

Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

NR 219.01	Purpose	NR 219.05	Alternate test procedures
NR 219.02	Applicability	NR 219.06	Laboratory certification or registration
NR 219.03	Definitions		
NR 219.04	Identification of test 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. 147.08 (1) and 144.95, 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.

**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. 147, Stats.

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

(2) Section NR 219.07 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149, HSS 157 or 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.

**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 analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. 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 219.06.

(2) 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 should be preserved at the time of collection by cooling to 4°C. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications

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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.

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. Register, November, 1992, No. 443, eff. 12-1-92.

**NR 219.05 Alternate test procedures.** Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. An alternate test procedure may only be used if the procedure has been approved by EPA.

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.

**NR 219.06 Approval of alternate test procedures.** History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. (1) to (3) and cr. (4), January, 1978, No. 265, eff. 2-1-78; renum. from NR 219.05 and am. Register, June, 1986, No. 366, eff. 7-1-86; r. Register, November, 1992, No. 443, eff. 12-1-92.

TABLE A  
LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and Units	Method <sup>1</sup>	EPA	Standard Methods 17th Ed.	USGS
<b>Bacteria:</b>				
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF) <sup>2</sup> , single step.	p132 <sup>3</sup> p124 <sup>3</sup>	9221C 9222C	8-0050-85 <sup>4</sup>
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or MF, single step <sup>3</sup>	p132 <sup>3</sup> p124 <sup>3</sup>	9221C 9222D	
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF <sup>2</sup> single step or two step	p114 <sup>3</sup> p108 <sup>3</sup>	9221B 9222B	8-0025-85 <sup>4</sup>
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF <sup>2</sup> with enrichment.	p114 <sup>3</sup> p111 <sup>3</sup>	9221B 9222B+B.5c	
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF <sup>2</sup> , or Plate count	p136 <sup>3</sup> p134 <sup>3</sup> p143 <sup>3</sup>	9230B 9230C	8-0055-85 <sup>4</sup>
<b>Enteroviruses:</b>				
6. Enteroviruses in water, plaque forming units per liter.	Absorption, elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 9 <sup>6</sup> Ch. 10 <sup>6</sup> Ch. 12 <sup>6</sup>	Ch. 6 <sup>6</sup> 9510 G 9510 G 9510 G	9510 B,C,D,E
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 <sup>6</sup> Ch. 10 <sup>6</sup> Ch. 12 <sup>6</sup>	9510 F Ch. 9 <sup>6</sup> 9510G 9510G	9510 G
<b>Mutagenicity:</b>				
8. Mutagenicity (revertants per liter)	Ames test, test strains TA97, TA98, TA100, and TA102.	Note 7		
<b>Acute and Chronic Toxicity:</b>				
9. Toxicity, acute, fresh water organisms, effluent <sup>10</sup>	Daphnia and Ceriodaphnia, 48-h static mortality. Fathead minnow, 48-h static mortality, or 48 to 96-h flow-through mortality.	p 39 <sup>8</sup> p 41 <sup>8</sup>		
10. Toxicity, chronic, fresh water organisms, percent effluent. <sup>10</sup>	Fathead minnow larval survival and growth. Fathead minnow embryo-larval survival and teratogenicity. Ceriodaphnia survival and reproduction. Selenastrum growth.	1000.0 <sup>9</sup> 1001.0 <sup>9</sup> 1002.0 <sup>9</sup> 1003.0 <sup>9</sup>		

## TABLE A NOTES:

- 1 The method used must be specified when results are reported.
- 2 A 0.45  $\mu\text{m}$  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 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, CERL, 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 Peltier, W.H., and C.I. Weber, eds. September 1991. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-90/027.
- 9 Weber, C.I., W.H. Peltier, T.J. Norberg-King, W.B. Horning, II, F.A. Kessler, J.R. Menkedick, T.W. Neiheisel, P.A. Lewis, D.J. Klemm, O.H. Pickering, E.L. Robinson, J.M. Lazorchak, L.J. Wymer, and R.W. Freyberg. 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Surface Waters to Freshwater Organisms, Second Edition, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4-89/001).
- 10 Compliance monitoring must be performed in accordance with the specifications in "Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Testing", Wisconsin Department of Natural Resources, May 1992. Available from the Department of Natural Resources Office of Technical Services, P O Box 7921, Madison, WI 53707.

TABLE B  
LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA <sup>1</sup>	Standard Methods <sup>2</sup>	ASTM <sup>3</sup>	USGS <sup>4</sup>	Other
1. Acidity, as CaCO <sub>3</sub> , mg/L: Electrometric end point or phenolphthalein end point	305.1	2310 B(4a)	B1067-88		
2. Alkalinity, as CaCO <sub>3</sub> , mg/L: Electrometric or colorimetric: Titration to pH 4.5, manual Or automated	310.1 310.2	2320 B	D1067-88	1-1030-85	973.43 <sup>5</sup>
3. Aluminum-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma (ICP), Direct current plasma (DCP), or Colorimetric (Eriochrome cyanine R)	202.1 202.2 200.7 <sup>7</sup>	3111 D 3113 B 3120 B 3500-AID	D4190-88	1-3051-85	Note 34
4. Ammonia (as N), mg/L: Manual distillation <sup>8</sup> (at pH 9.5): Followed by: Nesslerization, Titration, Electrode, Automated photate, or Automated electrode	350.2 350.2 350.2 350.3 350.1	4500-NH <sub>3</sub> B 4500-NH <sub>3</sub> C 4500-NH <sub>3</sub> E 4500-NH <sub>3</sub> F & G 4500-NH <sub>3</sub> H	D1426-79(A) D1426-79(D) D1426-79(C)	1-3520-85 1-4522-85	973.49 <sup>5</sup> 973.46 <sup>5</sup> Note 9
5. Antimony - Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma	204.1 204.2 200.7 <sup>7</sup>	3111 B 3113 B 3120 B			
6. Arsenic - Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by AA (gaseous hydride), AA furnace, Inductively coupled plasma, Or, colorimetric (SDDC)	206.5 206.2 200.7 <sup>7</sup> 206.4	3114 3113-4d 3120 B 3500-As	D2972-84(B) D2972-84(A)	1-3062-85 1-3060-85	
7. Barium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, or DCP	208.1 208.2 200.7 <sup>7</sup>	3111 D 3113 B 3120 B		1-3084-85	Note 34
8. Beryllium-Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, DCP, or Colorimetric (alumina)	210.1 210.2 200.7 <sup>7</sup>	3111 D 3113 B 3120 B 3500-Ba D	D3654-88(A) D4190-88	1-3095-85	Note 34
9. Biochemical oxygen demand (BOD <sub>5</sub> ), mg/L: Winkler (Azide modifications) Or electrode method	405.1	5210		1-1578-76 <sup>9</sup>	973.44 <sup>5</sup> p. 17 <sup>11</sup>
10. Boron-Total, mg/L: Colorimetric (arsoniazin), Inductively coupled plasma, or DCP	212.3 200.7 <sup>7</sup>	4500 B B 3120 B	D4190-88	1-3112-85	Note 34
11. Bromide, mg/L: Titrimetric	320.1		D1246-82 (C)(1988)	1-1125-85	p. 544 <sup>12</sup>

TABLE II (continued)

## LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA <sup>1</sup>	Standard Methods <sup>A</sup>	ASTM <sup>B</sup>	USGS <sup>12</sup>	Other
12. Cadmium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration,	2131	3111 B or C	D3557-90(A or B)	1-3135-85 or 1-3136-85	974.27 <sup>5</sup> p.37 <sup>11</sup>
AA furnace, Inductively coupled plasma ICP, Volametry <sup>13</sup> , or Colorimetric (Dimethylac)	2132, 200.7 <sup>7</sup>	3113B 3120B	D4190-90 D3557-90(C)	1-1472-85	Note 34
13. Calcium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: Atomic absorption, Inductively coupled plasma, ICP, or EDTA titration	215.1, 200.7 <sup>7</sup> 215.2	3111 B 3120 B 3500-Ca D	D511-88(B) D511-88(A)	1-3152-85	Note 34
14. Carboxeous Biochemical oxygen demanded (CBOD <sub>5</sub> ), mg/L: with nitrification inhibitor <sup>14</sup>		5210 B			
15. Chemical oxygen demanded (COD), mg/L: Titrimetric	410.1 410.2 410.3 410.4	5220 B	D1252-88	1-3550 or 1-3552-85	973.46 <sup>5</sup> p.17 <sup>11</sup>
Automated and manual Spectrophotometric	410.4			1-3561-	Notes 15 or 16
16. Chloride, mg/L: Titrimetric (silver nitrate) or (Mercuric nitrate), Colorimetric (ferricyanide), manual or automated	325.3 325.1 or 325.2	4500-Cl <sup>-</sup> B 4500-Cl <sup>-</sup> C 4500-Cl <sup>-</sup> E	D1512-89(B) D1512-89(A) D1512-89(C)	1-1183-85 1-1184-85 1-1187-85 1-2187-85	973.51 <sup>5</sup>
17. Chlorine - Total residual, mg/L: amperometric, Starb EAS point direct	330.1 330.3	4500-Cl D 4500-Cl B	D1253-76(A) D1253-76(B) (1983) Part 18.3		
Back titration either end point <sup>17</sup> , or DPD-PAS, Spectrophotometric, DPD, or Electrode	330.2 330.4 330.5	4500-Cl C 4500-Cl F 4500-Cl G			Note 18
18. Chromium VI dissolved, ug/L: 0.45 micron filtration with: Extraction and atomic absorption, or Colorimetric (Diphenylcarbazide)	218.4	3111 A		1-1232-85 1-1230-85	307B <sup>19</sup>
19. Chromium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> (optional extraction) followed by: AA direct aspiration, AA chelation extraction AA furnace, Inductively coupled plasma, ICP, or Colorimetric (diphenylcarbazide),	218.1 218.3 218.2 200.7 <sup>7</sup>	3111 B 3111 C 3113B 3120B	D1687-84(D) D4190-88 D1687-84(A)	1-3236-85	974.24 <sup>5</sup> Note 34

TABLE B (continued)

## LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA <sup>1</sup>	Standard Methods <sup>A</sup>	ASTM <sup>B</sup>	USGS <sup>C</sup>	Other
20. Cobalt-Total <sup>5</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma, or DCP	219.1	3111 B (A or B)	D3558-90	1-3239-84	P-37 <sup>11</sup>
	219.2	3113 B			
	200.7 <sup>7</sup>	3120 B	D4190-88		Note 34
21. Color, Platinum Cobalt units or dominant wavelength etc. Irradiance, purity: Colorimetric, ADM1 Platinum cobalt, or Spectrophotometric	110.1	2129 E		1-1250-85	Note 20
	110.2	2120 B			
	110.3	2120 C			
22. Copper-Total <sup>5</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration,  AA furnace, Inductively coupled plasma DCP, Colorimetric (Neocuproine), or Bismethionate	220.1	3111 B or C	D1668-90 (A or B)	1-3271-85 or 1-3270-85	974.27 <sup>5</sup> p.39 <sup>11</sup>
	220.2	3113 B			
	200.7 <sup>7</sup>	3120 B	D4190-88		Note 34
		3500-Cu D or E	D1668-84(88)(A)		Note 21
23. Cyanide - Total, ug/L: Manual distillation with MgCl <sub>2</sub> Followed by titrimetric, Manual or Automated <sup>12</sup> spectrophotometric	335.2	4500-CN-C 4500-CN-D		1-3300-85	p. 22 <sup>11</sup>
	335.3	4500-CN-E	D2036-89(A) D2036-89(A)		
		4500-CN-G	D2036-89(B)		
24. Cyanide amenable to chlorination, ug/L: Manual distillation with MgCl <sub>2</sub> followed by titrimetric, manual or automated spectrophotometric	335.1	4500-CN-G	D2036-89(B)		
25. Fluoride - Total, mg/L: Manual distillation <sup>8</sup> Followed by manual or automated electrode, SPADNS,  Or automated complexone	340.2	4500-F-B 4500-F-C	D1179-88(B)	1-4327-85	
	340.1	4500-F-D	D1179-80(A) (1988)		
	340.3	4500-F-E			
26. Gold Total <sup>5</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration AA furnace, or DCP	231.1	3111 B			
	231.2	3113 B			Note 34
27. Hardness - Total as CaCO <sub>3</sub> , mg/L: Automated colorimetric, EDTA titration, (or the sum of Ca and Mg as their respective carbonates by ICP or AA direct aspiration) (See Parameters 13 and 33)	130.1				
	130.2	2340 C	D1126-86 (1990)	1-1338-85	973.52B <sup>5</sup>
28. Hydrogen Ion (pH), pH units: Electrometric Measurements or Automated Electrode	150.1	4500-H <sup>9</sup> B	D1293-84 (A or B) (1990)	1-1586-85	973.41 <sup>5</sup>  Note 23
29. Iridium - Total <sup>5</sup> , ug/L: Digestion <sup>6</sup> followed by:					

TABLE B (continued)

## LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA <sup>1</sup>	Standard Methods <sup>A</sup>	ASTM <sup>B</sup>	USGS <sup>C</sup>	Other
AA direct aspiration Or AA furnace	235.1 235.2	3111 B			
30. Iron-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration,	236.1	3111 B or C	D1068-84 (C or D)	1-3381-84	973.27 <sup>5</sup>
AA furnace, Inductively coupled plasma, DCP, or Colorimetric (Phenanthroline)	236.2 200.7 <sup>7</sup>	3113 B 3120 B 3500-Fe D	D1068-84(A)		Note 34 Note 24
31. Kjeldahl nitrogen - Total (as N), mg/L: Digestion and distillation Followed by titration Nesslerization or Electrode Automated phenate, Seal-automated block digester, Or potentiometric	351.3 351.3 351.3 351.3 351.1 351.2 351.4	4500-N org B or C 4500-NH <sub>3</sub> E 4500-NH <sub>3</sub> C 4500-NH <sub>3</sub> F or G 4500-NH <sub>3</sub> H	3590-84(A) D3390-89(A) D3390-89(A)	1-4551-78 <sup>8</sup>	973.46 <sup>5</sup>
32. Lead-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration,	239.1	3111 B or C	D3359-85 (A or B)	1-3399-90	974.27 <sup>5</sup>
AA furnace, Inductively coupled plasma, DCP, Voltammetry <sup>13</sup> or Colorimetric (Dithionite)	239.2 200.7 <sup>7</sup>	3113 B 3120 B 3500-Pb D	D4190-88 D3359-90(C)		Note 34
33. Magnesium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: Atomic absorption, Inductively coupled plasma, DCP, or Gravimetric	242.1 200.7 <sup>7</sup>	3111 B 3120 B 3500-Mg D	D511-88(B) D511-77(A)	1-3447-85	974.27 <sup>5</sup> Note 34
34. Manganese-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, DCP, Colorimetric (Periodate), or Periodate	243.1 243.2 200.7 <sup>7</sup>	3111 B or C 3113 B 3120 B 3500-Mn D	D358-90 (A or B) D4190-88 D358-84(A)(1988)	1-3454-85	974.27 <sup>5</sup> Note 34 920.203 <sup>5</sup> Note 25
35. Mercury - Total <sup>6</sup> , ug/L: Cold vapor, manual or automated	245.1 245.2	3142 B	D3223-86	1-3462-85	977.22 <sup>5</sup>
36. Molybdenum-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, or DCP	266.1 266.2 200.7 <sup>7</sup>	3111 D 3113 B 3120 B		1-3490-85	Note 34
37. Nickel-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, DCP, or	249.1 249.2 200.7 <sup>7</sup>	3111 B or C 3113 B 3120 B	D1886-90 (A or B) D4190-88	1-3499-85	Note 34



TABLE B (continued)

## LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Units & Methods	EPA <sup>1</sup>	Standard Methods <sup>2</sup>	ASTM <sup>3</sup>	USGS <sup>4</sup>	Other
Colorimetric (Heptoxime)		3500-NI D			
38. Nitrate (as N), mg/L: Diacine sulfate, or Nitrate-nitrite N minus Nitrite N (see parameters 39 and 40)	352.1		D992-71		973.50 <sup>5</sup> 419D <sup>19</sup> P. 28 <sup>11</sup>
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction, manual Or automated, or automated hydrazine	353.3 353.2 353.1	4500-NO <sub>3</sub> E 4500-NO <sub>3</sub> F 4500-NO <sub>3</sub> H	D867-60(B) D3867-90(A)	1-4545-85	
40. Nitrite (as N), mg/L: Spectrophotometric, manual or automated (Diazotization)	354.1	4400-NO <sub>2</sub> B	D1254-67	1-4540-85	Note 27
41. Oil and grease-Total recoverable, mg/L: Gravimetric (extraction)	413.1	5520 B			
42. Organic carbon - Total (TOC), mg/L: Combustion or oxidation	415.1	5310 B	D2579-85 (A or B)		973.47 <sup>5</sup> p. 14 <sup>26</sup>
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 Or manual single reagent or Manual two reagent	365.1 365.2 365.3	4500-P F 4500-P E	D515-88(A)	1-4601-85	973.56 <sup>5</sup> 973.55 <sup>5</sup>
45. Oxidum - Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, or AA furnace	252.1 252.2	3111 D			
46. Oxygen, dissolved, mg/L: Winkler (Azide modification) Or electrode	350.2 360.1	4500-O C 4500-O G	D888-81(C) (1988)	1-4575-78 <sup>19</sup> 1-4576-78 <sup>19</sup>	973.45B <sup>5</sup>
47. Palladium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, or DCP	253.1 253.2	3111 B			P 527 <sup>11</sup> P 528 <sup>11</sup> Note 34
48. Phenols, mg/L: Manual distillation <sup>28</sup> Followed by manual Or automated <sup>21</sup> colorimetric (AAP)	420.1 420.1 420.2		D1783-80 (A or B)		Note 29 Note 29
49. Phosphorus (elemental), mg/L: Gas-liquid chromatography					Note 30
50. Phosphorus - Total, mg/L: Persulfate digestion Followed by manual or Automated ascorbic acid Reduction, or semi-automated block digester	365.2 365.2 or 365.3 365.1 365.4	4500-P B5 4500-P E 4500-P F	D515-88 (A)	1-4600-85	973.55 <sup>5</sup> 973.56 <sup>5</sup>
51. Platinum-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by:					

TABLE B (continued)

Parameter, Units & Methods	LIST OF APPROVED INORGANIC TEST PROCEDURES				
	EPA <sup>1</sup>	Standard Methods <sup>A</sup>	ASTM <sup>B</sup>	USOS <sup>1</sup>	Other
AA direct aspiration, AA furnace, or DCP	255.1 255.2	3111 B			Note 34
52. Potassium - Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: Atomic absorption, Inductively coupled plasma, Flame photometric, or Colorimetric (cobaltinitrate)	258.1 200.7 <sup>7</sup>	3111 B 3120 B 3500-K D	D1428-82(A)	1-3620-85	973.53 <sup>5</sup> 317B <sup>19</sup>
53. Residue - total, mg/L: Gravimetric 103-105°C	160.3	2540 B		1-3750-85	
54. Residue - filterable, mg/L: Gravimetric, 180°C	160.1	2540 C		1-1750-85	
55. Residue - nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	2540 D		1-3765-85	
56. Residue - settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	2540 F			
57. Residue - volatile mg/L: Gravimetric, 550°C	160.4	2540 E		1-3753-85	
58. Rhodium - Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by: AA direct aspiration Or AA furnace	265.1 265.2	3111 B			
59. Ruthenium - Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by: AA direct aspiration Or AA furnace	267.1 267.2	3111 B			
60. Selenium - Total <sup>6</sup> , ug/L: Digestion <sup>6</sup> followed by: AA furnace, Inductively coupled plasma, or AA (gaseous hydride)	270.2 200.7 <sup>7</sup>	3113 B 3120 B 3114 B	D3839-88(A)	1-3667-85	
61. Silica - Dissolved, mg/L: 0.45 micron filtration: Followed by manual or automated colorimetric (Molybdo-silicic), or Inductively coupled plasma	370.1 200.7	4500-Si D	D659-88(B)	1-1700-85 1-2700-85	
62. Silver-Total <sup>11</sup> , mg/L: Digestion <sup>6</sup> followed by: AA direct aspiration, AA furnace, Colorimetric (Dithionite), Inductively coupled plasma, or DCP	272.1 272.2 200.7 <sup>7</sup>	3111 B or C 3113 B 3120 B		1-3720-85	973.27 <sup>5</sup> p. 37 <sup>11</sup> 319B <sup>19</sup> Note 34
63. Sodium-Total <sup>6</sup> , mg/L: Digestion <sup>6</sup> followed by: Atomic absorption, Inductively coupled plasma, DCP, or Flame photometric	273.1 200.7 <sup>7</sup>	3111 B 3120 B 3500-N1 D	D1428-82(A)	1-3735-85	973.54 <sup>4</sup> Note 34

TABLE B (continued)  
LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, Unit & Methods	EPA <sup>1</sup>	Standard Methods <sup>A</sup>	ASTM <sup>B</sup>	USGS <sup>C</sup>	Other
64. Specific conductance, microhm/cm at 25°C: Wheatstone bridge	1201	2510 B	D1125-87(A)	1-1780-85	973.40 <sup>D</sup>
65. Sulfate (as SO <sub>4</sub> ), mg/L: Automated colorimetric (barium chloranilate), Gravimetric, or Turbidimetric	375.1 375.3 375.4	4500-SO <sub>4</sub> <sup>2</sup> -C or D	D516-82(A) (1988) D516-88		925.54 <sup>E</sup> 426C <sup>F</sup>
66. Sulfide (as S), mg/L: Titrimetric (iodine) or Colorimetric (methylene blue)	376.1 376.2	4500-S <sup>2</sup> -E 4500-S <sup>2</sup> -D		1-3840-85	228A <sup>G</sup>
67. Sulfite (as SO <sub>3</sub> ), mg/L: Titrimetric (iodine-iodate)	377.1	4500-SO <sub>3</sub> <sup>2</sup> -E	D1339-84(C)		
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1	5540 C	D2330-88		
69. Temperature, °C: Thermometric	470.1	2550 B			Note 34
70. Thallium - Total <sup>H</sup> , µg/L: Digestion <sup>H</sup> followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma	279.1 279.2 200.7 <sup>I</sup>	3111 B 3113 B			
71. Tin - Total <sup>H</sup> , µg/L: Digestion <sup>H</sup> followed by: AA direct aspiration or AA furnace	282.1 282.2	3111 B 3113 B		1-3850-78 <sup>J</sup>	
72. Titanium - Total <sup>H</sup> , mg/L: Digestion <sup>H</sup> followed by: AA direct aspiration, AA furnace, or DCP	283.1 283.2	3111 D 3113 B			Note 34
73. Turbidity, NTU: Nephelometric	180.1	2130 B	D1889-88a	1-3860-85	
74. Vanadium - Total <sup>H</sup> , µg/L: Digestion <sup>H</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, DCP, or Colorimetric (Gallic acid)	285.1 285.2 200.7 <sup>I</sup>	3111 D 3113 B 3120 B	D4190-88 D3373-84(A) (1988)		Note 34
75. Zinc - Total <sup>H</sup> , mg/L: Digestion <sup>H</sup> followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, DCP, Colorimetric (Dithionite), or Colorimetric (Zincron)	289.1 289.2 200.7 <sup>I</sup>	3111 B or C 3113 B 3120 B	D1691-90(A or B) D4190-88 3500-Zn E 3500-Zn F	1-3900-55	974.27 <sup>K</sup> P.37 <sup>L</sup> Note 34 Note 34

TABLE B NOTES

<sup>1</sup> 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.

- <sup>2</sup> "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, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
- <sup>3</sup> "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- <sup>4</sup> "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.
- <sup>5</sup> "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.
- <sup>6</sup> For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979". One (4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and thallium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.
- Note: If the digestion included in one of the other approved references is different than the above, the EPA procedure will be used.
- Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals will be followed. Sample digestion for dissolved metals may be omitted for AA (direct aspiration or graphite furnace) and ICP 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.
- <sup>7</sup> The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", is given in Appendix C of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136). Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20540.
- <sup>8</sup> 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.
- <sup>9</sup> Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 15, 1976, Technicon AutoAnalyzerII. Available from Technicon Industrial Systems, Bauschert Avenue, Tarrytown, NY 10591.
- <sup>10</sup> 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.
- <sup>11</sup> "American National Standard on Photographic Processing Efficiency", April 2, 1975. Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.
- <sup>12</sup> "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.
- <sup>13</sup> The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- <sup>14</sup> Carboxaceous biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional BOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required, can the permittee report data obtained using the nitrification inhibitor.
- <sup>15</sup> OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West loop, P.O. Box 2980, College Station, TX 77840.
- <sup>16</sup> Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- <sup>17</sup> The back titration method will be used.
- <sup>18</sup> ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.
- <sup>19</sup> 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.

- <sup>20</sup> "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.
- <sup>21</sup> Copper, Bifunctionalate Method, Method 8506, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- <sup>22</sup> 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 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.
- <sup>23</sup> 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.
- <sup>24</sup> 1, 10-Phenanthroline Method for Iron, Hach Method 8008, 1982. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- <sup>25</sup> 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.
- <sup>26</sup> "Methods for Analysis of Organic Substances in Water", by D. F. Gorlitz 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.
- <sup>27</sup> Nitrite Nitrogen, Hach Method 8507. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- <sup>28</sup> Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1 + 9 NaOH.
- <sup>29</sup> 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  $10.0 \pm 0.2$ . 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.
- <sup>30</sup> "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", by R. F. Addison and R. G. Arltman, 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.
- <sup>31</sup> 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  $\text{Na}_2\text{S}_2\text{O}_3$  and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- <sup>32</sup> The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 15th Edition. Available on inter-library loan.
- <sup>33</sup> The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 13th Edition. Available on inter-library loan.
- <sup>34</sup> "Water Temperature-Influential Factors, Field Measurement, and Data Presentation", by H. H. Stevan, Jr., J. Fiele, and G. P. Smoot. USGS-TWRI Book 1, Chapter D1, 1975. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- <sup>35</sup> Zincocin Method of Zinc Method 8009. Hach Handbook for Water Analysis, 1979, pp. 2-235 and 2-333. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- <sup>36</sup> "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method A1E8029," 1986-Revised 1991, Applied Research Laboratories, Inc., 24911 Avenue Stanford, Valencia, CA 91355.

TABLE C  
List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter <sup>1</sup>	GC	GC/MS	FPLC	Standard Method <sup>2</sup>	ASTM <sup>3</sup>	Other
1. Acenaphthene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
2. Acenaphthylene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
3. Acroben	603	624, 1624	610	6410 B, 6440 B	D4657-87	
4. Acrylonitrile	603	624, 1624	610	6410 B, 6440 B	D4657-87	
5. Anthracene	602	624, 1624	610	6410 B, 6440 B	D4657-87	
6. Benzene	602	624, 1624	610	6410 B, 6440 B	D4657-87	
7. Benzidine	605	625, 1625	605	6210 B, 6250 B		
8. Benzofuranone	610	625, 1625	610	6410 B, 6440 B	D4657-87	
9. Benzofuran	610	625, 1625	610	6410 B, 6440 B	D4657-87	
10. Benzofuranone	610	625, 1625	610	6410 B, 6440 B	D4657-87	
11. Benzofuranone	610	625, 1625	610	6410 B, 6440 B	D4657-87	
12. Benzofuranone	610	625, 1625	610	6410 B, 6440 B	D4657-87	
13. Benzyl alcohol	606	625, 1625	610	6410 B		Note 3, p. 130
14. Benzyl butyl phthalate	611	625, 1625	610	6290 B, 6410 B		
15. Bis(2-chloroethyl) methane	611	625, 1625	610	6410 B		
16. Bis(2-chloroethyl) ether	611	625, 1625	610	6410 B		
17. Bis(2-ethylhexyl)phthalate	606	624, 1624	610	6410 B, 6290 B		
18. Bromochloromethane	601	624, 1624	610	6410 B, 6290 B		
19. Bromoform	601	624, 1624	610	6410 B, 6290 B		
20. Bromonethane	601	624, 1624	610	6410 B		
21. 4-Bromophenyl phenyl ether	611	625, 1625	610	6290 B, 6410 B		Note 3, p. 130
22. Carbon tetrachloride	604	624, 1624	610	6210 B, 6230 B, 6250 B		
23. 4-Chloro-3-methylphenol	601	624, 1624	610	6210 B, 6230 B		
24. Chlorobenzene	601	624, 1624	610	6210 B, 6230 B		
25. Chloroethane	601	624, 1624	610	6210 B, 6230 B		
26. Chloroform	601	624, 1624	610	6210 B, 6230 B		
27. Chloroform	601	624, 1624	610	6210 B, 6230 B		
28. Chloroform	601	624, 1624	610	6210 B, 6230 B		
29. Chloroform	601	624, 1624	610	6210 B, 6230 B		
30. 2-Chlorophenol	604	624, 1624	610	6410 B, 6420 B		
31. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
32. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
33. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
34. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
35. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
36. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
37. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
38. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
39. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
40. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
41. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
42. Chloroethane	611	625, 1625	610	6410 B, 6420 B		
43. trans-1,2-Dichloroethane	604	624, 1624	610	6410 B, 6440 B, 6220 B	D4656-87	
44. 2,4-Dichlorophenol	604	624, 1624	610	6410 B, 6440 B, 6220 B	D4656-87	
45. 1,2-Dichloropropane	601	624, 1624	610	6410 B, 6440 B, 6220 B		
46. cis-1,3-Dichloropropane	601	624, 1624	610	6410 B, 6440 B, 6220 B		
47. trans-1,3-Dichloropropane	601	624, 1624	610	6410 B, 6440 B, 6220 B		

TABLE C (continued)  
List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter <sup>1</sup>	OC	OCMS	HPLC	Standard Method <sup>2</sup>	ASTM <sup>10</sup>	Other
48. Diethyl phthalate	606	625 1625		6410 B		
49. 2,4-Diacetylphenol	604	625 1625		6420 B, 6410 B		
50. Diethyl phthalate	606	625 1625		6410 B		
51. Di-n-butyl phthalate	606	625 1625		6410 B		
52. Di-n-octyl phthalate	604	625 1625		6420 B, 6410 B		
53. 2,4-Dinitrophenol	609	625 1625		6410 B		
54. 2,4-Dinitrotoluene	609	625 1625		6410 B		
55. 2,6-Dinitrotoluene	602	624 1624		6220 B, 6210 B		
56. Epichlorohydrin	610	625 1625	610	6410 B, 6440 B	D4657-87	
57. Ethylbenzene	610	625 1625	610	6410 B, 6440 B	D4657-87	
58. Fluoranthene	610	625 1625		6410 B		
59. Fluorene	612	625 1625		6410 B		
59a. 1,2,3,4,6,7,8-Hepsochloro-dibenzo-p-dioxin	612	625 1625		6410 B		
59b. 1,2,3,4,6,7,8-Hepsochloro-dibenzofuran	612	625 1625		6410 B		
59c. 1,2,3,4,7,8,9-Hepsochloro-dibenzofuran	612	625 1625		6410 B		
60. Hexachlorobenzene	612	625 1625		6410 B		
61. Hexachlorobutadiene	612	625 1625		6410 B		
62. Hexachlorocyclopentadiene	612	625 1625		6410 B		
62a. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	612	625 1625		6410 B		
62b. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	612	625 1625		6410 B		
62c. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	612	625 1625		6410 B		
62d. 1,2,3,4,7,8-Hexachlorodibenzofuran	612	625 1625		6410 B		
62e. 1,2,3,4,6,7,8-Hexachlorodibenzofuran	612	625 1625		6410 B		
62f. 1,2,3,4,6,7,8-Hexachlorodibenzofuran	612	625 1625		6410 B		
63. Hexachlorobenzene	612	625 1625		6410 B		
64. Heptachlorobenzene	612	625 1625		6410 B		
65. Heptachloro-dipyrrene	612	625 1625	610	6410 B, 6440 B	D4657-87	
66. Heptachloro-cyclopentadiene	609	625 1625		6410 B		
67. Methylene chloride	601	624 1624		6220 B		
68. 2-Methyl-4,6-dinitrophenol	604	625 1625		6420 B, 6410 B		
69. Naphthalene	610	625 1625	610	6410 B, 6440 B		
70. Nitrobenzene	609	625 1625		6410 B		
71. 4-Nitrophenol	604	625 1625		6410 B, 6420 B		
72. N-Nitrosodimethylamine	607	625 1625		6410 B		
73. N-Nitrosodi-n-propylamine	607	625 1625		6410 B		
74. N-Nitrosodiphenylamine	607	625 1625		6410 B		
74a. Octachlorodibenzofuran	612	625 1625		6410 B		
74b. Octachlorodibenzofuran	612	625 1625		6410 B		
74c. 2,2'-Oxybis (1-chloropropane)	611	625 1625		6410 B		
75. PCB-1016	608	625		6410 B		Note 3, p.43
76. PCB-1221	608	625		6410 B		Note 3, p.43
77. PCB-1222	608	625		6410 B		Note 3, p.43
78. PCB-1242	608	625		6410 B		Note 3, p.43
79. PCB-1248	608	625		6410 B		Note 3, p.43
80. PCB-1254	608	625		6410 B		Note 3, p.43
81. PCB-1254	608	625		6410 B		Note 3, p.43
82. PCB-1260	608	625		6410 B, 6430 B		Note 3, p.43

Note 3, p. 130; Note 6, p. S102

Note 3, p. 130

Note 3, p.43

Note 3, p.43

Note 3, p.43

Note 3, p.43

Note 3, p.43

TABLE C (continued)  
List of Approved Test Procedures for Non-Pesticide Organic Compounds

EPA METHOD NUMBER 27

Parameter	CC	COMS	HPLC	Standard Methods <sup>6</sup>	ASTM <sup>20</sup>	Other
82a. 1,2,3,7,8-Pentachlorodibenzo-p-dioxin		1613A <sup>6</sup>				
82b. 1,2,3,7,8-Pentachlorodibenzofuran		1613A <sup>6</sup>				
82c. 2,3,4,7,8-Pentachlorodibenzofuran		1613A <sup>6</sup>				
87a. 2,3,7,8-Tetrachlorodibenzofuran		1613A <sup>6</sup>				
83. Pentachlorophenol	604	625 1625	610	6410 B, 6430 B	D4657-87	Note 3, p.140
84. Benanthracene	604	625 1625	610	6410 B, 6440 B		
85. Phenol	604	625 1625	610	6410 B, 6410 B		
86. Pyrene	610	625 1625	610	6410 B, 6440 B	D4657-87	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin		5*613, 1613 A				
88. 1,1,2,2-Tetrachloroethane	601	624 1624		6230 B, 6210 B		Note 3, p.130
89. Trichloroethene	601	624 1624		6230 B, 6210 B		Note 3, p.130
90. Toluene	612	625 1624		6210 B, 6230 B		
91. 1,2,4-Trichlorobenzene	612	625 1625		6410 B		Note 3, p.130
92. 1,1,1-Trichloroethane	601	624 1624		6210 B, 6230 B		
93. 1,1,2-Trichloroethane	601	624 1624		6210 B, 6230 B		
94. Trichloroethene	601	624 1624		6210 B, 6230 B		Note 3, p.130
95. Tetrahydrofuran	601	624		6210 B, 6230 B		
96. Tetrahydrofuranmethane	604	625 1625		6410 B, 6240 B		
97. 2,4-Dichlorophenol	601	624 1624		6210 B, 6230 B		
97. Vinyl chloride						

TABLE C NOTES

- All parameters are expressed in micrograms per liter (µg/L).
- The full text of Methods 601-613, 624, 625, 1624, and 1625, are given in Appendix A of 40 CFR part 136. 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. 20540.
- \*Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorobenzene and Pesticides in Water and Wastewater; Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, 26 W. St. Clair, Cincinnati, Ohio 45268.
- 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.
- Method 625 may be extended to include benzidine, benzochlorocyclopentadiene, 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.
- 625 Screening only.
- \*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.
- 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 6.2 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.



8 Method 1613 Revision A: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution, ERGOCHERMS, Environmental Protection Agency, Federal Register, page 5088, February 1991. Available from the Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.

9 "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

10 "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

TABLE D  
List of Approved Test Procedures for Pesticides<sup>1</sup>

Parameter (micrograms per liter)	Method	Swabber <sup>A</sup> EPA <sup>101</sup>	Methods	ASTM <sup>10</sup>	Other
1. Aldrin	GC GC/MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30
2. Aroclora	GC				Note 3, p. 83; Note 6, p. 366
3. Atrazine	TLC				Note 3, p. 94; Note 6, p. 316
4. Atrazine	GC				Note 3, p. 83; Note 6, p. 366
5. Atrazine	GC				Note 3, p. 83; Note 6, p. 366
6. Atrazine methyl	GC				Note 3, p. 25; Note 6, p. 351
7. Bifenthrin	TLC				Note 3, p. 104; Note 6, p. 364
8. $\alpha$ -BHC	GC GC/MS	608 5025	6630 B & C 6410 B	D3086-90	Note 3, p. 7
9. $\beta$ -BHC	GC	608	6630 C	D3086-90	
10. $\alpha$ -BHC	GC/MS	625	6410 B	D3086-90	
11. $\gamma$ -BHC (Lindane)	GC	608	6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30
12. Carbof	GC		6630 B	D3086-90	Note 3, p. 7
13. Carbaryl	TLC				Note 3, p. 94; Note 6, p. 366
14. Carbofenthothion	GC				Note 4, p. 30; Note 6, p. 373
15. Chlordane	GC	608 625	6630 B & C	D3086-90	Note 3, p. 7
16. Chlorophospham	GC/MS				Note 3, p. 104; Note 6, p. 364
17. 2,4-D	TLC				Note 3, p. 115; Note 4, p. 35
18. 4,4'-DDD	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
19. 4,4'-DDE	GC/MS	625	6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30
20. 4,4'-DDT	GC	608	6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30
21. Dacron-O	GC/MS	625	6630 B & C	D3086-90	Note 3, p. 25; Note 6, p. 351
22. Dacron-S	GC				Note 3, p. 25; Note 6, p. 351
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 30
24. Dieldrin	GC				Note 3, p. 115
25. Dieldrin	GC				Note 4, p. 30; Note 6, p. 373
26. Dieldrin	GC		6630 B & C	D3086-90	Note 3, p. 7
27. Dieldrin	GC				Note 3, p. 7; Note 4, p. 30
28. Dieldrin	GC	608	6630 B & C		

TABLE D (continued)  
List of Approved Test Procedures for Pesticides<sup>1</sup>

Parameter (microgram per liter)	Method	Standard <sup>2,3</sup> EPA <sup>4,5</sup>	Methods	ASTM <sup>6</sup>	Other
29. Dioxathion	GC/MS	625	6410 B		Note 4, p. 30; Note 6, p. 573.
30. Dithionon	GC			D3086-90	Note 3, p. 25; Note 6, p. 571.
31. Diuron	TLC				Note 3, p. 104; Note 6, p. 564.
32. Endosulfan I	GC	608	6630 B & C		Note 3, p. 7.
33. Endosulfan II	GC/MS	725	6410 B	D3086-90	Note 3, p. 7.
34. Endosulfan sulfate	GC/MS	725	6410 B		
35. Etofen	GC	608	6410 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
36. Etofen aldehyde	GC/MS	725	6410 B & C	D3086-90	
37. Ethion	GC/MS	625	6410		Note 4, p. 30; Note 6, p. 573.
38. Fenoxon-TCA	TLC	608	6630 B & C	D3086-90	Note 3, p. 104; Note 6, p. 564.
39. Fenoxon-TCA	TLC	608	6410 B		Note 3, p. 104; Note 6, p. 564.
40. Heptachlor	GC/MS	602	6430 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
41. Heptachlor epoxide	GC/MS	625	6410 B		Note 3, p. 7; Note 4, p. 30; Note 6 p.573.
42. Lufenon	GC				Note 4, p. 30; Note 6, p. 573.
43. Lufenon	TLC				Note 3, p. 104; Note 6, p. 564.
44. Malathion	GC		6630 C		Note 3, p. 25; Note 4, p. 30; Note 6, p.551
45. Methidathion	TLC			D3086-90	Note 3, p. 94; Note 6, p. 560.
46. Methoxychlor	GC		6630 B & C		Note 3, p. 7; Note 4, p. 30.
47. Methylmercaptan	TLC				Note 3, p. 94; Note 6, p. 560.
48. Mirex	GC		6630 B & C		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. 564.
50. Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. 564.
51. Nebuton	TLC				Note 3, p. 104; Note 6, p. 564.
52. Parathion methyl	GC		6630 C	D3086-90	Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		6630 B & C		Note 3, p. 25.
54. PCNB	GC		6630 B & C		Note 3, p. 7.
55. Permethrin	GC			D3086-90	Note 3, p. 83; Note 6, p. 568.
56. Permethrin	GC				Note 3, p. 83; Note 6, p. 568.
57. Permethrin	GC				Note 3, p. 83; Note 6, p. 568.
58. Propazine	GC				Note 3, p. 83; Note 6, p. 568.
59. Propazine	TLC				Note 3, p. 104; Note 6, p. 564.
60. Propazine	TLC				Note 3, p. 94; Note 6, p. 560.
61. Sebacinon	TLC				Note 3, p. 83; Note 6, p. 568.
62. Sulfuron	TLC				Note 3, p. 104; Note 6, p. 564.
63. Simazine	GC				Note 3, p. 83; Note 6, p. 568.
64. Sulfonac	GC		6630 B & C		Note 3, p. 7.
65. Sulfonac	TLC				Note 3, p. 104; Note 6, p. 564.
66. Sweep	TLC		6640 B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-T	GC		6640 B		Note 3, p. 115.
68. 2,4,5-TP (Silver)	GC				Note 3, p. 83; Note 6, p. 568.
69. Terbutylazine	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
70. Toxaphene	GC				

TABLE D (continued)  
List of Approved Test Procedures for Pesticides<sup>1</sup>

Parameter (micrograms per liter)	Method	Standard <sup>A</sup> EPA <sup>2,3</sup>	Methods	ASTM <sup>4</sup>	Other
70. Trifluralin	GCMS GC	625	6410 B 6630 B		Note 3, p. 7.

TABLE D NOTES

<sup>A</sup> "Standard Methods for the Examination of Water and Wastewater", 17th 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.

<sup>3</sup> 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.

<sup>1</sup> 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.

<sup>2</sup> 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 Pesticides". 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.

<sup>3</sup> "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, CERL, U.S. Environmental Protection Agency, 36 W. St. Clair, Cincinnati, Ohio 45268.

<sup>4</sup> "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.

<sup>5</sup> The method may be extended to include  $\alpha$ (alpha)-BHC,  $\delta$ (delta)-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

<sup>6</sup> "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.

<sup>7</sup> 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.

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
53. Residue, total	P/G	Cool, 4°C	7 days
54. Residue, Filterable	P/G	Cool, 4°C	7 days
55. Residue, Nontitratable (TSS)	P/G	Cool, 4°C	7 days
56. Residue, Settlingable	P/G	Cool, 4°C	48 hours
57. Residue, Volatile	P/G	Cool, 4°C	7 days
61. Silica	P	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 sodium hydroxide 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

  

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
13. 18-20, 22, 24-26, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days
6, 57, 90. Purgeable aromatic	G, Teflon-lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> HCl to pH<2 <sup>6</sup>	14 days
3, 4. Acrocin and acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , Adjust pH to 4-5 <sup>10</sup>	14 days
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> days after extraction.	7 days until extraction, 40 days after extraction. <sup>13</sup>
7, 38. Benzaldehydes <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, <sup>13</sup> 7 days until extraction, 40 days after extraction.
14, 17, 48, 50-52. Phthalate esters <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
72-74. Nitroaromatics <sup>11,14</sup>	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction.
76-82. PCBs <sup>11</sup> acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction.
54, 55, 65, 69. Nitroaromatics and isophorone <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> store in dark.	7 days until extraction, 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 38, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> store in dark.	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 75. Halocethers <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
29, 35-37, 60-63, 91. Chlorinated hydrocarbons <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction.
59e-59i, 62e-62i, 74h-74i, 82e-82i, 87, 87m. Chlorinated Dioxins and Furans.	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.

  

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
TABLE D - Pesticide Tests: 1-70. Pesticides <sup>11</sup>	P/G	HNO <sub>3</sub> to pH<2	6 months

  

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
TABLE E - Radiological Tests 1-5 Alpha, beta, and radium	G, Teflon-lined cap	Cool, 4°C, pH 5.9 <sup>15</sup>	7 days until extraction, 40 days after extraction.

TABLE F NOTES:

- <sup>1</sup> Polyethylene (P) or Glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic)
- <sup>2</sup> Sample preservation should be performed immediately upon sample collection. 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 spitting are completed.
- <sup>3</sup> When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 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<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 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).
- <sup>4</sup> 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. 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 219.06 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.
- <sup>5</sup> Should only be used in the presence of residual chlorine.
- <sup>6</sup> 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.
- <sup>7</sup> Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- <sup>8</sup> Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- <sup>9</sup> Samples receiving no pH adjustment must be analyzed within seven days of sampling.
- <sup>10</sup> 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.
- <sup>11</sup> 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 benzidiazine).
- <sup>12</sup> 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.
- <sup>13</sup> Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- <sup>14</sup> For the analysis of diphenylnitrosamine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- <sup>15</sup> 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<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

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 social services. Other laboratory test results, including effluent toxicity, submitted to the department under this chapter 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,
- (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; r. and recr. Register, June, 1986, No. 366, eff. 7-1-86; renum. from NR 219.07 r. and recr. Tables A through F, Register, November, 1992, No. 443, eff. 12-1-92; am. (intro.) eff. 7-1-93.