

ENVIRONMENTAL PROTECTION AGENCY**40 CFR Part 136**

[FRL-5162-7]

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Technical Amendments**AGENCY:** Environmental Protection Agency (EPA).**ACTION:** Final rule: Technical amendments.

SUMMARY: This action under the Clean Water Act (CWA) section 304(h) amends 40 CFR Part 136 to add clarifying footnotes to the lists of approved test procedures, to update method citations in Tables IA, IB, IC, and ID, to amend the incorporation by reference section of the regulation accordingly, and to correct certain typographical errors and omissions in the Technical Amendments appearing in the **Federal Register** of January 31, 1994.

EFFECTIVE DATE: This amendment becomes effective on May 4, 1995. The incorporation by reference of the publications listed in this document are approved by the Director of Federal Register as of May 4, 1995.

FOR FURTHER INFORMATION CONTACT: James E. Longbottom, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Telephone Number: (513) 569-7308.

SUPPLEMENTARY INFORMATION:**I**

These technical amendments update and/or correct errors and inadvertent omissions in the references to analytical methods already approved under section 304(h) to the current editions published by EPA, U.S. Geological Survey, Standard Methods for the Examination of Water and Wastewater (Standard Methods), the American Society for Testing and Materials (ASTM), and the Association of Official Analytical Chemists (AOAC) International. No new methods are introduced. EPA has carefully reviewed each approved method for substantive changes between the current editions and the previously approved editions. Methods cited in this amendment that were not previously cited are substantively the same as the approved EPA method and/or were derived from the EPA method.

II

References in Table IB, to the American Society for Testing and Materials (ASTM), have also been updated and corrected where appropriate to the 1994 edition. Several ASTM methods are no longer cited because they have been discontinued by ASTM and are not included in the 1994 Standards book.

III

The remaining amendments in this notice are very minor and are typographical or editorial in nature. The parts of Tables IA, IC and ID, and certain notes to Tables IB, IC, and ID where reference updates, corrections, and clarifications have been made are reprinted in this notice for the information and use of the regulated community. Table IB has been reprinted in its entirety for the convenience of the user.

Unless otherwise indicated in this notice, the methods contained in the Standard Methods 18th edition and the ASTM Standards 1994 edition are previously approved methods that were rebaloted without technical change or were not rebaloted. Any changes are editorial, typographical, or grammatical.

IV Regulatory Requirements**A. Executive Order 12866**

Under Executive Order 12866, EPA must determine whether a regulation is "major" and, therefore, requires a regulatory impact analysis. EPA has determined that these technical amendments are not major as they will not result in an effect on the economy of \$100 million or more, a significant increase in cost or prices, or any of the effects described in the Executive Order. These amendments simply specify analytical techniques which may be used by laboratories in measuring concentrations of certain analytes and, therefore, have no adverse economic impacts.

B. Administrative Procedure Act

The Administrative Procedure Act, 5 U.S.C. 553 (b)(B), authorizes an agency to forego notice and comment rulemaking when the agency for good cause finds that notice and public procedure thereon are impracticable, unnecessary or contrary to the public interest. EPA believes that public comment on the foregoing technical amendments is unnecessary because the updates to method references do not change the methods contained therein. In publishing the new editions of their test protocols, ASTM and Standard Methods have balloted these methods

for reapproval without technical change or the methods were republished as unballoted. Additionally, the typographical errors corrected in the CFR do not amend substantive requirements. Therefore, notice and public procedure is unnecessary and does not apply to this Technical Amendment Notice.

C. Regulatory Flexibility Act

This amendment is consistent with the objectives of the Regulatory Flexibility Act (5 U.S.C. 602 *et seq.*) because it will not have a significant economic impact on a substantial number of small entities. The procedures cited in this rule give all laboratories the flexibility to use these procedures or already approved alternative procedures.

D. Paperwork Reduction Act

This rule contains no request for information activities and, therefore, no information collection request (ICR) was submitted to the Office of Management and Budget (OMB) for review in compliance with the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.*

List of Subjects in 40 CFR Part 136

Environmental protection, Incorporation by reference, Reporting and recordkeeping requirements, Water pollution control.

Dated: February 15, 1995.

Joseph K. Alexander,

Acting Assistant Administrator for Research and Development, U.S. Environmental Protection Agency.

40 CFR part 136 is amended as follows:

PART 136—[AMENDED]

1. The authority citation for part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307, and 501(a) Pub. L. 95-217, 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).

2. Section 136.3 is amended as follows:

a. In paragraph (a) by revising entries 1 and 2 of Table IA, Table IB, entries 33 and 37 and Notes 3 and 5^a of Table IC, entries 8, 9, 10, and 22 and Note 5 of Table ID;

b. In paragraph (b) by revising Reference 10; and

c. In paragraph (e), in table II, under "Table IB-Inorganic Tests:" by revising entry 10 and under "Metals:" by revising entries "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”, 42 and 61, to read as follows:

§ 136.3 Identification of test procedures.

(a) * * *

TABLE IA.—LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter, units and method	Method ¹	Reference (method No. or page)			
		EPA ²	Standard methods 18th ed.	ASTM	USGS ³
Bacteria:					
1. Coliform (fecal), number per 100 mL	Most Probable Number (MPN), 5 tube, 3 dilution.	p. 132	9221C and E.		
	Membrane filter (MF) ⁴ , single step.	p. 124	9222D		B-0050-85
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution	p. 132	9221C and E .		
	MF ⁴ , single step ⁵	p. 124	9222D		
* * *	* * *	* * *	* * *	* * *	* * *

Table IA Notes:

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

² Bordner, R.H., and J.A. Winter, eds. 1978. "Microbiological Methods for Monitoring the Environment, Water and Waste". Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency. EPA-600/8-78-017.

³ Britton, L.J., and P.E. Greeson, P.E., eds., 1989. "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," Techniques of Water Resources Investigations of the U.S. Geological Survey, Techniques of Water Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, U.S. Geological Survey, U.S. Department of Interior, Reston, Virginia.

⁴ A 0.45 µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and to be free of extractables which could interfere with their growth.

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

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and method	Reference (method number or page)				
	EPA ^{1,35}	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	973.43. ³
				I-2030-85	
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 Spectrometry (ICP/AES) ³⁶ .	⁵ 200.7	3120 B			
Direct Current Plasma (DCP) ³⁶			D4190-82(88)		Note 34.
Colorimetric (Eriochrome cyanine R).		3500-Al 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)		

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 or Colorimetric (SDDC)	⁵ 200.7 206.4	3120 B 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.
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 3. ⁴
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.	974.27, ³ p. 37. ⁹
AA furnace	213.2	3113 B	D3557-90(D)		
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.	410.1 410.2 410.3	5220 C	D1252-88(A)	I-3560-85	973.46, ³ p. 17. ⁹
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	973.51. ³
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 DPD-FAS	330.2 330.4	4500-Cl C 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	974.27. ³

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
AA chelation-extraction	218.3	3111 C			
AA furnace	218.2	3113 B	D1687-92(C)		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶ or			D4190-82(88)		Note 34.
Colorimetric (Diphenylcarbazide)		3500-Cr D			
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			
(Bicinchoninate)		Or E			Note 19.
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. Cyanide amenable to chlorination,mg/L:					
Manual distillation with MgCl ₂ followed by titrimetric or Spectrophotometric.	335.1	4500-CN G	D2036-91(B)		
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	150.1	4500-H ⁺ B	D1293-84(90)(A or B) ..	I-1586-85	973.41. ³
Automated electrode					Note 21.
29. Iridium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration or	235.1	3111 B			
AA furnace	235.2				
30. Iron—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	236.1	3111 B or C	D1068-90(A or B)	I-3381-85	974.27. ³
AA furnace	236.2	3113 B	D1068-90(C)		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶ or			D4190-82(88)		Note 34.
Colorimetric (Phenanthroline)		3500-Fe D	D1068-90(D)		Note 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
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, or:	351.2		D3590-89(B)		
Manual or block digester Potentiometric.	351.4		D3590-89(A)		
32. Lead—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	239.1	3111 B or C	D3559-90(A or B)	I-3399-85	974.27. ³
AA furnace	239.2	3113 B	D3559-90(D)		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶			D4190-82(88)		Note 34.
Voltametry ¹¹ or			D3559-90(C)		
Colorimetric (Dithizone)		3500-Pb D			
33. Magnesium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	242.1	3111 B	D511-93(B)	I-3447-85	974.27. ³
ICP/AES	⁵ 200.7	3120 B			
DCP, or					Note 34.
Gravimetric		3500-Mg D			
34. Manganese—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration ³⁶	243.1	3111 B	D858-90(A or B)	I-3454-85	974.27. ³
AA furnace	243.2	3113 B	D858-90(C)		
ICP/AES ³⁶	⁵ 200.7	3120 B			
DCP ³⁶ or			D4190-82(88)		Note 34.
Colorimetric (Persulfate), or (Periodate)		3500-Mn D			920.203, ³ Note 23.
35. Mercury—Total, ⁴ mg/L:					
Cold vapor, manual or	245.1	3112 B	D3223-91	I-3462-85	977.22. ³
Automated	245.2				
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:					
Gravimetric (extraction)	413.1	5520 B ³⁸			
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:					

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
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
Electrode	360.1	4500-O G	D888-92(B)	I-1576-78 ⁸	
47. Palladium—Total, ⁴ mg/L; Diges- tion 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; Diges- tion 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 wash- ing 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; Diges- tion ⁴ followed by:					
AA direct aspiration, or	265.1	3111 B			
AA furnace	265.2				
59. Ruthenium—Total, ⁴ mg/L; Diges- tion ⁴ followed by:					
AA direct aspiration, or	267.1	3111 B			
AA furnace	267.2				
60. Selenium—Total, ⁴ mg/L; Diges- tion ⁴ followed by:					
AA furnace	270.2	3113 B	D3859-93(B)		

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 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	
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 fol- lowed 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; Diges- tion ⁴ followed by:					
AA direct aspiration	286.1	3111 D			
AA furnace	286.2		D3373-93		
ICP/AES	⁵ 200.7	3120 B			
DCP, or			D4190-82(88)		Note 34.
Colorimetric (Gallic acid)		3500-V D			
75. Zinc—Total, ⁴ mg/L; Digestion ⁴ fol- lowed 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	⁵ 200.7	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:

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

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

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

⁴ For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 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.

⁵ The 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.

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

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

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

⁹ American 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).

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

¹² Carbonaceous 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.

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

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

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

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

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

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

¹⁹ Copper, Bicinchoninate 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.

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

³⁸Only the trichlorofluoromethane extraction solvent is approved.

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

Parameter ¹	EPA method number ^{2,7}			Standard methods 18th ed.	ASTM	Other
	GC	GC/MS	HPLC			
* * * * *	*	*	*	*	*	*
33. Dibenzo(a,h)anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-92.	*
* * * * *	*	*	*	*	*	*
37. 1,4-Dichlorobenzene	601, 602, 612	624, 625, 1625	6410 B, 6220 B, 6230 B.		*
* * * * *	*	*	*	*	*	*

Table IC Notes:

¹All parameters are expressed in micrograms per liter (µg/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 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.

* * * * *

^{5a}625, Screening only.

* * * * *

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.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹

Parameter	Method	EPA ^{2,7}	Standard methods 18th ed.	ASTM	Other
* * * * *	*	*	*	*	*
8. α-BHC	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
	GC/MS	⁵ 625	6410 B		
9. β-BHC	GC	608	6630	D3086-90	
	GC/MS	⁵ 625	6410 B		
10. δ-BHC	GC	608	6630 B & C	D3086-90	
	GC/MS	⁵ 625	6410 B		
* * * * *	*	*	*	*	*
22. Demeton-S	GC		Note 3, p. 25: Note 6, p. S51.
* * * * *	*	*	*	*	*

Table ID Notes:

¹Pesticides 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.

²The 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.

* * * * *

⁵The 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).

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

* * * * *
 (b) * * *
 References, Sources, Costs, and Table Citations
 * * * * *
 (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.
 * * * * *
 (e) * * *

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

Parameter	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IB—Inorganic Tests:			
10. Boron	P, PFTE, or Quartz	HNO ₃ to pH<2	6 months.
Metals ⁷			
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, G	HNO ₃ to pH<2	6 months.
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.
61. Silica	P, PFTE, or Quartz	Cool, 4°C	28 days.

Table II—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 is 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 (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).

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 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.

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