

SUBCHAPTER D—WATER PROGRAMS (CONTINUED)

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.

136.1 Applicability.

136.2 Definitions.

136.3 Identification of test procedures.

136.4 Application for alternate test procedures.

136.5 Approval of alternate test procedures.

APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95-217, 91 Stat. 1566, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40, and,

(b) Reports required to be submitted by discharges under the NPDES established by parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the CWA, as amended.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.2 Definitions.

As used in this part, the term:

(a) *Act* means the Clean Water Act of 1977, Pub. L. 95-217, 91 Stat. 1566, et seq. (33 U.S.C. 1251 et seq.) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at appendix B of this part.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, IE, and IF. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, IE, and IF. The references and the sources which are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the FEDERAL REGISTER. The discharge parameter values for which reports are required must be determined by one of

§ 136.3

the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, IE, and IF, or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§136.4 and 136.5. Under certain circumstances (paragraph (b) or (c) of this section or 40 CFR 401.13) other test procedures

40 CFR Ch. I (7-1-02 Edition)

may be used that may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method ¹	EPA	Standard methods, 18th Ed.	ASTM	USGS
Bacteria:					
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube 3 dilution, or Membrane filter (MF) ² , single step	p. 132 ³ p. 124 ³	9221C E ⁴ 9222D ⁴	B-0050-85 ⁵
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or MF, single step ⁶	p. 132 ³ p. 124 ³	9221C E ⁴ 9222D ⁴	
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or MF ² single step or two step	p. 114 ³ p. 108 ³	9221B ⁴ 9222B ⁴	B-0025-85 ⁵
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or MF ² with enrichment	p. 114 ³ p. 111 ³	9221B ⁴ 9222(B+B.5c) ⁴	
5. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution MF ² , or Plate count	p. 139 ³ p. 136 ³ p. 143 ³	9230B ⁴ 9230C ⁴	B-0055-85 ⁵
Aquatic Toxicity:					
6. Toxicity, acute, fresh water organisms, LC50, percent effluent.	Daphnia, Ceriodaphnia, Fathead Minnow, Rainbow Trout, Brook Trout, or Bannerfish Shiner mortality	Sec. 9 ⁷		
7. Toxicity, acute, estuarine and marine organisms, LC50, percent effluent.	Mysid, Sheepshead Minnow, or Menidia spp. mortality	Sec. 9 ⁷		
8. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent.	Fathead minnow larval survival and growth	1000.0 ⁸		
	Fathead minnow embryo-larval survival and teratogenicity	1001.0 ⁸		
	Ceriodaphnia survival and reproduction	1002.0 ⁸		
	Selenastrum growth	1003.0 ⁸		
9. Toxicity, chronic, estuarine and marine organisms, NOEC or IC25, percent effluent.	Sheepshead minnow larval survival and growth	1004.0 ⁹		
	Sheepshead minnow embryo-larval survival and teratogenicity	1005.0 ⁹		
	Menidia beryllina larval and growth	1006.0 ⁹		
	Mysidopsis bahia survival, growth, and fecundity	1007.0 ⁹		
	Arbacia punctulata fertilization	1008.0 ⁹		
	Champia parvula reproduction	1009.0 ⁹		

Notes to Table IA:

¹The method must be specified when results are reported.

²A 0.45 um 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.

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

⁴APHA. 1992. Standard Methods for the Examination of Water and Wastewater. American Public Health Association. 18th Edition. Amer. Publ. Hlth. Assoc., Washington, DC.

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

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

⁷USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. August 1993, EPA/600/4-90/027F.

⁸USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency USEPA. 1994. Cincinnati, Ohio (July 1994, EPA/600/4-91/002).
⁹Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (July 1994, EPA/600/4-91/003). These methods do not apply to marine waters of the Pacific Ocean.

§ 136.3

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and method	Reference (method number or page)				
	EPA ^{1,3,5}	STD methods 18th ed.	ASTM	USGS ²	Other
1. Acidity, as CaCO ₃ , mg/L: Electrometric endpoint or phenolphthalein endpoint	305.1	2310 B(4a)	D1067-92		
2. Alkalinity, as CaCO ₃ , mg/L: Electrometric or Colorimetric titration to pH 4.5, manual or automated.	310.1 310.2	2320 B	D1067-92	I-1030-85 I-2030-85	973.43. ³
3. Aluminum—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	202.1	3111 D		I-3051-85	
AA furnace	202.2	3113 B			
Inductively Coupled Plasma/Atomic Emission Spec- trometry (ICP/AES) ³⁶	⁵ 200.7	3120 B			
Direct Current Plasma (DCP) ³⁶			D4190-82(88)		Note 34.
Colorimetric (Eriochrome cyanine R)		3500-AI D			
4. Ammonia (as N), mg/L:					
Manual, distillation (at pH 9.5), ⁶ followed by	350.2	4500-NH ₃ B			973.49. ³
Nesslerization	350.2	4500-NH ₃ C	D1426-93(A)	I-3520-85	973.49. ³
Titration	350.2	4500-NH ₃ E			
Electrode	350.3	4500-NH ₃ F or G	D1426-93(B)		
Automated phenate, or	350.1	4500-NH ₃ H		I-4523-85	
Automated electrode					Note 7.
5. Antimony—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	204.1	3111 B			
AA furnace	204.2	3113 B			
ICP/AES ³⁶	⁵ 200.7	3120 B			
6. Arsenic—Total, ⁴ mg/L:					
Digestion ⁴ followed by	206.5				
AA gaseous hydride	206.3	3114 B 4.d	D2972-93(B)	I-3062-85	
AA furnace	206.2	3113 B	D2972-93(C)		
ICP/AES, ³⁶ or	⁵ 200.7	3120 B			
Colorimetric (SDDC)	206.4	3500-As C	D2972-93(A)	I-3060-85	
7. Barium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	208.1	3111 D		I-3084-85	
AA furnace	208.2	3113 B	D4382-91		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶					Note 34.
8. Beryllium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	210.1	3111 D	D3645-93(88)(A)	I-3095-85	
AA furnace	210.2	3113 B	D3645-93(88)(B)		
ICP/AES	⁵ 200.7	3120 B			
DCP, or			D4190-82(88)		Note 34.

40 CFR Ch. I (7-1-02 Edition)

	Colorimetric (aluminon)	3500-Be D			
9.	Biochemical oxygen demand (BOD ₅), mg/L:				
	Dissolved Oxygen Depletion	405.1	5210 B	I-1578-78 ⁸	973.44, ³ p. 17. ⁹
10.	Boron ³⁷ —Total, mg/L:				
	Colorimetric (curcumin)	212.3	4500-B B	I-3112-85	
	ICP/AES, or	⁵ 200.7	3120 B		
	DCP		D4190-82(88)		Note 34
11.	Bromide, mg/L:				
	Titrimetric	320.1	D1246-82(88)(C)	I-1125-85	p. S44. ¹⁰
12.	Cadmium—Total, ⁴ mg/L; Digestion ⁴ followed by:				
	AA direct aspiration ³⁶	213.1	3111 B or C	D3557-90(A or B)	I-3135-85 or I-3136-85 ..
	AA furnace	213.2	3113 B	D3557-90(D)	974.27, ³ p. 37. ⁹
	ICP/AES ³⁶	⁵ 200.7	3120 B	I-1472-85	
	DCP ³⁶		D4190-82(88)		Note 34.
	Voltametry, ¹¹ or		D3557-90(C)		
	Colorimetric (Dithizone)		3500-Cd D		
13.	Calcium—Total, ⁴ mg/L; Digestion ⁴ followed by:				
	AA direct aspiration	215.1	3111 B	D511-93(B)	I-3152-85
	ICP/AES	⁵ 200.7	3120 B		
	DCP, or				Note 34.
	Titrimetric (EDTA)	215.2	3500-Ca D	D511-93(A)	
14.	Carbonaceous biochemical oxygen demand (CBOD ₅), mg/L ¹² :				
	Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B		
15.	Chemical oxygen demand (COD), mg/L; Titrimetric, or.	410.1	5220 C	D1252-88(A)	I-3560-85
		410.2			I-3562-85
		410.3			
	Spectrophotometric, manual or automated	410.4	5220 D	D1252-88(B)	I-3561-85
					Notes 13 or 14.
16.	Chloride, mg/L:				
	Titrimetric (silver nitrate) or		4500-Cl ⁻ B	D512-89(B)	I-1183-85
	(Mercuric nitrate)	325.3	4500-Cl ⁻ C	D512-89(A)	I-1184-85
	Colorimetric, manual or				I-1187-85
	Automated (Ferricyanide)	325.1 or 325.2	4500-Cl ⁻ E		I-2187-85
17.	Chlorine—Total residual, mg/L; Titrimetric:				
	Amperometric direct	330.1	4500-Cl D	D1253-86(92)	
	Iodometric direct	330.3	4500-Cl B		
	Back titration ether end- point ¹⁵ or	330.2	4500-Cl C		
	DPD-FAS	330.4	4500-Cl F		
	Spectrophotometric, DPD	330.5	4500-Cl G		
	Or Electrode				Note 16.
18.	Chromium VI dissolved, mg/L; 0.45 micron filtration followed by:				
	AA chelation-extraction or	218.4	3111 C		I-1232-85
	Colorimetric (Diphenylcarbazide)		3500-Cr D	D1687-92(A)	I-1230-85
19.	Chromium—Total, ⁴ mg/L; Digestion ⁴ followed by:				
	AA direct aspiration ³⁶	218.1	3111 B	D1687-92(B)	I-3236-85
	AA chelation-extraction	218.3	3111 C		974.27, ³
	AA furnace	218.2	3113 B	D1687-92(C)	

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA 1.35	STD methods 18th ed.	ASTM	USGS ²	Other
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP, ³⁶ or		3500—Cr D	D4190—82(88)		Note 34.
Colorimetric (Diphenylcarbazide)					
20. Cobalt—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	219.1	3111 B or C	D3558—90(A or B)	I—3239—85	p. 37. ⁹
AA furnace	219.2	3113 B	D3558—90(C)		
ICP/AES	⁵ 200.7	3120 B			
DCP			D4190—82(88)		Note 34.
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:					
Colorimetric (ADMI), or	110.1	2120 E			Note 18.
(Platinum cobalt), or	110.2	2120 B		I—1250—85	
Spectrophotometric	110.3	2120 C			
22. Copper—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	220.1	3111 B or C	D1688—90(A or B)	I—3270—85 or I3271—85 ...	974.27 ³ p. 37. ⁹
AA furnace	220.2	3113 B	D1688—90(C)		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶ or			D4190—82(88)		Note 34.
Colorimetric (Neocuproine) or		3500—Cu D			Note 19.
(Bicinchoninate)		Or E			
23. Cyanide—Total, mg/L:					
Manual distillation with MgCl ₂ followed by		4500—CN C	D2036—91(A)		
Titrimetric, or		4500—CN D			p. 22. ⁹
Spectrophotometric, manual or	³¹ 335.2	4500—CN E	D2036—91(A)	I—3300—85	
Automated ²⁰	³¹ 335.3				
24. Available Cyanide, mg/L					
Cyanide amenable to chlorination (CATC), Manual distillation with MgCl ₂ followed by titrimetry or spectrophotometry.	335.1	4500—CN G	D2036—91(B).		
Flow injection and ligand exchange, followed by amperometry.					⁴⁴ OIA—1677
25. Fluoride—Total, mg/L:					
Manual distillation ⁶ followed by		4500—F B			
Electrode, manual or	340.2	4500—F C	D1179—93(B)		
Automated				I—4327—85	
Colorimetric (SPADNS)	340.1	4500—F D	D1179—93(A)		
Or Automated complexone	340.3	4500—F E			
26. Gold—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	231.1	3111 B			
AA furnace, or	231.2				
DCP					Note 34.
27. Hardness—Total, as CaCO ₃ , mg/L					
Automated colorimetric,	130.1				

	Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).	130.2	2340 B or C	D1126-86(92)	I-1338-85	973.52B. ³
28.	Hydrogen ion (pH), pH units					
	Electrometric measurement, or Automated electrode	150.1	4500-H ⁺ B	D1293-84(90)(A or B)	I-1586-85	973.41. ³ Note 21.
29.	Iridium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
	AA direct aspiration or AA furnace	235.1 235.2	3111 B			
30.	Iron—Total, ⁴ mg/L; Digestion ⁴ followed by:					
	AA direct aspiration ³⁶ or AA furnace	236.1 236.2	3111 B or C 3113 B	D1068-90(A or B) D1068-90(C)	I-3381-85	974.27. ³
	ICP/AES ³⁶ or DCP ³⁶ or Colorimetric (Phenanthroline)	⁵ 200.7	3120 B	D4190-82(88) D1068-90(D)		Note 34. Note 22.
31.	Kjeldahl Nitrogen—Total, (as N), mg/L:					
	Digestion and distillation followed by:	351.3	4500-NH ₃ B or C	D3590-89(A)		
	Titration	351.3	4500-NH ₃ E	D3590-89(A)		973.48. ³
	Nesslerization	351.3	4500-NH ₃ C	D3590-89(A)		
	Electrode	351.3	4500-NH ₃ F or G			
	Automated phenate colorimetric	351.1			I-4551-78 ₈	
	Semi-automated block digester colorimetric	351.2		D3590-89(B)		
	Manual or block digester potentiometric	351.4		D3590-89(A)		
	Block Digester, followed by:					
	Auto distillation and Titration, or Nesslerization					Note 39. Note 40.
	Flow injection gas diffusion					Note 41.
32.	Lead—Total, ⁴ mg/L; Digestion ⁴ followed by:					
	AA direct aspiration ³⁶ or AA furnace	239.1 239.2	3111 B or C 3113 B	D3559-90(A or B) D3559-90(D)	I-3399-85	974.27. ³
	ICP/AES ³⁶ or DCP ³⁶ or Voltametry ¹¹ or Colorimetric (Dithizone)	⁵ 200.7	3120 B	D4190-82(88) D3559-90(C)		Note 34.
33.	Magnesium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
	AA direct aspiration or ICP/AES	242.1 ⁵ 200.7	3111 B 3120 B	D511-93(B)	I-3447-85	974.27. ³
	DCP, or Gravimetric		3500-Mg D			Note 34.
34.	Manganese—Total, ⁴ mg/L; Digestion ⁴ followed by:					
	AA direct aspiration ³⁶ or AA furnace	243.1 243.2	3111 B 3113 B	D858-90(A or B) D858-90(C)	I-3454-85	974.27. ³
	ICP/AES ³⁶ or DCP ³⁶ or Colorimetric (Persulfate), or (Periodate)	⁵ 200.7	3120 B	D4190-82(88) 3500-Mn D		Note 34. 920.203. ³ Note 23.
35.	Mercury—Total, ⁴ mg/L:					
	Cold vapor, manual, or Automated	245.1 245.2	3112 B	D3223-91	I-3462-85	³ 977.22

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA 1,35	STD methods 18th ed.	ASTM	USGS ²	Other
Oxidation, purge and trap, and cold vapor atomic fluorescence spectrometry (ng/L).	⁴³ 1631	
36. Molybdenum—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	246.1	3111 D	I-3490-85	
AA furnace	246.2	3113 B		
ICP/AES	⁵ 200.7	3120 B		
DCP		Note 34.
37. Nickel—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	249.1	3111 B or C	D1886-90(A or B)	I-3499-85	
AA furnace	249.2	3113 B	D1886-90(C)		
ICP/AES ³⁶	⁵ 200.7	3120 B		
DCP ³⁶ , or	D4190-82(88)		Note 34.
Colorimetric (heptoxime)	3500-Ni D		
38. Nitrate (as N), mg/L:					
Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).	352.1	973.50, ³ 419 D, 17 p. 28, ⁹
39. Nitrate-nitrite (as N), mg/L:					
Cadmium reduction, Manual or	353.3	4500-NO ₃ - E	D3867-90(B)		
Automated, or	353.2	4500-NO ₃ - F	D3867-90(A)	I-4545-85	
Automated hydrazine	353.1	4500-NO ₃ - H		
40. Nitrite (as N), mg/L; Spectrophotometric:					
Manual or	354.1	4500-NO ₂ - B	Note 25.
Automated (Diazotization)	I-4540-85	
41. Oil and grease—Total recoverable, mg/L:	413.1	5520 B ³⁸	
Gravimetric (extraction)					
Oil and grease and non-polar material, mg/L:					
Hexane extractable material (HEM): <i>n</i> -Hexane extraction and gravimetry ⁴² .	1664, Rev. A	
Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry ⁴² .	1664, Rev. A	
42. Organic carbon—Total (TOC), mg/L:					
Combustion or oxidation	415.1	5310 B, C, or D	D2579-93 (A or B)	973.47, ³ p. 14. ²⁴
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	365.1	4500-P F	I-4601-85	973.56. ³
Manual single reagent	365.2	4500-P E	D515-88(A)	973.55. ³
Manual two reagent	365.3	
45. Osmium—Total ⁴ , mg/L; Digestion ⁴ followed by:					
AA direct aspiration, or	252.1	3111 D	
AA furnace	252.2	
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification), or	360.2	4500-O C	D888-92(A)	I-1575-78 ⁸	973.45B. ³

§ 136.3

40 CFR Ch. I (7-1-02 Edition)

Electrode	360.1	4500-O G	D888-92(B)	I-1576-78 ⁸	
47. Palladium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration, or	253.1	3111 B			p. S27, ¹⁰
AA furnace	253.2				p. S28, ¹⁰
DCP					Note 34.
48. Phenols, mg/L:					
Manual distillation ²⁶	420.1				Note 27.
Followed by:					
Colorimetric (4AAP) manual, or	420.1				Note 27.
Automated ¹⁹	420.2				
49. Phosphorus (elemental), mg/L:					
Gas-liquid chromatography					Note 28.
50. Phosphorus—Total, mg/L:					
Persulfate digestion followed by	365.2	4500-P B,5			973.55. ³
Manual or	365.2 or 365.3	4500-P E	D515-88(A)		
Automated ascorbic acid reduction	365.1	4500-P F		I-4600-85	973.56. ³
Semi-automated block digester	365.4		D515-88(B)		
51. Platinum—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	255.1	3111 B			
AA furnace	255.2				
DCP					Note 34.
52. Potassium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	258.1	3111 B		I-3630-85	973.53. ³
ICP/AES	⁵ 200.7	3120 B			
Flame photometric, or		3500-K D			
Colorimetric					317 B. ¹⁷
53. Residue—Total, mg/L:					
Gravimetric, 103-105°	160.3	2540 B		I-3750-85	
54. Residue—filterable, mg/L:					
Gravimetric, 180°	160.1	2540 C		I-1750-85	
55. Residue—nonfilterable (TSS), mg/L:					
Gravimetric, 103-105° post washing of residue	160.2	2540 D		I-3765-85	
56. Residue—settleable, mg/L:					
Volumetric, (Imhoff cone), or gravimetric	160.5	2540 F			
57. Residue—Volatile, mg/L:					
Gravimetric, 550°	160.4			I-3753-85	
58. Rhodium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration, or	265.1	3111 B			
AA furnace	265.2				
59. Ruthenium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration, or	267.1	3111 B			
AA furnace	267.2				
60. Selenium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA furnace	270.2	3113 B	D3859-93(B)		
ICP/AES, ³⁶ or	⁵ 200.7	3120 B			
AA gaseous hydride		3114 B	D3859-93(A)	I-3667-85	
61. Silica ³⁷ —Dissolved, mg/L; 0.45 micron filtration followed by:					
Colorimetric, Manual or	370.1	4500-Si D	D859-88	I-1700-85	

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA 1,35	STD methods 18th ed.	ASTM	USGS ²	Other
Automated (Molybdosilicate), or				I-2700-85	
ICP	⁵ 200.7	3120 B			
62. Silver—Total, ⁴ mg/L; Digestion ^{4,29} followed by:					
AA direct aspiration	272.1	3111 B or C		I-3720-85	974.27, ³ p. 37. ⁹
AA furnace	272.2	3113 B			
ICP/AES	⁵ 200.7	3120 B			
DCP					Note 34.
63. Sodium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	273.1	3111 B		I-3735-85	973.54, ³
ICP/AES	⁵ 200.7	3120 B			
DCP, or					Note 34.
Flame photometric		3500 Na D			
64. Specific conductance, micromhos/cm at 25 °C:					
Wheatstone bridge	120.1	2510 B	D1125-91(A)	I-1780-85	973.40, ³
65. Sulfate (as SO ₄), mg/L:					
Automated colorimetric (barium chloranilate)	375.1				
Gravimetric	375.3	4500-SO ₄ -2 C or D			925.54, ³
Turbidimetric, or	375.4		D516-90		426C. ³⁰
66. Sulfide (as S), mg/L:					
Titrimetric (iodine), or	376.1	4500-S- ² E		I-3840-85	
Colorimetric (methylene blue)	376.2	4500-S- ² D			
67. Sulfite (as SO ₃), mg/L:					
Titrimetric (iodine-iodate)	377.1	4500-SO ₃ -2 B			
68. Surfactants, mg/L:					
Colorimetric (methylene blue)	425.1	5540 C	D2330-88		
69. Temperature, °C:					
Thermometric	170.1	2550 B			Note 32.
70. Thallium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	279.1	3111 B			
AA furnace	279.2				
ICP/AES, or	⁵ 200.7	3120 B			
71. Tin—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	282.1	3111 B		I-3850-78 ⁸	
AA furnace, or	282.2	3113 B			
ICP/AES	⁵ 200.7				
72. Titanium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	283.1	3111 D			
AA furnace	283.2				
DCP					Note 34.
73. Turbidity, NTU:					
Nephelometric	180.1	2130 B	D1889-88(A)	I-3860-85	
74. Vanadium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	286.1	3111 D			
AA furnace	286.2		D3373-93		

ICP/AES	5200.7	3120 B			
DCP, or			D4190-82(88)		Note 34.
Colorimetric (Gallic acid)		3500-V D			
75. Zinc—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	289.1	3111 B or C	D1691-90 (A or B)	I-3900-85	974.27, ³ p. 37. ⁹
AA furnace	289.2				
ICP/AES ³⁶	5200.7	3120 B			
DCP, ³⁶ or			D4190-82(88)		Note 34.
Colorimetric (Dithizone) or		3500-Zn E			
(Zincon)		3500-Zn F			Note 33.

Table IB Notes:

¹Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-600/4-79-020, Revised March 1983 and 1979 where applicable.

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

³Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 15th ed. (1990).

⁴For 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 and 1983". One (section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples 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 may also benefit by this vigorous digestion, however, vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICP/AES as well as determinations for certain elements such as antimony, arsenic, the noble metals, mercury, selenium, silver, tin, and titanium require a modified sample digestion procedure and in all cases the method write-up should be consulted for specific instructions and/or cautions.

NOTE TO TABLE IB NOTE 4: If the digestion procedure for direct aspiration AA 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 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.

⁵The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

⁶Manual 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.

⁷Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, dated February 19, 1976, (Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

⁸The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).

⁹American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

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

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

¹²Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required can the permittee report data using the nitrification inhibitor.

¹³OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

¹⁴Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

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

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

¹⁷The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

¹⁸National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

¹⁹Copper, Biocinchonate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

- ²⁰ After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.
- ²¹ Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
- ²² Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²³ Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.
- ²⁴ Wershaw, R.L., et al, "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14.
- ²⁵ Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²⁶ Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- ²⁷ The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.
- ²⁸ R. F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, vol. 47, No. 3, pp. 421-426, 1970.
- ²⁹ Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na₂S₂O₃ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- ³⁰ The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.
- ³¹ EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.
- ³² Stevens, H.H., Ficke, J.F., and Smoot, G.F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation", Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975.
- ³³ Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, CO 80537.
- ³⁴ "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.
- ³⁵ Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals".
- ³⁶ "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, NC 28106-0200, April 16, 1992. Available from the CEM Corporation.
- ³⁷ When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
- ³⁸ Only the trichlorofluoromethane extraction solvent is approved.
- ³⁹ Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, Perstop Analytical Corporation.
- ⁴⁰ Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, Perstop Analytical Corporation.
- ⁴¹ Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, Perstop Analytical Corporation.
- ⁴² Method 1664, Revision A "n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry" EPA-821-R-98-002, February 1999. Available at NTIS, PB-121949. U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.
- ⁴³ The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA-821-R-96-011) are recommended to preclude contamination at low-level, trace metal determinations.
- ⁴⁴ Available Cyanide, Method OIA-1677 (Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry), ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842-9010.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter ¹	EPA method number ²⁷					Other
	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	
1. Acenaphthene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
2. Acenaphthylene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
3. Acrolein	603	⁴ 604, 1624			
4. Acrylonitrile	603	⁴ 624, 1624	610			
5. Anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
6. Benzene	602	624, 1624	6210 B, 6220 B		
7. Benzidine	⁵ 625, 1625	605			Note 3, p.1.
8. Benzo(a)anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
9. Benzo(a)pyrene	610	625, 1625	610	6410 B, 6440 B	D4657-92	

10. Benzo(b)fluoranthene	610	625, 1625	610	6410 B, 6440 B
11. Benzo(g, h, i)perylene	610	625, 1625	610	6410 B, 6440 B
12. Benzo(k)fluoranthene	610	625, 1625	610	6410 B, 6440 B
13. Benzyl chloride				
14. Benzyl butyl phthalate	606	625, 1625		6410 B
15. Bis(2-chloroethoxy) methane	611	625, 1625		6410 B
16. Bis(2-chloroethyl) ether	611	625, 1625		6410 B
17. Bis (2-ethylhexyl) phthalate	606	625, 1625		6410 B, 6230 B
18. Bromodichloromethane	601	624, 1624		6210 B, 6230 B
19. Bromoform	601	624, 1624		6210 B, 6230 B
20. Bromomethane	601	624, 1624		6210 B, 6230 B
21. 4-Bromophenylphenyl ether	611	625, 1625		6410 B
22. Carbon tetrachloride	601	624, 1624		6230 B, 6410 B
23. 4-Chloro-3-methylphenol	604	625, 1625		6410 B, 6420 B
24. Chlorobenzene	601, 602	624, 1624		6210 B, 6220 B 6230 B
25. Chloroethane	601	624, 1624		6210 B, 6230 B
26. 2-Chloroethylvinyl ether	601	624, 1624		6210 B, 6230 B
27. Chloroform	601	624, 1624		6210 B, 6230 B
28. Chloromethane	601	624, 1624		6210 B, 6230 B
29. 2-Chloronaphthalene	612	625, 1625		6410 B
30. 2-Chlorophenol	604	625, 1625		6410 B, 6420 B
31. 4-Chlorophenylphenyl ether	611	625, 1625		6410 B
32. Chrysene	610	625, 1625	610	6410 B, 6440 B
33. Dibenzo(a,h)anthracene	610	625, 1625	610	6410 B, 6440 B
34. Dibromochloromethane	601	624, 1624		6210 B, 6230 B
35. 1, 2-Dichlorobenzene	601,602,612	624,625,1625		6410 B, 6230 B, 6220 B
36. 1, 3-Dichlorobenzene	601,602,612	624,625,1625		6410 B, 6230 B, 6220 B
37. 1,4-Dichlorobenzene	601, 602, 612	624, 625, 1625		6410 B, 6220 B, 6230 B
38. 3, 3-Dichlorobenzidine		625, 1625	605	6410 B
39. Dichlorodifluoromethane	601			6230 B
40. 1, 1-Dichloroethane	601	624, 1624		6230 B, 6210 B
41. 1, 2-Dichloroethane	601	624, 1624		6230 B, 6210 B
42. 1, 1-Dichloroethene	601	624, 1624		6230 B, 6210 B
43. trans-1, 2-Dichloroethene	601	624, 1624		6230 B, 6210 B
44. 2, 4-Dichlorophenol	604	625, 1625		6420 B, 6410 B
45. 1, 2-Dichloropropane	601	624, 1624		6230 B, 6210 B
46. cis-1, 3-Dichloropropene	601	624, 1624		6230 B, 6210 B
47. trans-1, 3-Dichloropropene	601	624, 1624		6230 B, 6210 B
48. Diethyl phthalate	606	625, 1625		6410 B
49. 2, 4-Dimethylphenol	604	625, 1625		6420 B, 6410 B
50. Dimethyl phthalate	606	625, 1625		6410 B
51. Di-n-butyl phthalate	606	625, 1625		6410 B
52. Di-n-octyl phthalate	606	625, 1625		6410 B
53. 2,4-Dinitrophenol	604	625, 1625		6420 B, 6410 B
54. 2,4-Dinitrotoluene	609	625, 1625		6410 B
55. 2, 6-Dinitrotoluene	609	625, 1625		6410 B

D4657-92
D4657-92
D4657-92

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

Note 3, p.130.

Note 3, p.130.

Note, p.130.

D4657-92
D4657-92

Environmental Protection Agency

§ 136.3

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter ¹	EPA method number ^{2,7}					
	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	Other
56. Epichlorohydrin						Note 3, p.130 Note 6, p.S102.
57. Ethylbenzene	602	624, 1624		6220 B, 6210 B		
58. Fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
59. Fluorene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
60. 1,2,3,4,6,7,8-Heptachlorodibenzofuran		1613				
61. 1,2,3,4,7,8,9-Heptachlorodibenzofuran		1613				
62. 1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin		1613				
63. Hexachlorobenzene	612	625, 1625		6410 B		
64. Hexachlorobutadiene	612	625, 1625		6410 B		
65. Hexachlorocyclopentadiene	612	625, 1625 ⁵		6410 B		
66. 1,2,3,4,7,8-Hexachlorodibenzofuran		1613				
67. 1,2,3,6,7,8-Hexachlorodibenzofuran		1613				
68. 1,2,3,7,8,9-Hexachlorodibenzofuran		1613				
69. 2,3,4,6,7,8-Hexachlorodibenzofuran		1613				
70. 1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin		1613				
71. 1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin		1613				
72. 1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin		1613				
73. Hexachloroethane	616	625, 1625		6410 B		
74. Ideno(1,2,3- <i>cd</i>)pyrene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
75. Isophorone	609	625, 1625		6410 B		
76. Methylene chloride	601	624, 1624		6230 B		Note 3, p. 130.
77. 2-Methyl-4,6-dinitrophenol	604	625, 1625		6420 B, 6410 B		
78. Naphthalene	610	625, 1625	610	6410 B, 6440 B		
79. Nitrobenzene	609	625, 1625		6410 B	D4657-87	
80. 2-Nitrophenol	604	625, 1625		6410 B, 6420 B		
81. 4-Nitrophenol	604	625, 1625		6410 B, 6420 B		
82. N-Nitrosodimethylamine	607	625, 1625		6410 B		
83. N-Nitrosodi- <i>n</i> -propylamine	607	625, 1625 ⁵		6410 B		
84. N-Nitrosodiphenylamine	607	625, 1625 ⁵		6410 B		
85. Octachlorodibenzofuran		1613				
86. Octachlorodibenzo- <i>p</i> -dioxin		1613				
87. 2,2-Oxybis(1-chloropropane)	611	625, 1625		6410 B		
88. PCB-1016	608	625		6410 B		Note 3, p. 43.
89. PCB-1221	608	625		6410 B		Note 3, p. 43.
90. PCB-1232	608	625		6410 B		Note 3, p. 43.
91. PCB 1242	608	625		6410 B		Note 3, p. 43.
92. PCB-1248	608	625				
93. PCB-1254	608	625		6410 B		Note 3, p. 43.
94. PCB-1260	608	625		6410 B, 6630 B		Note 3, p. 43.
95. 1,2,3,7,8-Pentachlorodibenzofuran		1613				
96. 2,3,4,7,8-Pentachlorodibenzofuran		1613				
97. 1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin		1613				

§ 136.3

40 CFR Ch. I (7-1-02 Edition)

98. Pentachlorophenol	604	625, 1625	6410 B, 6630 B		Note 3, p. 140.
99. Phenanthrene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
100. Phenol	604	625, 1625	6420 B, 6410 B		
101. Pyrene	610	625, 1625	610	6410 B, 6440 B	D4657-87	
102. 2,3,7,8-Tetrachlorodibenzofuran	1613			
103. 2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	613, 1613 ⁵			
104. 1,1,2,2-Tetrachloroethane	601	624, 1624	6230 B, 6210 B		Note 3, p. 130.
105. Tetrachloroethene	601	624, 1624	6230 B, 6410 B		Note 3, p. 130.
106. Toluene	602	624, 1624	6210 B, 6220 B		
107. 1,2,4-Trichlorobenzene	612	625, 1625	6410 B		Note 3, p. 130.
108. 1,1,1-Trichloroethane	601	624, 1624	6210 B, 6230 B		
109. 1,1,2-Trichloroethane	601	624, 1624	6210 B, 6230 B		Note 3, p. 130.
110. Trichloroethene	601	624, 1624	6210 B, 6230 B		
111. Trichlorofluoromethane	601	624	6210 B, 6230 B		
112. 2,4,6-Trichlorophenol	604	625, 1625	6410 B, 6240 B		
113. Vinyl chloride	601	624, 1624	6210 B, 6230 B		

Table 1C notes:

- ¹All parameters are expressed in micrograms per liter (µg/L) except for Method 1613 in which the parameters are expressed in picograms per liter (pg/L).
- ²The full text of Methods 601-613, 624, 625, 1624, and 1625, are given at appendix A, "Test Procedures for Analysis of Organic Pollutants," of this part 136. The full text of Method 1613 is incorporated by reference into this part 136 and is available from the National Technical Information Services as stock number PB95-104774. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, "Definition and Procedures for the Determination of the Method Detection Limit," of this part 136.
- ³"Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.
- ⁴Method 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.
- ⁵Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.
- ^{6a}625, Screening only.
- ^{6b}"Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).
- ⁷Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601-603, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 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.
- NOTE: These warning limits are promulgated as an "interim final action with a request for comments."
- ⁸"Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation Revised 10/28/94.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES 1

Parameter	Method	EPA ²⁷	Standard methods 18th Ed.	ASTM	Other
1. Aldrin	GC GC/MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
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. α-BHC	GC GC/MS	608 ⁵ 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; note 8.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES 1—Continued

Parameter	Method	EPA ^{2,7}	Standard methods 18th Ed.	ASTM	Other
9. β-BHC	GC	608	6630 C	D3086-90	Note 8.
	GC/MS	⁵ 625	6410 B		
10. δ-BHC	GC	608	6630 C	D3086-90	Note 8.
	GC/MS	⁵ 625	6410 B		
11. δ-BHC (Lindane)	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
12. Captan	GC		6630 B	D3086-90	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	6630 B & C	D3086-90	Note 3, p. 7; note 8.
	GC/MS	625	6410 B		
16. Chlorpropham	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		6640 B		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; note 8.
	GC/MS	625	6410 B		
19. 4,4'-DDE	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
20. 4,4'-DDT	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
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		6630 B & C		Note 3, p. 7.
27. Dicofol	GC			D3086-90	
28. Dieldrin	GC	608	6630 B & C		Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
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	6630 B & C	D3086-90	Note 3, p. 7; note 8.
	GC/MS	⁵ 625	6410 B		
33. Endosulfan II	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 8.
	GC/MS	⁵ 625	6410 B		
34. Endosulfan Sulfate	GC	608	6630 C		Note 8.
	GC/MS	625	6410 B		
35. Endrin	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	⁵ 625	6410 B		
36. Endrin aldehyde	GC	608			Note 8.
	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	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
41. Heptachlor epoxide	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 6, p. S73; note 8.
	GC/MS	625	6410 B		
42. Isodrin	GC				Note 4, p. 30; Note 6, p. S73.
43. Linuron	GC				Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		6630 C		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		6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
47. Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S60.
48. Mirex	GC		6630 B & C		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
50. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
51. Nuburon	TLC				Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		6630 C		Note 3, p. 25.
54. PCNB	GC		6630 B & C		Note 3, p. 7.
55. Perthane	GC			D3086-90	
56. Prometron	GC				Note 3, p. 83; Note 6, p. S68.
57. Prometryn	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		6630 B & C		Note 3, p. 7.
65. Swep	TLC				Note 3, p. 104; Note 6, p. S64.
66. 2,4,5-T	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-TP (Silvex)	GC		6640 B		Note 3, p. 115.
68. Terbutylazine	GC				Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
70. Trifluralin	GC		6630 B		Note 3, p. 7.

Table ID notes:

- ¹ Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.
- ² The full text of Methods 608 and 625 are given at Appendix A. "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B. "Definition and Procedure for the Determination of the Method Detection Limit", of this Part 136.
- 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.
- ⁴ "Methods for Analysis of Organic Substances in Water and Fluvial Sediments," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).
- ⁵ The method may be extended to include α -BHC, γ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
- ⁶ "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency." Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

⁷ Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

NOTE: These warning limits are promulgated as an "Interim final action with a request for comments."

⁸ "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation, Revised 10/28/94.

TABLE IE.—LIST OF APPROVED RADIOLOGIC TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)			
		EPA ¹	Standard methods 18th Ed.	ASTM	USGS ²
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900	7110 B	D1943-90	pp. 75 and 78. ³
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	D1943-90	P. 79.
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	D1890-90	pp. 75 and 78. ³
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	D1890-90	p. 79.
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	D2460-90	
(b)Ra, pCi per liter	Scintillation counter	903.1	7500Ra C	D3454-91	p. 81.

Table IE notes:

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

² Fishman, M.J. and Brown, Eugene," Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

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".

TABLE IF.—LIST OF APPROVED METHODS FOR PHARMACEUTICAL POLLUTANTS

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

Table 1F note:

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

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, IE, and IF are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources. The full texts of all the test procedures cited are available for inspection at the National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, 26 West Martin Luther King Dr., Cincinnati, OH 45268 and the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

REFERENCES, SOURCES, COSTS, AND
TABLE CITATIONS:

(1) The full texts of Methods 601-613, 624, 625, 1613, 1624, and 1625 are printed in appendix A of this part 136. The full text for determining the method detection limit when using the test procedure

is given in appendix B of this part 136. The full text of Method 200.7 is printed in appendix C of this part 136. Cited in: Table IB, Note 5; Table IC, Note 2; and Table ID, Note 2.

(2) USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8-78/017. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-290329/AS. Cost: \$36.95. Table IA, Note 3.

(3) "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979, or "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, EPA-600/4-79-020, Revised March 1983. Available from: ORD Publications, CERL, U.S. Environmental

§ 136.3

40 CFR Ch. I (7-1-02 Edition)

Protection Agency, Cincinnati, Ohio 45268, Table IB, Note 1.

(4) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IC, Note 3; Table D, Note 3.

(5) "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," U.S. Environmental Protection Agency, EPA-600/4-80-032, 1980. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IE, Note 1.

(6) American Public Health Association. 1992. Standard Methods for the Examination of Water and Wastewater. 18th Edition. Amer. Publ. Hlth. Assoc., 1015 15th Street NW, Washington, DC 20005. Cost: \$160.00. Table IA, Note 4.

(7) Ibid, 15th Edition, 1980. Table IB, Note 30; Table ID.

(8) Ibid, 14th Edition, 1975. Table IB, Notes 17 and 27.

(9) "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 NW., Washington, DC 20036. Cost available from publisher. Table IB, Note 10; Table IC, Note 6; Table ID, Note 6.

(10) Annual Book of ASTM Standards, Water and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994 in 40 CFR 136.3, Tables IB, IC, ID and IE.

(11) USGS. 1989. U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, Virginia. Available from: USGS Books and Open-File Reports Section, Federal Center, Box 25425, Denver, Colorado 80225. Cost: \$18.00. Table IA, Note 5.

(12) "Methods for Determination of Inorganic Substances in Water and

Fluvial Sediments," by M.J. Fishman and Linda C. Friedman, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5 Chapter A1 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$108.75 (subject to change). Table IB, Note 2.

(13) "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," N.W. Skougstad and others, editors. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1 (1979). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$10.00 (subject to change), Table IB, Note 8.

(14) "Methods for the Determination of Organic Substances in Water and Fluvial Sediments," Wershaw, R.L., et al, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$0.90 (subject to change). Table IB, Note 24; Table ID, Note 4.

(15) "Water Temperature—Influential Factors, Field Measurement and Data Presentation," by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$1.60 (subject to change). Table IB, Note 32.

(16) "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 76-77 (1976). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: \$13.50 (subject to change). Table IE, Note 2.

(17) "Official Methods of Analysis of the Association of Official Analytical Chemicals", Methods manual, 15th Edition (1990). Price: \$240.00. Available from: The Association of Official Analytical Chemists, 2200 Wilson Boulevard, Suite 400, Arlington, VA 22201. Table IB, Note 3.

(18) "American National Standard on Photographic Processing Effluents," April 2, 1975. Available from: American

Environmental Protection Agency

§ 136.3

National Standards Institute, 1430 Broadway, New York, New York 10018. Table IB, Note 9.

(19) "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. Cost available from publisher. Table IB, Note 18.

(20) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 7.

(21) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 14.

(22) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 13.

(23) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 16.

(24) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979, Method and price available from Hach Chemical Company, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 19.

(25) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA. October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 21.

(26) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 389 Loveland, Colorado 80537. Table IB, Note 22.

(27) Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 23.

(28) Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 25.

(29) Zincon Method for Zinc, Method 8009. Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 33.

(30) "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. Table IB, Note 28.

(31) "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes", Method AES 0029, 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table B, Note 34.

(32) "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1992. Available from the CEM Corporation. Table IB, Note 36.

(33) "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk" Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220-9E-10, St. Paul, MN 55144-1000. Method available from 3M Corporation. Table IC, Note 8 and Table ID, Note 8.

(34) USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms.

Fourth Edition, December 1993. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (EPA/600/4-90/027F). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-91-167650. Cost: \$31.00. Table IA, Note 17. See changes in the manual, listed in Part V of this rule.

(35) "Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 39.

(36) "Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 40.

(37) "Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 41.

(38) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4-91/002). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-92-139492. Cost: \$31.00. Table IA, Note 8.

(39) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition, July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-91/003. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ.

No. PB-92-139484. Cost: \$45.00. Table IA, Note 9.

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

(41) USEPA. 2001. Method 1631, Revision C, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry." March 2001. Office of Water, U.S. Environmental Protection Agency (EPA-821-R-01-024). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161. Publication No. PB2001-102796. Cost: \$25.50. Table IB, Note 43.

(42) USEPA, January 1999 Errata for the Effluent and Receiving Water Testing Manuals: Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms; Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms; and Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. U.S. Environmental Protection Agency, Office of Research and Development, Duluth, MN. EPA-600/R-98/182.

(43) Method OIA-1677, Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry. August 1999. ALPKEM, OI Analytical, Box 648,

Environmental Protection Agency

§ 136.3

Wilsonville, Oregon 97070 (EPA-821-R-99-013). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161. Publication No. PB99-132011. Cost: \$22.50. Table IB, Note 44.

(c) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters

cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded, by the Regional Administrator, to the Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Table IA—Bacteria Tests:			
1-4 Coliform, fecal and total	P, G	Cool, 4C, 0.008% Na ₂ S ₂ O ₃ ⁵ ...	6 hours.
5 Fecal streptococci	P, G	Cool, 4C, 0.008% Na ₂ S ₂ O ₃ ⁵ ...	6 hours.
Table IA—Aquatic Toxicity Tests:			
6-10 Toxicity, acute and chronic	P, G	Cool, 4 °C ¹⁶	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, Gdo	Do.
4. Ammonia	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz.	HNO ₃ TO pH<2	6 months.
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 ₂ SO ₄ to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, Gdo	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 ₂ SO 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 ₂ SO 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, 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 boron, chromium VI and mercury.	P, Gdo	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H ₂ SO ₄ to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HCl or H ₂ SO ₄ or H ₃ PO 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.
47. Winklerdo	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, Gdo	7 days.
55. Residue, Nonfilterable (TSS)	P, Gdo	7 days.
56. Residue, Settleable	P, Gdo	48 hours.
57. Residue, volatile	P, Gdo	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, Gdo	Do.
65. Sulfate	P, Gdo	Do.
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.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests ⁸ 13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ .	14 days.
6, 57, 106. Purgeable aromatic hydrocarbonsdo	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ HCl to pH2 ⁹ .	Do.
3, 4. Acrolein and acrylonitriledo	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ adjust pH to 4–5 ¹⁰ .	Do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹ .	G, Teflon-lined cap..	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction.
7, 38. Benzidines ¹¹dodo	7 days until extraction. ¹³
14, 17, 48, 50–52. Phthalate esters ¹¹do	Cool, 4 °C	7 days until extraction; 40 days after extraction.
82–84. Nitrosamines ^{11 14}do	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ store in dark.	Do.
88–94. PCBs ¹¹do	Cool, 4 °C	Do.
54, 55, 75, 79. Nitroaromatics and isophorone ¹¹do	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵ store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹dodo	Do.
15, 16, 21, 31, 87. Haloethers ¹¹do	Cool, 4 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	Do.
29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons ¹¹do	Cool, 4 °C	Do.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs ¹¹dodo	Do.
aqueous: field and lab preservation.	G	Cool, 0–4 °C, pH<9, 0.008% Na ₂ S ₂ O ₃ ⁵ .	1 year.
Solids, mixed phase, and tissue: field preservation..do	Cool, <4 °C	7 days.
Solids, mixed phase, and tissue: lab preservationdo	Freeze, <–10 °C	1 year.
Table ID—Pesticides Tests: 1–70. Pesticides ¹¹do	Cool, 4°C, pH 5–9 ¹⁵	Do.
Table IE—Radiological Tests: 1–5. Alpha, beta and radium	P, G	HNO ₃ to pH<2	6 months.

Table II Notes
¹ Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).
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 is completed.

Environmental Protection Agency

§ 136.4

³When 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 (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) 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).

⁴Samples 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 for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

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

⁶Maximum 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.

⁷Samples should be filtered immediately on-site before adding preservative for dissolved metals.

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

⁹Sample requiring no pH adjustment must be analyzed within seven days of sampling.

¹⁰The 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.

¹¹When 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).

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

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

¹⁴For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵The 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% Na₂S₂O₃.

¹⁶Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 4°C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987; 55 FR 24534, June 15, 1990; 55 FR 33440, Aug. 15, 1990; 56 FR 50759, Oct. 8, 1991; 57 FR 41833, Sept. 11, 1992; 58 FR 4505, Jan. 31, 1994; 60 FR 17160, Apr. 4, 1995; 60 FR 39588, 39590, Aug. 2, 1995; 60 FR 44672, Aug. 28, 1995; 60 FR 53542, 53543, Oct. 16, 1995; 62 FR 48403, 48404, Sept. 15, 1997; 63 FR 50423, Sept. 21, 1998; 64 FR 4978, Feb. 2, 1999; 64 FR 10392, Mar. 4, 1999; 64 FR 26327, May 14, 1999; 64 FR 30433, 30434, June 8, 1999; 64 FR 73423, Dec. 30, 1999; 66 FR 32776, June 18, 2001]

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in trip-

licate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

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

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published