CR 85-184

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Carroll D. Besadny
Secretary

BOX 7921
MADISON, WISCONSIN 53707

STATE OF WISCONSIN)
) ss
DEPARTMENT OF NATURAL RESOURCES)

TO ALL TO WHOM THESE PRESENTS SHALL COME, GREETINGS:

I, Bruce B. Braun, Deputy Secretary of the Department of Natural Resources and custodian of the official records of said Department, do hereby certify that the annexed copy of Natural Resources Board Order No. TS-42-85 was duly approved and adopted by this Department on February 27, 1986. I further certify that said copy has been compared by me with the original on file in this Department and that the same is a true copy thereof, and of the whole of such original.

IN TESTIMONY WHEREOF, I have hereunto set my hand and affixed the official seal of the Department at General Executive Facility #2 in the City of Madison, this 16th day of April, 1986.

Bruce B. Braun

Bruce B. Braun, Deputy Secretary

(SEAL)

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ORDER OF THE STATE OF WISCONSIN NATURAL RESOURCES BOARD
REPEALING, RENUMBERING AND AMENDING, AMENDING,
REPEALING AND RECREATING, AND CREATING RULES

IN THE MATTER of repealing s. NR 219.06; renumbering and amending ss. NR 219.04 and 219.05; amending ss. NR 101.13(8), 219.01, and 219.02(1); repealing and recreating ss. NR 219.03 and 218.09; and creating s. NR 219.04 of the Wisconsin Administrative Code, pertaining to analytical and preservation procedures for effluent discharges from point sources.

TS-42-85

Analysis Prepared by the Department of Natural Resources

The effect of the amendments to ss. NR 101.13(8), NR 218.09, NR 219.01, NR 219.02(1), NR 219.04 and NR 219.05, and the creation of ss. NR 219.03 and NR 219.04 will be to delete out of date analytical references, cite more current analytical references, add analytical methodology not previously cited, and prescribe sample preservation procedures. The rules amended and created here will reflect the federal requirements which were revised in the October 26, 1985, federal register.

Pursuant to the authority vested in the State of Wisconsin Natural Resources Board by ss. 147.04(5) and 227.02(1)(e), Stats., the State of Wisconsin Natural Resources Board hereby repeals, renumbers and amends, amends, repeals and recreates, and creates rules interpreting s. 147.04(5), Stats., as follows:

SECTION 1. NR 101.13(8) is amended to read:

NR 101.13(8) ~~Methods of analyzing for pollutants shall be those set forth~~ Required pollutant analytical methodology and sample preservation procedures are in ~~Wis. Adm. Code chapter~~ ch. NR 219 unless, on request, an alternate or modified method or procedure has been approved previously in writing by the department. ~~Appropriate sample preservation and laboratory procedures shall be used to avoid sample deterioration and interference with prescribed analyses.~~

SECTION 2. NR 218.09 is repealed and recreated to read:

NR 218.09 STORAGE OF SAMPLES. Methods for preserving samples in storage prior to analysis and the limits on such storage are set forth in ch. NR 219.

SECTION 3. NR 219.01 (intro.), as amended in TS-16-85, is amended to read:

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 dischargers from point sources as authorized by ss. 147.04(5) and 144.95, Stats.

SECTION 4. NR 219.02(1) (intro.), as amended in TS-16-85, is amended to read:

NR 219.02 APPLICABILITY. (1) The procedures prescribed herein shall, except as provided in s. NR ~~219.05~~ 219.06, be used in the determination of concentrations and quantities of pollutant parameters as required for:

SECTION 5. NR 219.03 is repealed and recreated to read:

NR 219.03 DEFINITIONS. As used in this chapter:

(1) "Administrator" means the administrator of the U.S. environmental protection agency.

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

(3) "Director" means the director of the U.S. environmental protection agency, environmental monitoring and support laboratory, Cincinnati, Ohio 45268.

(4) "Regional administrator" means the regional administrator of region V, U.S. environmental protection agency.

SECTION 6. NR 219.04 and 219.05 are renumbered 219.05 and 219.06 respectively and amended to read:

NR 219.05 APPLICATION FOR ALTERNATE TEST PROCEDURES. (1) SPECIFIC DISCHARGES. Any person may apply to the regional administrator for approval of an alternate test procedure for a specific discharge. ~~Such application may be made in the following manner:~~ Any application for an alternate test procedure under this subsection shall be submitted to the regional administrator through the department. The application shall be made by letter in triplicate and shall:

~~(a) The applicant shall submit an application to the regional administrator through the department.~~

~~(b) The application for an alternate test procedure shall be made by letter in triplicate; and~~

~~1.~~ (a) Provide the name and address of the responsible person or firm making the discharge (if not the applicant), the number of the existing or pending permit, the name of the issuing agency, and the discharge serial number,

~~2.~~ (b) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested,

~~3.~~ (c) Provide justification for using testing procedures other than those specified in ~~ch. NR 219~~ this chapter, and

4. (d) Provide a detailed description of the proposed alternate test procedure, together with references to published studies on the applicability of the alternate test procedure to the effluents in question and the test data required in s. NR 149.12.

(2) NATIONWIDE USE. Any person may apply to the director~~, environmental monitoring and support laboratory, Cincinnati, Ohio 45268~~ for approval of an alternate test procedure for nationwide use. ~~Such application shall be made in the following manner:~~ Any application for an alternate test procedure under this subsection shall be made by letter in triplicate and shall:

~~(a) The application for an alternate procedure shall be made by letter, in triplicate, and~~

~~1.~~ (a) Provide the name and address of the responsible person or firm making the request,

~~2.~~ (b) Identify the ~~pollutant(s)~~ pollutants or ~~parameter(s)~~ parameters for which nationwide approval of an alternate testing procedure is being requested,

~~3.~~ (c) Provide a detailed description of the proposed alternate test procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the ~~pollutant(s)~~ pollutants or ~~parameter(s)~~ parameters in wastewater from representative or specified industrial or other categories, and

~~4.~~ (d) Provide comparability data for the performance of the proposed alternate test procedure to the approved test procedures.

NR 219.06 APPROVAL OF ALTERNATE TEST PROCEDURES. (1) SPECIFIC DISCHARGE. The regional administrator has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

~~(2)~~ (a) Within 30 days of receipt of an application, the department ~~will~~ shall forward ~~such~~ the application proposed by the responsible person or firm making the discharge, together with its recommendations, to the regional administrator. Where the ~~director~~ department recommends rejection of the application for scientific and technical reasons which the ~~director~~ department provides, the regional administrator shall deny the application.

~~(3)~~ (b) Within 90 days of the receipt of an application for an alternate test procedure proposed by the responsible person or firm making the discharge, the regional administrator ~~will~~ shall notify the applicant and the department agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure.

~~(4)~~ (2) NATIONWIDE USE. Within 90 days of the receipt by the director ~~of the environmental monitoring and support laboratory, Cincinnati,~~ of an application for an alternate test procedure for nationwide use, the director ~~of the environmental monitoring and support laboratory, Cincinnati,~~ shall notify the applicant of ~~his/her~~ the recommendation of the director to the administrator to approve or reject the application or

shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the administrator to satisfy the applicable requirements of this chapter shall be approved for nationwide use: ; alternate test procedures determined by the administrator not to meet the requirements of 40 CFR part 136 shall be rejected. Notice of these determinations shall be submitted for publication in the federal register not later than 15 days after such notification and determination is made.

SECTION 7. NR 219.04 is created to read:

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 effluent 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. 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.

SECTION 8. NR 219.06 is repealed.

TABLE A

LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and Units	Method[1]	EPA[2]	Standard[2A] Methods 15th Ed.	USGS[3]
Bacteria:				
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF)[4], single step.	p. 132 p. 124	908C 909C	B-0050-77
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or, MF[4], single step[4A].	p. 132 p. 124	908C 909C	
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF[4] single step or two step.	p. 114 p. 108 p. 132	908A 909A 908C	B-0025-77
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF[4] with enrichment.	p. 114 p. 111	908A 909 (A+A.5c)	
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF[4]; or, plate count	p. 139 p. 136 p. 143	910A 910B 910C	B-0055-77[5]

TABLE A Notes:

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

[2] "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. St. Claire, Cincinnati, Ohio 45268.

[2A] "Standard Methods for the Examination of Water and Wastewater", 15th 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, 1981. Available on inter-library loan.

[3] "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples", edited by P.E. Greeson, 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.

[4] 0.45 micrometer membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth and development.

[4A] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B-0055-77) is made in a boiling water bath to avoid scorching of the medium.

TABLE B
List of Approved Inorganic Test Procedures

	Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
1.	Acidity, as $CACO_3$, mg/L, Electrometric end point or phenolphthalein end point.	305.1	402(4.d)	D1067-70(E)		
2.	Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric:					
	Titration to pH 4.5, manual	310.1	403	D1067(B)	I-1030-78	P. 548[2]
	Or automated	310.2			I-2030-78	
3.	Aluminum - Total[3], mg/L:					
	Digestion[3] followed by:					
	AA direct aspiration	202.1	303C		I-3051-78	
	AA furnace	202.2	304			
	Inductively coupled plasma					Method 200.7[4]
	or colorimetric (Eriochrome cyanine R)		306B			
4.	Ammonia (as N), mg/L: Manual distillation[5] (at pH 9.5):					
	Followed by	350.2	417A			
	Nesslerization	350.2	417B	D1426-79(A)	I-3520-78	P. 553[2]
	Titration	350.2	417D			
	Electrode	350.3		D1426-79(D)		
	Automated phenate, or	350.1	417F	D1426-79(C)	I-4523-78	
	Automated electrode					([6])
5.	Antimony - Total[3], mg/L:					
	Digestion[3] followed by:					
	AA direct aspiration	204.1	303A			
	AA furnace, or	204.2	304			
	Inductively coupled plasma					Method 200.7[4]
6.	Arsenic - Total[3], mg/L:					
	Digestion[3] followed by	206.5				
	AA (gaseous hydride)	206.3	303E	D2972-78(B)	I3062-78	
	AA furnace	206.2	304			
	Inductively coupled plasma					Method 200.7[4]
	Or, colorimetric (SDDC)	206.4	307B	D2972-78(A)	I-3060-78	

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
7. Barium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	208.1	303C		I-3084-78	
AA furnace, or	208.2	304			
Inductively coupled plasma					Method 200.7[4]
8. Beryllium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	210.1	303C	D3645-78	I-3095-78	
AA furnace	210.2	304			
Inductively coupled plasma					Method 200.7[4]
Or, colorimetric (aluminum)		309B			
9. Biochemical oxygen demand (BOD_5), mg/L:					
Winkler (Azide modification)	405.1	507		I-1578-78	P. 17[8]
Or electrode method					P. 548[2]
10. Boron - Total, mg/L:					
Colorimatic (curcumin) or	212.3	404A		I-3112-78	
Inductively coupled plasma					Method 200.7[4]
11. Bromide, mg/L: Titrimetric	320.1		D1246-77(C)	I-1125-78	P.S44[25]
12. Cadmium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	213.1	303A or 303B	D3557-78 (A or B)	I-3135-78 or I-3136-78	Pg. 557[2]
AA furnace	213.2	304			P. 37[8]
Inductively coupled plasma					Method 200.7[4]
Voltametry[9] or			D3557-78(C)		
Colorimetric (Dithizone)		310B			

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
13. Calcium - Total[3], mg/L:					
Digestion[3] followed by:					
Atomic absorption	215.1	303A	D511-77(C)	I-3152-78	
Inductively coupled plasma					Method 200.7[4]
Or EDTA titration	215.2	311C	D511-77(B)		
14. Carbonaceous Biochemical oxygen demand ($CBOD_5$), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor.		507(5.e.6)			
15. Chemical oxygen demand (COD), mg/L:					
Titrimetric colorimetric					
(mid-level) or	410.1	508A	D1252-78	I-3560-78	P 550[2] and
(low-level)	410.2	508A (4.b)		I-3562-78	P 17[8] and
(High level) for saline	410.3			I-3561-78	([10])
Automated and manual colorimetric	410.4				
Spectrophotometric					([11])
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or		407A	D512-67(B)	I-1183-78	
Mercuric nitrate	325.3	407B	D512-67(A)	I-1184-78	P. 554[2]
Colorimetric (ferricyanide) manual or			D512-67(C)	I-1187-78	
automated	325.1 or 325.2	407D		I-2187-78	
17. Chlorine - Total residual, mg/L:					
Idometric titrimetric[12] back					
amperometric or	330.2	408B	D1253-76(A)		
starch-iodine end point	330.2	408B	D1253-76(B)		
DPD-FAS	330.4	408D			
Spectrophotometric, DPD; or	330.5	408E			
Electrode					([27])
18. Chromium VI dissolved, mg/L: 0.45 micron filtration with:					
Extraction and atomic absorption, or	218.4	303B		I-1232-78	
Colorimetric (Diphenylcarbazide)				I-1230-78	

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
19. Chromium - Total[3], mg/L: Digestion[3] (optional extraction)					
followed by:	218.3				
AA direct aspiration	218.1	303A or 303B	D1687-77(D)	I-3236-78	P. 557[2]
AA furnace	218.2	304			
Inductively coupled plasma					Method 200.7[4]
Or colorimetric (Diphenylcarbazide)		312A	D1687-77(A)		
20. Cobalt - Total[3], mg/L: Digestion[3] followed by:					
AA direct aspiration	219.1	303A or 303B	D3558-77 (A or B)	I-3240-78 or I-3239-78	P. 37[8]
AA furnace, or	219.2	304			
Inductively coupled plasma					Method. 200.7[4]
21. Color, Platinum Cobalt units or dominant wavelength hue, luminance, purity:					
Colorimetric, ADMI	110.1	204D			([13])
Platinum cobalt; or	110.2	204A		I-1250-78	
Spectrophotometric	110.3	204B			
22. Copper - Total[3], mg/L: Digestion[3] followed by:					
AA direct aspiration	220.1	303A or 303B	D1688-77	I-3271-78 or I-3270-78	P557[2] and P37[8]
AA furnace	220.2	304			
Inductively coupled plasma					Method 200.7[4]
Colorimetric (Neocuproine)		313B	D1688-77(A)		
Bicinchoninate					([14])
23. Cyanide - Total, mg/L:					
Manual distillation with $MgCl_2$	335.2	412B			
Followed by titrimetric	335.2	412C			P 22[8]
Manual or	335.2	412D	D2036-75(A)		
Automated[15] spectrophotometric	335.3		D2036-75(A)	I-3300-78	

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
24. Cyanide amenable to chlorination, mg/L: Manual distillation with $MgCl_2$ followed by titrimetric, manual or automated [15] spectrophotometric.	335.1	412F	D2036-75(B)		
25. Fluoride - Total, mg/L:					
Manual distillation[5]		413A			
Followed by manual or automated electrode	340.2	413B	D1179-72(B)	I-4327-78	
SPADNS	340.1	413C	D1179-72(A)		
Or automated complexone	340.3	413E			
26. Gold - Total[3] mg/L:					
Digestion[3] followed by:					
AA direct aspiration	231.1	303A			
Or AA furnace	231.2	304			
27. Hardness - Total as $CaCo_3$, mg/L:					
Automated colorimetric	130.1				
EDTA titration	130.2	314B	D1126-67(B)	I-1338-78	P.556[2]
Inductively coupled plasma					Method 200.7[4]
Or atomic absorption	215.1+	303A		I-3153-78+	
(sum of Ca and Mg as their respective carbonates)	242.1			I-3448-78	
28. Hydrogen ion (pH), pH units:					
Electrometric	150.1	423	D1293-78(A) or D1293-78(B)	I-1586-78	P. 547.[2]
Measurements; or automated electrode					([16])
29. Iridum - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	235.1	303A			
Or AA furnace	235.2	304			
30. Iron - Total[3], mg/L: Digestion[3]					
followed by:		303A or 303B	D1068-77		
AA direct aspiration	236.1	303B	(C or D)	I-3381-78	P. 557[2]
AA furnace	236.2	304			
Inductively coupled plasma					Method 200.7[4]
Or colorimetric (Phenanthroline)		315B	D1068-77(A)		([17])

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
31. Kjeldahl nitrogen - Total (as N), mg/L:					
Digestion and distillation	351.2	420A or B			P. 552[2]
Followed by titration	351.3	417D	D3590-77		
Nesslerization or	351.3	417B			
Electrode	351.3	417E			
Automated phenate	351.1			I-4551-78	
Semi-automated block digester	351.2			I-4552-78	
Or potentiometric	351.4				
32. Lead - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	239.1	303A or 303B	D3559-78(A or B)	I-3399-78	P. 557[2]
AA furnace	239.2	304			
Inductively coupled plasma					Method 200.7[4]
Voltametry[9] or			D3559-78(C)		
Colorimetric (Dithizone)		316B			
33. Magnesium - Total[3]; mg/L:					
Digestion[3] followed by:					
Atomic absorption	242.1	303A	D511-77(B)	I-3447-78	P. 557[2]
Inductively coupled plasma					Method 200.7[4]
Or gravimetric		318B	D511-77(A)		
34. Manganese - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	243.1	303A or 303B	D858-77	I-3454-78	P. 557[2]
AA furnace	243.2	304	(B or C)		
Inductively coupled plasma					Method 200.7[4]
Or colorimetric (Persulfate)		319B	D858-77(A)		P. 564[2]
Periodate					([18]) and P. 227.[28]
35. Mercury - Total[3], mg/L:					
Cold vapor, manual or	245.1	303F	D3223-79	I-3462-78	P. 559[2]
automated	245.2				
36. Molybdenum - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	246.1	303C		I-3490-78	
AA furnace, or	246.2	304			
Inductively coupled plasma					Method 200.7[4]

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
37. Nickel - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	249.1	303A or 303B	D1886-77 (C or D)		I-3499-78
AA furnace	249.2	304			
Inductively coupled plasma					Method 200.7[4]
Or colorimetric (Heptoxime)		321B			
38. Nitrate (as N), mg/L:					
Brucine sulfate, or	352.1		D092-71	I-1540-78	P. 554[2] and P. 427.[28]
Nitrate-nitrite N minus Nitrite N	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	P.28[8]
39. Nitrate-nitrite (as N), mg/L:					
Cadmium reduction, manual	353.3	418C	D3867-79(B)		
Or automated, or	353.2	418F	D3867-79(A)	I-4545-78	
automated hydrazine	353.1				
40. Nitrite (as N), mg/L:					
Spectrophotometric, manual or automated (Diazotization)	354.1	419	D1254-67	I-4540-78	(19)
41. Oil and grease-Total recoverable, mg/L:					
Gravimetric (extraction)	413.1	503A			
42. Organic carbon - Total (TOC), mg/L:					
Combustion or oxidation	415.1	505	D2579-78(A) or D2579-78(B)		P.551[2] and P.4[20]
43. Organic nitrogen (as N), mg/L:					
Total Kjeldahl N minus ammonia N	See parameters 31 and 4	420A	D3590-77-D1426-79(A)	See parameters 31 and 4	PP. 552-53[2]
44. Orthophosphate (as P), mg/L:					
Ascorbic acid method, automated	365.1	424G		I-4601-78	
Or manual single reagent or	365.2	424F	D515-78(A)		P.561[2]
Manual two reagent	365.3				

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
45. Osmium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration, or	252.1	303C			
AA furnace	252.2	304			
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification)	360.2	421B	D1589-60(A)	I-1575-78	P. 550[2]
Or electrode	360.1	421F		I-1576-78	
47. Palladium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	253.1				P. S27[25]
Or AA furnace	253.2				P. S28[25]
48. Phenols, mg/L:					
Manual distillation	420.1		D1783-70		(26)
Followed by manual	420.1		(A or B)		(26)
Or automated[15] colorimetric (4AAP)	420.2				
49. Phosphorus (elemental), mg/L:					
Gas-liquid chromatography					(21)
50. Phosphorus - Total, mg/L:					
Persulfate digestion	365.2	424C(III)			P. 561[2]
Followed by manual or	365.2 or 365.3	424F	D515-78(A)		
Automated ascorbic acid	365.1	424G		I-4600-78	
Reduction, or semi-automated block digestor	365.4			I-4603-78	
51. Platinum - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	255.1	303A			
Or AA furnace	255.2	304			
52. Potassium - Total[3], mg/L:					
Digestion[3] followed by:					
Atomic absorption	258.1	303A		I-3630-78	P. 560[2]
Inductively coupled plasma					Method 200.7[4]
Flame photometric, or		322B	D1428-64(A)		
Colorimetric (cobaltinitrite)					P. 235.[28]

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
53. Residue - total, mg/L: Gravimetric 103-105°C	160.3	209A		I-3750-78	
54. Residue - filterable, mg/L: Gravimetric, 180°C	160.1	209B		I-1750-78	
55. Residue - nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	209D		I-3765-78	
56. Residue - settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	209F			
57. Residue - volatile mg/L: Gravimetric, 550°C	160.4	209E		I-3753-78	
58. Rhodium - Total[3], mg/L: Digestion[3] followed by:					
AA direct aspiration	265.1	303A			
Or AA furnace	265.2	304			
59. Ruthenium - Total[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	267.1	303A			
Or AA furnace	267.2	304			
60. Selenium - Total[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	270.2	304			
Inductively coupled plasma					Method 200.7[4]
or AA (gaseous hydride)	270.3	303E	D3859-79	I-3667-78	
61. Silica - Dissolved, mg/L: 0.45 micron filtration:					
Followed by manual or automated colorimetric (Molybdosilicate), or	370.1	425C	D859-68(B)	I-1700-78 I-2700-78	
Inductively coupled plasma					Method 200.7[4]

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
62. Silver - Total[22], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	272.1	303A or 303B		I-3720-78	P. 557[2] and P. 37[8]
AA furnace	272.1	304			
Colorimetric (Dithizone), or		324B			
Inductively coupled plasma					Method 200.7[4]
63. Sodium - Total[3], mg/L:					
Digestion[3] followed by:					
Atomic absorption	273.1	303A		I-3735-78	P. 561[2]
Inductively coupled plasma					Method 200.7[4]
Or flame photometric		325B	D1428-64(A)		
64. Specific conductance, mhos/cm:					
Wheatstone bridge	120.1	205	D1125-77(A)	I-1780-78	P. 547[2]
65. Sulfate (as SO_2), mg/L:					
Automated colorimetric (barium chloroanilate)	375.1				
Gravimetric, or	375.3	426A or 426B	D516-68(A)		PP. 562-63[2]
Turbidimetric	375.4	426C	D516-68(B)		
66. Sulfide (as S), mg/L:					
Titrimetric (iodine) or	376.1	427D		I-3840-78	
Colorimetric (methylene blue)	376.2	427C			
67. Sulfite (as SO_4), mg/L:					
Titrimetric (iodine iodate)	377.1	428	D1339-78(C)		
68. Surfactants, mg/L: Colorimetric methylene blue)	425.1	512A	D2330-68(A)		
69. Temperature, °C: Thermometric	170.1	212			(23)

TABLE B
List of Approved Inorganic Test Procedures
(continued)

Parameter, Units & Methods	EPA 1979[A]	Standard Methods[B] 15th Ed.	ASTM[C]	USGS[1]	Other
70. Thallium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	279.1	303A			
AA furnace, or	279.2	304			
Inductively coupled plasma					Method 200.7[4]
71. Tin - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration or	282.1	303A		I-3850-78	
AA furnace	282.2	304			
72. Titanium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	283.1	303C			
AA furnace	283.2	304			
73. Turbidity, NTU: Nephelometric	180.1	214A	D1889-71	I-3860-78	
74. Vanadium - Total[3], mg/L:					
Digestion[3] followed by:					
AA direct aspiration	286.1	303C			
AA furnace	286.2	304			
Inductively coupled plasma					Method 200.7[4]
Or colorimetric (Gallic acid)		327B	D3373-75		
75. Zinc - Total[3], mg/L: Digestion[3] followed by:					
AA direct aspiration	289.1	303A or 303B	D1691-77(D)	I-3900-78	P. 557[2]
AA furnace	289.2	304	D1691-77(C)		P. 37[8]
Inductively coupled plasma					Method 200.7[4]
Colorimetric (Dithizone)		328C			
Or Colorimetric (Zincon)					([24])

TABLE B NOTES

[A] "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 United States Environmental Protection Agency, March 1979. Available from ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[B] "Standard Methods for the Examination of Water and Wastewater", 15th 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, 1981. For parameters 34, 38, 48 and 52, see 14th Edition, 1976. Available on inter-library loan.

[C] "Annual Book of Standards, Part 31, Water", American Society for Testing and Materials, 1980, 1916 Race Street, Philadelphia, PA 19103. The analytical standards that appear in the newer editions are allowable if the standard number is identical to the one cited in the table.

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 78-679, or "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", N.W. Skougstad, et al., U.S. Geological Survey, Techniques of Water Resources Investigation, Book 5, Chapter A1, 1979. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[2] "Official Methods of Analysis of the Association of Official Analytical Chemists," 13th Edition (1980), The Association of Official Analytical Chemists, 1111 N. 19th Street, Suite 210, Arlington, VA 22209. Available on inter-library loan.

[3] For the determination of total metals the samples are 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 hydrocholoric 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 colormetric 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 titanium 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 procedure for direct aspiration or graphite furnace atomic absorption analysis included in one of the other approved references is different than the above, the EPA procedure must 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 must be followed. Sample digestion of the filtrate for dissolved metals, or digestion of the original sample solution for total metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample has a low COD and the filtrate meets the following criteria:

(a) Is visibly transparent
(b) Has no perceptible odor, and
(c) Is free of particulate or suspended matter following acidification.

TABLE B NOTES
(Continued)

[4] 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. 20402.

[5] 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.

[6] Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon Auto-Analyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, New York 10591.

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

[8] "American National Standard on Photographic Processing Effluents", April 2, 1975. Available from American National Standards Institute, 1430 Broadway, New York, New York 10018.

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

[10] Chemical Oxygen Demand Method 8000, Hach Handbook of Water Analysis 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

[11] OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

[12] The back titration method will be used.

[13] "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, N.Y. 10016.

[14] Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis 1979. Published by Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

[15] 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, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

TABLE B NOTES
(Continued)

[16] Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976. Technicon Auto-Analyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, New York 10591.

[17] I, 10-Phenathroline Method for Iron, Hach Method 8008, 1980. Published by Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

[18] Periodate Oxidation Method for Manganese, Method 8034. Hatch Handbook for Water Analysis, 1979. Published by Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

[19] Nitrite Nitrogen, Hach Method 8507. Published by Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

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

[21] "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 Center, 52 Vanderbilt Avenue, New York, NY 10164.

[22] Recommended 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 recommended method is satisfactory.

[23] "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.

[24] Zincon Method for Zinc Method 8009. Hatch Handbook for Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80539.

[25] "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. 20005.

[26] 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.

TABLE B NOTES
(Continued)

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

[28] 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.

TABLE C
List of Approved Test Procedures for Non-Pesticide Organic Compounds
EPA METHOD NUMBER [2,7]

	Parameter[1]	GC	GC/MS	HPLC	Other
1.	Acenaphthene	610	625, 1625	610	
2.	Acenaphthylene	610	625, 1625	610	
3.	Acrolein	603	[4]624, 1624		
4.	Acrylonitrile	603	[4]624, 1624		
5.	Anthracene	610	625, 1625	610	
6.	Benzene	602	624, 1624		
7.	Benzidine		[5]625, 1625	605	Note 3, p. 1
8.	Benzo(a)anthracene	610	625, 1625	610	
9.	Benzo(a)pyrene	610	625, 1625	610	
10.	Benzo(b)fluoranthene	610	625, 1625	610	
11.	Benzo(g,h,i)perylene	610	625, 1625	610	
12.	Benzo(k)fluoranthene	610	625, 1625	610	
13.	Benzyl chloride				Note 3, p. 130; Note 6, p. S102
14.	Benzyl butyl phthalate	606	625, 1625		
15.	Bis(2-chloroethoxy) methane	611	625, 1625		
16.	Bis(2-chloroethyl)ether	611	625, 1625		
17.	Bis(2-ethylhexyl)phthalate	606	625, 1625		
18.	Bromodichloromethane	601	624, 1624		
19.	Bromoform	601	624, 1624		
20.	Bromomethane	601	624, 1624		
21.	4-Bromophenylphenyl ether	611	625, 1625		
22.	Carbon tetrachloride	601	624, 1624		Note 3, p. 130
23.	4-Chloro-3-methylphenol	604	625, 1625		
24.	Chlorobenzene	601, 602	624, 1624		Note 3, p. 130
25.	Chloroethane	601	624, 1624		
26.	2-Chloroethylvinyl ether	601	624, 1624		
27.	Chloroform	601	624, 1624		Note 3, p. 130
28.	Chloromethane	601	624, 1624		
29.	2-Chloronaphthalene	612	625, 1625		
30.	2-Chlorophenol	604	625, 1625		
31.	4-Chlorophenylphenyl ether	611	625, 1625		
32.	Chrysene	610	625, 1625	610	
33.	Dibenzo(a,h)anthracene	610	625, 1625	610	
34.	Dibromochloromethane	601	624, 1624		
35.	1,2-Dichlorobenzene	601, 602, 612		624, 625, 1625	
36.	1,3-Dichlorobenzene	601, 602, 612		624, 625, 1625	
37.	1,4-Dichlorobenzene	601, 602, 612		625, 1624, 1625	
38.	3,3-Dichlorobenzidine		625, 1625	605	
39.	Dichlorodifluoromethane	601			
40.	1,1-Dichloroethane	601	624, 1624		
41.	1,2-Dichloroethane	601	624, 1624		
42.	1,1-Dichloroethene	601	624, 1624		
43.	trans-1,2-Dichloroethene	601	624, 1624		

TABLE C
List of Approved Test Procedures for Non-Pesticide Organic Compounds
EPA METHOD NUMBER [2,7]
(Continued)

	Parameter[1]	GC	GC/MS	HPLC	Other
44.	2,4-Dichlorophenol	604	625, 1625		
45.	1,2-Dichloropropane	601	624, 1624		
46.	cis-1,3 Dichloropropene	601	624, 1624		
47.	trans-1,3-Dichloropropene	601	624, 1624		
48.	Diethyl phthalate	606	625, 1625		
49.	2,4-Dimethlyphenol	604	625, 1625		
50.	Dimethyl phthalate	606	625, 1625		
51.	Di-n-butyl phthalate	606	625, 1625		
52.	Di-n-octyl phthalate	606	625, 1625		
53.	2,4-Dinitrophenol	604	625, 1625		
54.	2,4-Dinitrotoluene	609	625, 1625		
55.	2,6-Dinitrotoluene	609	625, 1625		
56.	Epichlorohydrin				Note 3. p. 130; Note 6, p. S102
57.	Ethylbenzene	602	624, 1624		
58.	Fluoranthene	610	625, 1625	610	
59.	Fluorene	610	625, 1625	610	
60.	Hexachlorobenzene	612	625, 1625		
61.	Hexachlorobutadiene	612	625, 1625		
62.	Hexachlorocyclopentadiene	612	[5]625, 1625		
63.	Hexachloroethane	612	625, 1625		
64.	Ideno (1,2-3-cd)pyrene	610	625, 1625	610	
65.	Isophorone	609	625, 1625		
66.	Methylene chloride	601	624, 1624		Note 3, p. 130
67.	2-Methyl-4,6-dinitrophenol	604	625, 1625		
68.	Naphthalene	610	625, 1625	610	
69.	Nitrobenzene	609	625, 1625		
70.	2-Nitrophenol	604	625, 1625		
71.	4-Nitrophenol	604	625, 1625		
72.	N-Nitrosodimethylamine	607	[5]625, 1625		
73.	N-Nitrosodi-n-propylamine	607	625, 1625		
74.	N-Nitrosodiphenylamine	607	[5]625, 1625		
75.	2,2-Oxybis (1-chloropropane)	611	625, 1625		
76.	PCB-1016	608	625		Note 3, p. 43
77.	PCB-1221	608	625		Note 3, p. 43
78.	PCB-1232	608	625		Note 3. p. 43
79.	PCB-1242	608	625		Note 3, p. 43
80.	PCB-1248	608	625		Note 3, p. 43
81.	PCB-1254	608	625		Note 3, p. 43
82.	PCB-1260	608	625		Note 3. p. 43
83.	Pentachlorophenol	604	625, 1625		Note 3. p. 140
84.	Phenanthrene	610	625, 1625	610	

TABLE C

List of Approved Test Procedures for Non-Pesticide Organic Compounds

EPA METHOD NUMBER [2,7]

(Continued)

	Parameter[1]	GC	GC/MS	HPLC	Other
85.	Phenol	604	625, 1625		
86.	Pyrene	610	625, 1625	610	
87.	2,3,7,8-Tetrachlorodibenzo-p-dioxin		[5a]613		
88.	1,1,2,2-Tetrachloroethane	601	624, 1624		Note 3, p. 130
89.	Tetrachloroethene	601	624, 1624		Note 3, p. 130
90.	Toluene	602	624, 1624		
91.	1,2,4-Trichlorobenzene	612	625, 1625		Note 3, p. 130
92.	1,1,1-Trichloroethane	601	624, 1624		
93.	1,1,2-Trichloroethane	601	624, 1624		Note 3, p. 130
94.	Trichloroethene	601	624, 1624		
95.	Trichlorofluoromethane	601	624		
96.	2,4,6-Trichlorophenol	604	625, 1625		
97.	Vinyl chloride	601	624, 1624		

TABLE C NOTES

[1] All parameters are expressed in micrograms per liter (ug/L).

[2] The full text of Methods 601-613, 624, 625, 1624, and 1625, are given in Appendix A of the Federal Register, October 26, 1984 (Part VIII, 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 the Federal Register, October 26, 1984 (Part VIII, 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," 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.

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

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethyamine, 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.

[5a] 625 Screening only.

[6] "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.

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1624, and 1625, (see Appendix A of the Federal Register, October 26, 1984, 40 CFR part 136) 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.

TABLE D
List of Approved Test Procedures for Pesticides[1]

Parameter (micrograms per liter)	Method	EPA[2,7]	Standard[2A] Methods 15th Ed.	ASTM[2B]	Other
1. Aldrin	GC	608	509A	D3086	Note 3, p. 7; Note 4, p.30
	GC/MS	625			
2. Ametryn	GC				Note 3, p. 83; Note 6, p. S68.
3. Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4. Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC				Note 3. p. 83; Note 6, p. S68.
6. Azinphos methyl	GC				Note 3. p. 25; Note 6, p. S51.
7. Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8. a(alpha)-BHC	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[5]625			
9. b(beta)-BHC	GC	608		D3086	
	GC/MS	625			
10. d(delta)-BHC	GC	608		D3086	
	GC/MS	[5]625			
11. g(gamma)-BHC (Lindane)	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30
	GC/MS	625			
12. Captain	GC		509A		Note 3. p. 7.
13. Carbaryl	TLC				Note 3. p. 94; Note 6. p. S60.
14. Carbophenothion	GC				Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC	608	509A	D3086	Note 3, p. 7
	GC/MS	625			
16. Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		509B		Note 3, p. 115; Note 4, p. 35.
18. 4,4'-DDD	GC	608	509A	D3086	Note 3. p. 7; Note 4, p. 30.
	GC/MS	625			
19. 4,4'-DDE	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
20. 4,4'-DDT	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30
	GC/MS	625			
21. Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC				Note 3, p. 25; Note 6, p. S51.
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6 p. S51
24. Dicamba	GC				Note 3, p. 115.
25. Dichlofenthion	GC				Note 4, p. 30; Note 6, p. S73.
26. Dichloran	GC		509A		Note 3, p. 7.
27. Dicofol	GC			D3086	
28. Dieldrin	GC	608	509A		Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
29. Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30. Disulfoton	GC				Note 3, p. 25; Note 6, p. S51.
31. Diuron	TLC				Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[5]625			

TABLE D
List of Approved Test Procedures for Pesticides[1]
(Continued)

Parameter (micrograms per liter)	Method	EPA[2,7]	Standard[2A] Methods 15th Ed.	ASTM[2B]	Other
33. Endosulfan II	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[5]625			
34. Endosulfan sulfate	GC	608			
	GC/MS	625			
35. Endrin	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	[5]625			
36. Endrin aldehyde	GC	608			
	GC/MS	625			
37. Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38. Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39. Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30
	GC/MS	625			
41. Heptachlor epoxide	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30; Note 6 p.S73
	GC/MS	625			
42. Isodrin	GC				Note 4, p. 30; Note 6, p. S73.
43. Linuron	TLC				Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		509A		Note 3, p. 25; Note 4, p.30; Note 6, p.S51
45. Methiocarb	TLC				Note 3, p. 94; Note 6, p. S60.
46. Methoxychlor	GC		509A	D3086	Note 3, p. 7; Note 4, p. 30.
47. Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S60.
48. Mirex	GC		509A		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64
50. Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
51. Neburon	TLC				Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		509A		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		509A		Note 3, p. 25.
54. PCNB	GC		509A		Note 3, p. 7.
55. Perthane	GC			D3086	
56. Prometron	GC				Note 3, p. 83; Note 6, p. S68.
57. Prometron	GC				Note 3, p. 83; Note 6, p. S68.
58. Propazine	GC				Note 3, p. 83; Note 6, p. S68.
59. Propham	TLC				Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC				Note 3, p. 94; Note 6, p. S60.
61. Secbumeton	TLC				Note 3, p. 83; Note 6, p. S68.
62. Siduron	TLC				Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC				Note 3, p. 83; Note 6, p. S68.
64. Strobane	GC		509A		Note 3, p. 7.
65. Swep	TLC				Note 3, p. 104; Note 6, p. S64.
66. 2,4,5-T	GC		509B		Note 3, p. 115; Note 4, p. 35.

TABLE D
List of Approved Test Procedures for Pesticides[1]
(Continued)

Parameter (micrograms per liter)	Method	EPA[2,7]	Standard[2A] Methods 15th Ed.	ASTM[2B]	Other
67. 2,4,5-TP (Silvex)	GC		509B		Note 3, p. 115.
68. Terbuthylazine	GC				Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30
	GC/MS	625			
70. Trifluralin	GC		509A		Note 3, p. 7.

TABLE D NOTES

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

[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 the Federal Register, October 26, 1984 (Part VIII, 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.

[2A] "Standard Methods for the Examination of Water and Wastewater", 15th Edition, Joint Editorial Board, American Public Heath Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1981. Available on inter-library loan.

[2B] "Annual Book of Standards, Part 31, Water", American Society for Testing and Materials, 1980. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[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", by D.F. Goerlitz and Eugene Brown: USGS-TWRI, Book 5, Chapter A3, 1972. 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 Federal Register, October 26, 1984, 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.

TABLE E

List of Approved Radiological Test Procedures

Parameter and units	Methods	EPA[1]	Standard[1A] Methods 15th Ed.	ASTM[1B]	USGS[2]
1. Alpha-Total, p^{Ci} per liter	Proportional or scintillation counter	900.0	703	D1943-66	pp. 75 and 78[3]
2. Alpha-Counting error, p^{Ci} per liter	Proportional or scintillation counter	Note 4.	703	D1943-66	p. 79
3. Alpha-Counting error, p^{Ci} per liter	Proportional counter	900.0	703	D1890-66	pp. 75 and 78[3]
4. Beta-Counting error, p^{Ci} per liter	Proportional counter	Note 4.	703	D1890-66	p. 79
5. (a) Radium-Total, p^{Ci} per liter	Proportional counter	903.0	705	D2460-70	
(b) ^{226}Ra, p^{Ci} per liter	Scintillation counter	903.1	706	D3454-79	p. 81

TABLE E NOTES

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 Update), United States Environmental Protection Agency, 1980. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[1A] "Standard Methods for the Examination of Water and Wastewater" 15th 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, 1981. Available on inter-library loan.

[1B] "Annual Book of Standards, Part 31, Water", American Society for Testing and Materials, 1980, 1916 Race Street, Philadelphia, PA 19103. The analytical standards that appear in newer editions are allowable if the standard number is identical to the one cited in the table.

[2] "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters", by M.J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76-177 (1976). Available from: U.S. Geological Survey, 604 S. Pickett St., Alexandria, VA 22304.

[3] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total".

[4] See Appendix B 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. 20402.

Note: Copies of the publications referred to in footnotes of the tables under sub. (1) are available for inspection at the offices of the department of natural resources, the secretary of state, the revisor of statutes and the federal register information center, room 8301, 1110 L street, N.W., Washington, D.C., 20408. Sources of the publications are identified in the footnotes.

TABLE F
Required Containers, Preservation Techniques, and Holding Times

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
Table A - Bacterial Tests:			
1-4. Coliform, fecal and total	P, G	Cool, 4°C, 0.008%, $Na_2S_2O_3$ [5]	6 hours
5. Fecal streptococci	P, G	Cool, 4°C, 0.008%, $Na_2S_2O_3$ [5]	6 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 organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Metals[7]:			
18. Chromium VI	P, G	Cool, 4°C	24 hours
35. Mercury	P, G	HNO_3 to pH 2	28 days
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals except chromium VI and mercury	P, G	HNO_3 to pH 2	6 months
38. Nitrate	P, G	Cool, 4°C	48 hours
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH 2	28 days
40. Nitrite	P, G	Cool, 4°C	48 hours
41. Oil and grease	G	Cool, 4°C, H_2SO_4 to pH 2	28 days
42. Organic carbon	P, G	Cool, 4°C, HCl or H_2SO_4 to pH 2	28 days
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately

TABLE F
Required Containers, Preservation Techniques, and Holding Times
(continued)

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
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	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
Table C - Organic Tests[8]			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons.	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	14 days
6, 57, 90. Purgeable aromatic	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5], HCl to pH 2[9]	14 days
3, 4. 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
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction. 40 days after extraction.
7, 38. Benzidines[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction[13]

TABLE F

Required Containers, Preservation Techniques, and Holding Times

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
14, 17, 48, 50-52. Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
72-74. 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.
76-82. PCBs[11] acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
54, 55, 65, 69. Nitroaromatics and isophorone [11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5] store in dark	7 days until extraction; 40 days after extraction
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons.[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5] store in dark	7 days until extraction; 40 days after extraction.
15, 16, 21, 31, 75. Haloethers[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction; 40 days after extraction.
29, 35-37, 60-63, 91. Chlorinated hydrocarbons[11]	G, Teflon-lined cap	Cool 4°C	7 days until extraction; 40 days after extraction.
87. TCDD[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction; 40 days after extraction.
TABLE D - 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.

TABLE F

Required Containers, Preservation Techniques, and Holding Times

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
TABLE E - Radiological Tests			
1-5 Alpha, beta, and radium	P, G	HNO_3 to pH 2	6 months

TABLE F NOTES

[1] Polyethylene (P) or Glass (G).

[2] 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 splitting are completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, 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% be weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods 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. 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] Sample 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 preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

TABLE F NOTES
(Continued)

[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% $Na_2S_2O_3$.

The foregoing rules were approved and adopted by the State of Wisconsin Natural Resources Board on February 27, 1986.

The rules contained herein shall take effect as provided in s. 227.026(1)(intro.), Stats.

Dated at Madison, Wisconsin April 16, 1986

STATE OF WISCONSIN DEPARTMENT OF NATURAL RESOURCES

By Carroll D. Besadny
Carroll D. Besadny, Secretary

(SEAL)

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