyanide),					I–1187–85	
manual or automated, or	325.1 or 325.2	9250	4500–Cl– E		I–2187–85	
Ion chromatography	300.0[1m]	9056				
17. Chlorine – Total residual, mg/L:						
amperometric,	330.1		4500–Cl D	D1253–86(92)		
Starch End point direct	330.3		4500–Cl B			
Back Titration either end point[17], or	330.2		4500–Cl C			
DPD–FAS,	330.4		4500–Cl F			
Spectrophotometric, DPD; or	330.5		4500–Cl G			
Electrode			4500–Cl I			Note 18
18. Chromium VI dissolved, ug/L:						
0.45 micron filtration with:						
Extraction and atomic absorption,	218.4	7197	3111 A		I–1232–85	
Coprecipitation and atomic absorption,		7195				
Differential pulse polarography,						
Colorimetric		7198				
(Diphenylcarbazide), or		7196A	3500–Cr D	D1687–92(A)	I–1230–85	307B[19]
Ion Chromatography	218.6[1g]					
19. Chromium, mg/L:						
Digestion[6] (optional extraction) followed by:						
AA direct aspiration[6m],	218.1	7190	3111 B	D1687–92(B)	I–3236–85	974.24[5]
AA chelation extraction,	218.3		3111 C			
AA furnace,	218.2 or 200.9[1g]	7191	3113B	D1687–92(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Direct current plasma[6m], or				D4190–82(88)		Note 36
Colorimetric (diphenylcarbazide),			3500–Cr D			
20. Cobalt, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	219.1	7200	3111 B (A or B)	D3558–90(AorB)	I–3239–84	
AA furnace, or	219.2 or 200.9[1g]	7201	3113 B	D3558–90(C)		
Inductively coupled plasma, or	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
Direct current plasma				D4190–82(88)		Note 36
21. Color, Platinum Cobalt units or dominant wavelength hue, luminance, purity:						
Colorimetric, ADMI	110.1		2120 E			Note 20
Platinum cobalt; or	110.2		2120 B		I–1250–85	
Spectrophotometric	110.3		2120 C			

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Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
22. Copper, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	220.1	7201	3111 B or C	D1688–90(AorB)	I–3271–85 or I–3270–85	974.27[5]
AA furnace,	220.2 or 200.9[1g]	7211	3113 B	D1688–90(C)		
Inductively coupled plasma[6m]	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
Direct current plasma[6m],				D4190–82(88)		Note 36
Colorimetric (Neocuproine), or			3500–Cu D or E			
Bicinchoninate						Note 21
23. Cyanide – Total, ug/L:						
Manual distillation with $MgCl_2$			4500–CN–C			
Followed by: titrimetric,			4500–CN–D			
Manual or	335.2	9010A	4500–CN–E	D2036–91(A)	I–3300–85	
Automated[22]	335.3	9010A				
spectrophotometric, or						
Semi–automated colorimetry	335.4[1m]	9012				
24. Cyanide amenable to chlorination, ug/L: Manual distillation with MgC_{12} followed by titrimetric, manual or automated spectrophotometric	335.1	9010A	4500–CN–G	D2036–91(B)		
25. Fluoride – Total, mg/L:						
Manual distillation[8]			4500–F–B			
Followed by manual or automated electrode,	340.2		4500–F–C	D1179–88(B)	I–4327–85	
SPADNS,	340.1		4500–F–D	D1179–88(A)		
Ion chromatography,	300.0[1m]	9056				
Or automated complexone	340.3		4500–F–E			
26. Gold, mg/L:						
Digestion[6] followed by:						
AA direct aspiration	231.1		3111 B			
AA furnace,	231.2		3113 B			
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
27. Hardness – Total as $CaCO_3$, mg/L:						
Automated colorimetric,	130.1					
EDTA titration,	130.2		2340 C	D1126–86(92)	I–1338–85	973.52B[5]
or the sum of Ca and Mg as their respective carbonates (by ICP or AA direct aspiration) (See Parameters 13 and 33)			2340 B			
28. Hydrogen ion (pH), pH units:						
Electrometric Measurements	150.1	9040B	4500–H^+ B	D1293–84(90) (A or B)	I–1586–85	973.41[5]
or						
Automated Electrode						Note 23
29. Iridium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	235.1		3111 B			
AA furnace, or	235.2					
Inductively coupled plasma	200.7[1g]	6010A				

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Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
30. Iron, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	236.1	7380	3111 B or C	D1068–90(AorB)	I–3381–84	973.275
AA furnace,	236.2 or 200.9[1g]	7381	3113 B	D1068–90(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Direct current plasma[6m], or				D4190–82(88)		Note 36
Colorimetric (Phenanthroline)			3500–Fe D	D1068–90(D)		Note 24
31. Kjeldahl nitrogen – Total (as N), mg/L:						
Digestion and distillation	351.3		4500–NorgBorC	D3590–89(A)		
Followed by titration	351.3		4500–NH_3 E	D3590–89(A)		937.46[5]
Nesslerization or	351.3		4500–NH_3 C	D3590–89(A)		
Electrode,	351.3		4500–NH_3 ForG			
Automated phenate,	351.1		4500–NH_3 H		I–4551–78[8]	
Semi–automated block digester,	351.2[1m]			D3590–89(B)		
Or potentiometric	351.4			D3590–89(A)		
32. Lead, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	239.1	7420	3111 B or C	D3559–90(AorB)	I–3399–90	974.27[5]
AA furnace,	239.2 or 200.9[1g]	7421	3113 B	D3559–90(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
Direct current plasma[6m],				D4190–82(88)		Note 36
Voltametry[13] or				D3559–90(C)		
Colorimetric (Dithizone)			3500–Pb D			
33. Magnesium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	242.1	7450	3111 B	D511–92(B)	I–3447–85	974.27[5]
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Direct current plasma, or						Note 36
Gravimetric			3500–Mg D			
34. Manganese, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	243.1	7460	3111 B	D858–90(AorB)	I–3454–85	974.27[5]
AA furnace,	243.2 or 200.9[1g]	7461	3113 B	D858–90(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Direct current plasma[6m],				D4190–82(88)		Note 36
Colorimetric (Persulfate), or			3500–Mn D			920.205[3]
Periodate						Note 25
35. Mercury – Total[6], ug/L:						
Cold vapor AA, manual or automated, or	245.1[1g] 245.2	7470A	3112 B	D3223–91	I–3462–85	977.22[5]
35f. Mercury, Total – Low Level ng/L[40]:						
Cold vapor atomic fluorescence (CVAF) with purge and trap concentration	1631D					
CVAF without purge and trap concentration	245.7					

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Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
35m. Mercury – Hg(II) and organomercurials, ug/L:						
HPLC with electrochemical detection	245.3[1g]					
36. Molybdenum, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	246.1	7480	3111 D		I–3490–85	
AA furnace,	246.2	7481	3113 B			
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry, or	200.8[1g]	6020				
Direct current plasma						Note 36
37. Nickel, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	249.1	7520	3111 B or C	D1886–90(AorB)	I–3499–85	
AA furnace,	249.2 or 200.9[1g]		3113 B	D1886–90(C)		
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Direct current plasma[6m], or				D4190–82(88)		Note 36
Colorimetric (Heptoxime)			3500–Ni D			
38. Nitrate (as N), mg/L:						
Brucine sulfate, or	352.1					973.50[5],419D[19]
Nitrate–nitrite N minus Nitrite N (see parameters 39 and 40)						
Ion chromatography	300.0[1m]	9056				
39. Nitrate–nitrite (as N), mg/L:						
Cadmium reduction, manual	353.3		4500–NO_3 E	D3867–90(B)		
or automated, or	353.2[1m]		4500–NO_3 F	D3867–90(A)	I–4545–85	
automated hydrazine	353.1		4500–NO_3 H			
Ion chromatography	300.0[1m]	9056				
40. Nitrite (as N), mg/L:						
Spectrophotometric, manual or	354.1		4500–NO_2 B			Note 27
automated (Diazotization), or					I–4540–85	
Ion chromatography[39]	300.0[1m]	9056				
41. Oil and grease–Total recoverable, mg/L:						
Gravimetric (freon extraction)	413.1	9070	5520 B			
Gravimetric (hexane extraction)	1664					
42. Organic carbon – Total (TOC), mg/L:						
Combustion or oxidation,	415.1	9060	5310 B or D	D2579–85(AorB)		973.47[5] p.142[6]
Persulfate oxidation	415.21[m]		5310C			
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		I–4601–85	973.56[5]
Or manual single reagent or	365.2		4500–P E	D515–88(A)		973.55[5]
Manual two reagent, or	365.3					
Ion chromatography	300.0[1m]	9056				

Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
45. Osmium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	252.1	7550	3111 D			
AA furnace, or	252.2					
Inductively coupled plasma	200.7[1g]	6010A				
46. Oxygen, dissolved, mg/L:						
Winkler (Azide modification)	360.2		4500–O C	D888–92(A)	I–1575–7810	973.45B[5]
Or electrode	360.1		4500–O G	D888–92(B)	I–1576–7810	
47. Palladium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	253.1		3111 B			
AA furnace,	253.2					
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
48. Phenols, ug/L:						
Manual distillation[28]	420.1		5530 B			Note 29
Followed by manual	420.1	9065	5530 D			
Or automated[22] colorimetric (4AAP), or	420.2	9066				Note 29
Semi–automated colorimetric	420.4[1m]					
49. Phosphorus (elemental), mg/L:						
Gas–Liquid chromatography						Note 30
50. Phosphorus – Total, mg/L:						
Persulfate digestion	365.2		4500–P B,5			973.55[5]
Followed by manual or	365.2 or 365.3		4500–P E	D515–88 (A)		
Automated ascorbic acid Reduction, or semi–automated block digestor	365.1[1m] 365.4		4500–P F		I–4600–85	973.56[5]
51. Platinum, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	255.1		3111 B			
AA furnace,	255.2					
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
52. Potassium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	258.1	7610	3111 B		I–3620–85	973.53[5]
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Flame photometric, or			3500–K D			
Colorimetric (cobalt nitrate)						317B[19]
53. Residue – total, (total solids), mg/L:						
Gravimetric 103–105°C	160.3		2540 B		I–3750–85	
54. Residue – filterable, (TDS), mg/L:						
Gravimetric, 180°C	160.1		2540 C		I–1750–85	
55. Residue – nonfilterable, (TSS), mg/L: Gravimetric, 103–105°C post washing of residue	160.2		2540 D		I–3765–85	
56. Residue – settleable, mg/L:						
Volumetric (Imhoff cone) or gravimetric	160.5		2540 F			

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
57. Residue – volatile mg/L:						
Gravimetric, 550°C	160.4		2540 E[38]		I–3753–85	
58. Rhodium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	265.1		3111 B			
AA furnace, or	265.2					
Inductively coupled plasma	200.7[1g]	6010A				
59. Ruthenium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	267.1		3111 B			
AA furnace, or	267.2					
Inductively coupled plasma	200.7[1g]	6010A				
60. Selenium, ug/L:						
Digestion[6] followed by:						
AA furnace,	270.2 or 200.9[1g]	7740	3113 B			
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
or AA (gaseous hydride)		7741A	3114 B[37]	D3859–88(A)	I–3667–85	
61. Silica – Dissolved, mg/L:						
0.45 micron filtration:						
Followed by manual or	370.1		4500–Si D	D859–88	I–1700–85	
automated colorimetric					I–2700–85	
(Molybdosilicate), or						
Inductively coupled plasma[6]	200.7[1g]	6010A	3120 B			
62. Silver[31], mg/L:						
Digestion[6] followed by:						
AA direct aspiration,		7760A	3111 B or C		I–3720–85	973.27[5]
AA furnace,	200.9[1g]	7761	3113 B			
Colorimetric (Dithizone),						319B[19]
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Or direct current plasma						Note 36
63. Sodium, mg/L:						
Digestion[6] followed by:						
Atomic absorption,	273.1	7770	3111 B		I–3735–85	973.54[5]
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Direct current plasma, or						Note 36
Flame photometric			3500–Na D	D1428–82(A)		
64. Specific conductance, micromhos/cm						
at 25°C: Wheatstone bridge	120.1	9050	2510 B	D1125–91(A)	I–1780–85	973.40[5]
65. Sulfate (as SO_4), mg/L:						
Automated colorimetric (barium chloroanilate),	375.1	9035				
Semi–automated colorimetric (methylthymol blue)	375.2[1m]	9036				
Gravimetric,	375.3		4500–SO_4^2CorD			925.54[5]
Turbidimetric, or	375.4	9038		D516–90		426C[32]
Ion chromatography	300.0[1m]	9056				
66. Sulfide (as S), mg/L:						
Titrimetric (iodine) or	376.1		4500–S^{2-}E		I–3840–85	228A[33]
Colorimetric (methylene blue)	376.2		4500–S^{2-}D			
67. Sulfite (as SO_3), mg/L:						
Titrimetric (iodine–iodate)	377.1		4500–$S0_3^{2-}$			

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Table B
List of Approved Inorganic Test Procedures for Wastewater

Parameter, Units & Methods	EPA[1]	SW–846[11,7]	Standard Methods[2,2m]	ASTM[3]	USGS[4]	Other
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1		5540 C	D2330–88		
69. Temperature, °C: Thermometric	170.1		2550 B			Note 34
70. Thallium, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	279.1	7840	3111 B			
AA furnace,	279.2 or 200.9[1g]	7841	3113 B			
Inductively coupled plasma, or	200.7[1g]	6010A				
Inductively coupled plasma–mass spectrometry	200.8[1g]	6020				
71. Tin, ug/L:						
Digestion[6] followed by:						
AA direct aspiration,	282.1	7870	3111 B		I–3850–7810	
AA furnace, or	282.2 or 200.9[1g]		3113 B			
Inductively coupled plasma	200.7[1g]	6010A				
72. Titanium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	283.1		3111 D			
AA furnace,	283.2		3113 B			
Direct current plasma, or						Note 36
Inductively coupled plasma	200.7[1g]	6010A				
73. Turbidity, NTU: Nephelometric	180.1[1m]		2130 B	D1889–88(A)	I–3860–85	
74. Vanadium, mg/L:						
Digestion[6] followed by:						
AA direct aspiration,	286.1	7910	3111 D			
AA furnace,	286.2	7911	3113 B			
Inductively coupled plasma,	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry	200.8[1g]					
Direct current plasma, or				D4190–82(88)		Note 36
Colorimetric (Gallic acid)			3500–V D			
75. Zinc, mg/L:						
Digestion[6] followed by:						
AA direct aspiration[6m],	289.1	7950	3111 B or C		I–3900–85	974.27[5]
AA furnace,	289.2 or 200.9[1g]	7951	3113 B			
Inductively coupled plasma[6m],	200.7[1g]	6010A	3120 B			
Inductively coupled plasma–mass spectrometry,	200.8[1g]	6020				
Direct current plasma[6m],				D4190–82(88)		Note 36
Colorimetric (Dithizone), or			3500–Zn E			
Colorimetric (Zincon)			3500–Zn F			Note 36

[1] "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.

[1g] "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.

[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] "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, 18th Edition, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

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[2m] The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[3] "1993 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, U.S. Geological Survey, Open−File Report 85−495, 1989, unless otherwise stated. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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

[6] 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, than 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.

[6m] Either of the following microwave digestion procedures may be used:
"Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM corporation, P.O. Box 200, Mattews, North Carolina 28106−0200, April 16, 1992. Available form the CEM Corporation.
"Test Methods for Evaluating Solid Waste", SW−846 method 3015. United States EPA SW−846, 3rd Edition. Footnote 11 lists the complete reference.

[7] SW−846 series 6000 and 7000 methods include SW−846 method 7000A, the general AA method description.

[8] 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.

[9] Ammonia, Automated Electrode Method, Industrial Method Number 379−75WE, dated February 19, 1976, Technicon AutoAnalyzerII. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[10] 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.

[11] "Test Methods for Evaluating Solid Waste", 3rd Edition, SW−846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC, (202) 512−1800.

[12] "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," from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1981. Available on inter−library loan.

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

[14] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD_5." 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 obtained using the nitrification inhibitor.

[15] OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West loop, P.O. Box 2980, College Station, TX 77840.

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

[17] The back titration method will be used.

[18] ORION Research Instruction Manual, Residual Chlorine Electrode Model 97−70, 1977. Available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

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

[20] "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.

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

[22] After the manual distillation is completed, the auto−analyzer manifolds in EPA Methods 335.03 (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.

[23] Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378−75WA, October 1976, Technicon AutoAnalyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[24] 1, 10−Phenanthroline Method for Iron, Hach Method 8008, 1980. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

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

[26] "Methods for Analysis of Organic Substances in Water", by D. F. Goerlitz and Eugene Brown: USGS−TWRI, Book 5, Chapter A3, p. 4, 1972. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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

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

[29] The approved method is that 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 spectrophotometric procedure. Available on inter−library loan.

[30] "Direct Determination of Elemental Phosphorus by Gas−Liquid Chromatography", by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421−426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North−Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

[31] 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 a 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 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

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

[33] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 13th Edition. Available on inter−library loan.

[34] "Water Temperature−Influential Factors, Field Measurement, and Data Presentation", by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS−TWRI Book 1, Chapter D1, 1975. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

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

[36] Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029, "1986 Revised 1991, Fison Instruments, Inc., 32 32 Commerce Center, Cherry Hill Drive, Danvers MA 01923.

[37] Use the digestion given in the method.

[38] The temperature must be maintained between 500−550° C, and not the temperature listed in the method.

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

[40] Quality control requirements for low level mercury are found in s. NR 106.145 (9) and (10). 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 the atomic fluorescence detector, the appropriate method citation is 245.1 (manual) or 245.2 (automated). If method 1631 is modified to eliminate the purge and trap step, the appropriate method citation is 245.7.

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], 3050A[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]	3015[13]		

[1] "Test Methods for Evaluating Solid Waste", 3rd Edition, SW−846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC 20402, (202) 512−1800.

[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.

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[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	EPA Method Number[1,6] GC	EPA Method Number[1,6] GC/MS	Standard Methods[8,13]	SW−846 Method Number[11,12] GC capillary	SW−846 Method Number[11,12] GC pkd[14]	SW−846 Method Number[11,12] GC/MS capillary	SW−846 Method Number[11,12] GC/MS pkd[14]	Other
I. Volatiles		624[3]		8021A	———	8260A	8240B	
A. Halogenated volatiles	601	1624	6230 B, 6210 B	———	8010B	———	———	
Bromodichloromethane								
Bromoform								
Bromomethane								
Carbon tetrachloride								Note 2, p.130
Chloroethane								
Chloroform								Note 2, p.130
Chloromethane								
Dibromochloromethane								
Dichlorodifluoromethane		———	not 6210 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								Note 2, p.130
1,1,2,2−Tetrachloroethane								Note 2, p.130
Tetrachloroethene								Note 2, p.130
1,1,1−Trichloroethane								
1,1,2−Trichloroethane								Note 2, p.130
Trichloroethene								
Trichlorofluoromethane	———							
Vinyl chloride								
B. Aromatic volatiles	602		6220B	———	8020A	———	———	
Benzene		1624	6210B					
Chlorobenzene	601	1624	6210B, 6230B					Note 2, p.130
1,2−Dichlorobenzene	601, 612	625, 1625	6230B, 6410B					
1,3−Dichlorobenzene	601, 612	625, 1625	6230B, 6410B					
1,4−Dichlorobenzene	601, 612	625, 1625	6230B, 6410B					
Ethylbenzene		1624	6210B					
Toluene		1624	6210B					
C. Other volatiles	603	1624,624[3]		8030A	———	8260A	8240B	
Acrolein								LC:8315 (SW−846)
Acrylonitrile				8031				LC: 8316 (SW−846)

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

	Parameter	EPA Method Number[1,6] GC	EPA Method Number[1,6] GC/MS	Standard Methods[8,13]	SW−846 Method Number[11,12] GC capillary	SW−846 Method Number[11,12] GC pkd[14]	SW−846 Method Number[11,12] GC/MS capillary	SW−846 Method Number[11,12] GC/MS pkd[14]	Other
II.	Phenols	604	625, 1625	6410B, 6420B	———	8040 A	8270B	8250A	
	4−Chloro−3−methylphenol								
	2−Chlorophenol								
	2,4−Dichlorophenol								
	2,4−Dimethlyphenol								
	2,4−Dinitrophenol								
	2−Methyl−4,6−dinitrophenol								
	2−Nitrophenol								
	4−Nitrophenol								
	Pentachlorophenol								Note 2, p.140
	Phenol								
	2,4,6−Trichlorophenol								
III.	Phthalate esters	606	625, 1625	6410 B	8061	8060	8270B	8250A	
	Benzyl butyl phthalate								
	Bis(2−ethylhexyl)phthalate								
	Diethyl phthalate								
	Dimethyl phthalate								
	Di−n−butyl phthalate								
	Di−n−octyl phthalate								
IV.	Nitrosamines	607	625, 1625	6410 B	———	8070	8270B	8250A	
	N−Nitrosodimethylamine		note 4						
	N−Nitrosodi−n−propylamine								
	N−Nitrosodiphenylamine		note 4						
V.	Polychlorinated biphenyls	608	625	6410 B	8081	8080 A	8270B	8250A	Note 2, p.43
	PCB−1016								
	PCB−1221								
	PCB−1232								
	PCB−1242								
	PCB−1248								———
	PCB−1254								
	PCB−1260								
VI.	Nitroaromatics & cyclic ketones	609	625, 1625	6410 B	———	8090	8270B	8250A	
	2,4−Dinitrotoluene								
	2,6−Dinitrotoluene								
	Isophorone								
	Nitrobenzene								
VII.	Polynuclear aromatic hydrocarbons	610/FID	625, 1625	6410 B, 6440 B	———	8100	8270B	8250A	Note 9; 610, LC: 8310 (SW−846)
	Acenaphthene								
	Acenaphthylene								
	Anthracene								
	Benzo(a)anthracene								
	Benzo(a)pyrene								
	Benzo(b)fluoranthene								
	Benzo(g,h,i)perylene								
	Benzo(k)fluoranthene								

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

	Parameter	EPA Method Number[1,6] GC	EPA Method Number[1,6] GC/MS	Standard Methods[8,13]	SW−846 Method Number[11,12] GC capillary	GC pkd[14]	GC/MS capillary	GC/MS pkd[14]	Other
	Chrysene								
	Dibenzo(a,h)anthracene								
	Fluoranthene								
	Fluorene								
	Ideno (1,2−3−cd)pyrene								
	Naphthalene				8021A				
	Phenanthrene								
	Pyrene								
VIII.	Haloethers	611	625, 1625	6410 B	———	8110	8270B	8250A	
	Bis(2−chloroethoxy) methane								
	Bis(2−chloroethyl)ether								
	4−Bromophenylphenyl ether								
	4−Chlorophenylphenyl ether								
	2,2−Oxybis (1−chloropropane)								
IX.	Chlorinated hydrocarbons	612	625, 1625	6410 B	8121	8120A	8270B 8260A	8250A, 8240A	
	Benzyl chloride	———	———	———		8010B	not 8270B	not 8250A	Note 2, p.130; Note 5, p.S102
	2−Chloronaphthalene						not 8260A	not 8240A	8410 (SW−846)
	Epichlorohydrin	———	———	———		8010B	not 8270B	not 8250A	Note 2, p.130; Note 5, p.S102
	Hexachlorobenzene				8081		not 8260A	not 8240A	8410 (SW−846)
	Hexachlorobutadiene				8021A			not 8240A	8410 (SW−846)
	Hexachlorocyclopentadiene		note 4		8081		not 8260A	not 8240A	8410 (SW−846)
	1,2,4−Trichlorobenzene				8021A			not 8240A	Note 2, p.130
	Hexachloroethane							not 8240A	8410 (SW−846)
	Benzidine		note 4		———	———	not 8260A	not 8240A	LC: 605
	3,3−Dichlorobenzidine			———	———		not 8260A	not 8240A	
X.	Polychlorinated dibenzo−p−dioxins and furans		1613 A[7]		———	———	8280, 8290	———	
	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								

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

Parameter	EPA Method Number[1,6] GC	EPA Method Number[1,6] GC/MS	Standard Methods[8,13]	SW−846 Method Number[11,12] GC capillary	GC pkd[14]	GC/MS capillary	GC/MS pkd[14]	Other
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]						Note 10
2,3,7,8−Tetrachlorodibenzofuran								

[1] "The full text of Methods 601−613, 624, 625, 1624, and 1625, are given in Appendix A of 40 CFR part 136," Test Procedures for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit." Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

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

[3] 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 1624.

[4] 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 1625, are preferred methods for these compounds.

[5] "Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of "Standard Methods for the Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.

[5m] 625 Sreening only.

[6] Each analyst must make an initial, one−time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601−613, 624, 625, 1613A, 1624, and 1625 in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on−going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

[7] Method 1613 Revision A: Tetra− through Octa− Chlorinated Dioxins and Furans by Isotope Dilution, HRGC/HRMS, Environmental Protection Agency, Federal Register, page 5098, February 1991. Available from the Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.

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

[9] 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.

[10] Method D4675−92, "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.

[11] "Test Methods for Evaluating Solid Waste", 3rd Edition. SW−846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512−1800.

[12] SW−846 methods 8021, 8061, 8081, and 8121 require one of the following sample preparation (extraction/clean−up) procedures: 3500/3510 (liquid−liquid extraction), 3500/3520 (continuous liquid−liquid extraction), or 5030 (purge and trap method). The required sample preparation procedure is given in the determinative procedure. Method 8021 requires 5030 (purge and trap). Methods 8081 and 8121 require either 3500/3510 or 3500/3520 in addition to 3600. Method 8061 requires 3510. For methods 8021, 8061, 8081, and 8121 see also SW−846 method 8000A.

[13] The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[14] In order to reference these methods, the laboratory must use a packed column for the GC separations.

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

	Parameter	Method	EPA[2,7]	SW−846[A,8] pkd[11]	SW−846[A,8] cap.	Standard Methods[R,9]	ASTM[c]	Other
1.	Aldrin	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630B&C 6410B	D3086−90	Note 3, p. 7; Note 4, p. 30
2.	Ametryn	GC						Note 3, p. 83; Note 6, p. 868
3.	Aminocarb	HPLC						Note 10
4.	Atraton	GC						Note 3, p.83; Note 6, p.S68
5.	Atrazine	GC		8140	8141A			Note 3. p.83; Note 6, p.S68

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	Parameter	Method	EPA[2,7]	SW–846[A,8] pkd[11]	SW–846[A,8] cap.	Standard Methods[R,9]	ASTM[c]	Other
6.	Azinphos methyl	GC GC/MS		8140 8250A	8141A 8270B			Note 3. p.25; Note 6, p.S51
7.	Barban	HPLC GC/MS		 8250A	 8270B			Note 10
8.	α–BHC	GC GC/MS	608 625[5]	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7
9.	β–BHC	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 C 6410 B	D3086–90	
10.	δ–BHC	GC GC/MS	608 625[5]	8080A 8250A	8081 8270B	6630C 6410B	D3086–90	
11.	γ–BHC(Lindane)	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630B & C 6410B	D3086–90	Note 3, p. 7; Note 4, p. 30
12.	Captan	GC GC/MS		 8250A	 8270B	6630B	D3086–90	Note 3, p. 7.
13.	Carbaryl	HPLC GC/MS		 8250A	 8270B			Note 10
14.	Carbophenothion	GC GC/MS		8140 8250A	8141A 8270B			Note 4, p.30; Note 6, p.S73
15.	Chlordane	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7
16.	Chloropropham	HPLC						Note 10
17.	2,4–D	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35
18.	4,4′–DDD	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3. p.7; Note 4, p.30
19.	4,4′–DDE	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7; Note 4, p.30
20.	4,4′–DDT	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7; Note 4, p.30
21.	Demeton–O	GC GC/MS		8140 8250A	8141A 8270B			Note 3, p.25; Note 6, p.S51
22.	Demeton–S	GC GC/MS		8140 8250A	8141A 8270B			Note 3, p.25; Note 6, p.S51
23.	Diazinon	GC		8140	8141			Note 3, p.25; Note 4, p.30; Note 6, p.S51
24.	Dicamba	GC		8150B	8151			Note 3, p.115
25.	Dichlofenthion	GC		8140	8141			Note 4, p.30; Note 6, p.S73
26.	Dichloran	GC				6630 B & C	D3086–90	
27.	Dicofol	GC						
28.	Dieldrin	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B		Note 3, p.7; Note 4, p.30
29.	Dioxathion	GC GC/MS		8140 8250A	8141A 8270B			Note 4, p.30; Note 6, p.S73
30.	Disulfoton	GC GC/MS		8140 8250A	8141A 8270B			Note 3, p.25; Note 6, p.S51
31.	Diuron	HPLC						Note 10
32.	Endosulfan I	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7
33.	Endosulfan II	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7
34.	Endosulfan sulfate	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 C 6410 B		

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

	Parameter	Method	EPA[2,7]	SW–846[A,8] pkd[11]	SW–846[A,8] cap.	Standard Methods[R,9]	ASTM[c]	Other
35.	Endrin	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7; Note 4, p.30
36.	Endrin aldehyde	GC GC/MS	608 625	8080A 8250A	8081 8270B	6410 B	D3086–90	
37.	Ethion	GC GC/MS		8140 8250A	8141A 8270B			Note 4, p.30; Note 6, p.S73
38.	Fenuron	HPLC						Note 3, p.104; Note 6, p.S64
39.	Fenuron–TCA	HPLC						Note 10
40.	Heptachlor	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086–90	Note 3, p.7; Note 4, p.30
41.	Heptachlor epoxide	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B 6410 B	D3086–90	Note 3, p.7; Note 4, p.30; Note 6 p.S73
42.	Isodrin	GC GC/MS		8080A 8250A	8081 8270B			Note 4, p.30; Note 6, p.S73
43.	Linuron	HPLC						Note 10
44.	Malathion	GC GC/MS		8140 8250A	8141A 8270B	6630 C		Note 3, p.25; Note 4, p.30; Note 6, p.S51
45.	Methiocarb	HPLC						Note 10
46.	Methoxychlor	GC GC/MS		8080A 8250A	8081 8270B	6630 B & C	D3086–90	Note 3, p.7; Note 4, p.30
47.	Mexacarbate	HPLC GC/MS		 8250A	 8270B			Note 10
48.	Mirex	GC GC/MS		8080A 8250A	8081 8270B	6630 B & C		Note 3, p.7
49.	Monuron	HPLC						Note 10
50.	Monuron–TCA	HPLC						Note 10
51.	Neburon	HPLC						Note 10
52.	Parathion methyl	GC GC/MS		8140 8250A	8141A 8270B	6630 C		Note 3, p.25; Note 4, p.30
53.	Parathion ethyl	GC GC/MS		8140 8250A	8141A 8270B	6630 C	D3086–90	Note 3, p.25
54.	PCNB	GC GC/MS		8080A 8250A	8081 8270B	6630 B & C		Note 3, p.7
55.	Perthane	GC		8080A	8081		D3086–90	
56.	Prometon	GC						Note 3, p.83; Note 6, p.S68
57.	Prometryn	GC						Note 3, p.83; Note 6, p.S68
57.	Propazine	GC						Note 3, p.83; Note 6, p.S68
58.	Propham	HPLC						Note 10
59.	Propoxur	HPLC						Note 10
60.	Secbumeton	HPLC						Note 10
61.	Siduron	HPLC						Note 10
62.	Simazine	GC		8140	8141A			Note 3, p.83; Note 6, p.S68
63.	Strobane	GC		8080A	8081	6630 B & C		Note 3, p.7
64.	Swep	HPLC						Note 10
65.	2,4,5–T	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35
66.	2,4,5–TP (Silvex)	GC		8150B	8151	6640 B		Note 3, p.115
67.	Terbuthylazine	GC						Note 3, p.83; Note 6, p.S68

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

	Parameter	Method	EPA[2,7]	SW−846[A,8] pkd[11]	SW−846[A,8] cap.	Standard Methods[R,9]	ASTM[c]	Other
68.	Toxaphene	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086−90	Note 3, p.7; Note 4, p.30
70.	Trifluralin	GC GC/MS		8080A 8080A	8081 8270B	6630 B		Note 3, p.7

[A]"Test Methods for Evaluating Solid Waste", 3rd Edition. SW−846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512−1800.

[B]"Standard Methods for the Examination of Water and Wastewater", 18th 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. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[C]"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.

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

[2]The full text of methods 608 and 625 are given in Appendix A of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136), "Test Procedure for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit". 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.

[4]"Methods for Analysis of Organic Substances in Water", 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 a(alpha)−BHC, d(delta)−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 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 in 40 CFR part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on−going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[8]Some of these methods require a preliminary extraction. Methods 8141 A and 8081 require the use of either SW−846 method 3500/3510 or 3500/3520. Methods 8151 and 8270 B include the extraction steps necessary for most compounds. For methods 8081, 8141, and 8151 see also SW−846 method 8000 A and 3600.

[9]The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

[10]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.

[11]In order to reference these methods, the laboratory must use a packed column for the GC separations.

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	D1890−90	pp. 75 and 78[5]

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Table E
List of Approved Radiological Test Procedures For Wastewater

Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]
4. Beta–Counting error, pCi	Proportional Counter	Appendix B	7110 B	D1890–90	p. 79
5. (a) Radium–Total	Proportional Counter	903.0	7500Ra B	D2460–90	
(b) 226Ra, pCi per liter	Scintillation Counter	903.1	7500Ra C	D3454–7991	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", 17th or 18th 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, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[3] "1993 Annual Book of Standards, Water" Section 11.01 and 11.02, Water and Environmental Technology, American Society for Testing and Materials, 1993. 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	Digestion	Method	Method Number
Metals[1]			
Arsenic	3050A	Inductively Coupled Plasma Emission	6010A
Arsenic	7061A	Gaseous Hydride[2]	7061A
Arsenic	3050A	Graphite Furnace	7060A
Beryllium	3050A	Inductively Coupled Plasma Emission	6010A
Beryllium	3050A	Flame Atomic Absorption	7090
Beryllium	3050A	Graphite Furnace	7091
Cadmium	3050A	Inductively Coupled Plasma Emission	6010A
Cadmium	3050A	Flame Atomic Absorption	7130
Cadmium	3050A	Graphite Furnace	7131A
Chromium	3050A	Inductively Coupled Plasma Emission	6010A
Chromium	3050A	Flame Atomic Absorption	7190
Chromium	3050A	Graphite Furnace	7191
Copper	3050A	Inductively Coupled Plasma Emission	6010A
Copper	3050A	Flame Atomic Absorption	7210
Lead	3050A	Inductively Coupled Plasma Emission	6010A
Lead	3050A	Flame Atomic Absorption	7420
Lead	3050A	Graphite Furnace[3]	7421
Mercury	7471A	Cold Vapor	7471A
Molybdenum	3050A	Inductively Coupled Plasma Emission	6010A
Molybdenum	3050A	Flame Atomic Absorption	7480
Molybdenum	3050A	Graphite Furnace	7481
Nickel	3050A	Inductively Coupled Plasma Emission	6010A
Nickel	3050A	Flame Atomic Absorption	7520
Selenium	3050A	Inductively Coupled Plasma Emission	6010A
Selenium	7741A	Gaseous Hydride[2]	7741A
Selenium	3050A	Graphite Furnace	7740
Zinc	3050A	Inductively Coupled Plasma Emission	6010A

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Table EM
Approved Analytical Methods For Sludge

Parameter	Digestion	Method	Method Number
Zinc	3050A	Flame Atomic Absorption	7950
Biological			
Enteric viruses	NA	Centrifuge Concentration	D 4994−89[4]
Fecal coliform	NA	Most Probable Number Membrane Filter	9221 E or 9222 D[5]
Helminth ova	NA	Density Gradient Flotation	[6]
Specific Oxygen Uptake Rate	NA	Respirometer	2710 B[5]
Salmonella	NA	Most Probable Number Selective Media Culture	9260 D.1[5] [7]
Physical			
Solids	NA	Gravimetric	2540 G[5]
Percent Volatiles Solids Reduction	NA	Calculation	[8]

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[1]"Test Methods for Evaluating Solid Waste", SW−846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987 and July 1992 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.

[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", 17th or 18th 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 ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1992. 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.

[7]"Determination and Enumeration of *Salmonella* and *Pseudomonas aeruginosa*", Kenner, B.A. and H.A. Clark, J. Water Pollution Control Federation, 46(9):2163−2171, 1994. Available from the Water Environment Federation, 601 Wythe St., Alexandria, VA 22314.

[8]"Environmental Regulations and Technology – Control of Pathogens and Bextors in Sewage Sludge", EPA−625/R−92/013, Environmental Protection Agency, Cincinnati, OH, 1992. 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 3051 is an acceptable alternate digestion procedure to SW−846 method 3050A.

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:				
1−5.	Bacteria	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$[5]	6 hours
6−7.	Enteroviruses	P,G	Cool, 4°C	24 hours
8.	Mutagenicity	G, Teflon−lined cap	Cool, 4°C	7 days
9−12.	Acute & chronic toxicity	P,G	Cool, 4°C	48 hours
TABLE B – Inorganic Tests:				
1.	Acidity	P,G	Cool, 4°C	14 days
2.	Alkalinity	P,G	Cool, 4°C	14 days
4.	Ammonia	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
9.	Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
11.	Bromide	P,G	None required	28 days
14.	Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
15.	Chemical oxygen demand	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
16.	Chloride	P,G	None required	28 days
17.	Chlorine, total residual	P,G	None required	Analyze immediately
21.	Color	P,G	Cool, 4°C	48 hours
23−24.	Cyanide, total and amenable to chlorination	P,G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid[5]	14 days[6]
25.	Fluoride	P	None required	28 days
27.	Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
28.	Hydrogen ion (pH)	P,G	None required	Analyze immediately
31.,43.	Kjeldahl and organicnitrogen	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
38.	Nitrate	P,G	Cool, 4°C	48 hours
39.	Nitrate−nitrite	P,G	Cool, 4°C, H_2SO_4 to pH	28 days
40.	Nitrite	P,G	Cool, 4°C	48 hours
41.	Oil and grease	G	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
42.	Organic carbon	G	Cool, 4°C, HCl or H_2SO_4 or H_3PO_4 to pH<2	28 days

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Table F
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Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
44.	Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours
46.	Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately
47.	Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48.	Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days
49.	Phosphorus (elemental)	G	Cool, 4°C	48 hours
50.	Phosphorus, total	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
53.	Residue, total	P,G	Cool, 4°C	7 days
54.	Residue, Filterable	P,G	Cool, 4°C	7 days
55.	Residue, Nonfilterable (TSS)	P,G	Cool, 4°C	7 days
56.	Residue, Settleable	P,G	Cool, 4°C	48 hours
57.	Residue, Volatile	P,G	Cool, 4°C	7 days
61.	Silica	P, or Quartz	Cool, 4°C	28 days
64.	Specific conductance	P,G	Cool, 4°C	28 days
65.	Sulfate	P,G	Cool, 4°C	28 days
66.	Sulfide	P,G	Cool, 4°C, add zinc acetate plus NaOH to pH >9	7 days
67.	Sulfite	P,G	None required	Analyze immediately
68.	Surfactants	P,G	Cool, 4°C	48 hours
69.	Temperature	P,G	None required	Analyze immediately
73.	Turbidity	P,G	Cool, 4°C	48 hours
TABLE B – Metals[7]:				
10.	Boron	P, or Quartz	HNO_3 to pH<2	6 months
18.	Chromium VI	P,G	Cool, 4°C	24 hours
35. & 35m.	Mercury	P,G, or Teflon	HNO_3 to pH<2	28 days
71.	Tin	P	HCl or HNO_3 to pH<2	6 months
3, 5–8, 10, 12, 13, Metals:19, 20, 22, 26, 29, (except Cr VI, Sn, Hg, & B)30, 32–34, 36, 37,45, 47, 51, 52, 58–60, 62, 63, 70–72,74, 75.		P,G	HNO_3 to pH<2	6 months
TABLE C – Organic Tests[8]:				
IA.	Purgeable halocarbons	G, Teflon–lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days
IB.	Purgeable aromatics	G, Teflon–lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HCl to ph<2	14 days
IC.	Acrolein and acrylonitrile	G, Teflon–lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] Adjust pH to 4–5[10]	14 days
II.	Phenols	G, Teflon–lined cap	Cool, 4°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, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days after extraction 13

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

Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
III.	Phthlate esters[11]	G, Teflon–lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
IV.	Nitrosamines[11,14]	G, Teflon–lined cap	Cool, 4°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, 4°C	7 days until extraction; 40 days after extraction
VI.	Nitroaromatics, cyclic ketones and isophorone[11]	G, Teflon–lined cap	Cool, 4° 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, 4° 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, 4° C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
IX.	Chlorinated hydrocarbons[11]	G, Teflon–lined cap	Cool, 4° C	7 days until extraction; 40 days after extraction
X.	Chorinated Dioxans and Furans	G, Teflon–lined cap	Cool, 4° C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
TABLE E – Pesticide Tests:				
1–70.	Pesticides[11]	G, Teflon–lined cap	Cool, 4°C, pH 5–9[15]	7 days until extraction; 40 days after extraction
1–5.	Alpha, beta, and radium	P,G	HNO_3 to pH<2	6 months

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

[2]All samples requiring preservation at 4°C must be cooled immediately after collection, and 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 4 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 4°C until compositing and sample splitting are completed.

[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 J, 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).

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[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. Virus samples can be stored indefinitely at −70°C. Samples used for toxicity tests are to be used for test initiation or for renewal of test solutions within 36 hours of collection as grab samples or after removal from composite samplers. 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.

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

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

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

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

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

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

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

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

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

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

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

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 health and family services. 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.