M-I *L.L.C.* Product Specifications

Barite Ore

Revision Date : May 14, 2002
Supersedes : May 1, 1993

Property	Procedure	Specification
Density	API Spec 13A Sec 2.3	As Specified by Contract
Water Soluble Alkaline Earth Metals as Calcium	API Spec 13A Sec 2.6	250 mg/kg, maximum
Total Cadmium	40 CFR136 Table 1B	3.0 mg/kg, maximum
Total Lead	40 CFR136 Table 1B	1000 mg/kg, maximum prefered
Total Mercury	40 CFR136 Table 1B	1.0 mg/kg, maximum
High Temperature Caustic Soluble Carboantes and Sulfides	M-I Procedure #0180	CO_3 -- 1500 mg/kg, maximum S -- 25 mg/kg, maximum
Particle Size as shipped		200 mm, maximum

Signature Not Verified

Tim Hubbard
2002.06.24 12:01:10 -06'00'
I am approving this document

M-I QA Procedure : 0180
Procedure Name : HTCE
Procedure Date : August 9, 1996
Supersedes : None Previous

High Temperature Caustic Extraction Method
For the Quantitative Determination of Carbonates and Sulfides

1. Place 100.0 ml of a freshly prepared 2.0% NaOH solution into a 500 ml 316 Stainless Steel aging cell for each sample to be tested and also for a blank. The 2.0% NaOH should be freshly prepared, using 20.0 gm of Reagent Grade Sodium Hydroxide and diluted to 1 liter in a volumetric flask with deionized or distilled water. The flask should be tightly sealed while storing the stock solution.
2. Weigh 200.00 gms of each barite sample and slowly add to the aging cell.
3. Seal the aging cells and place in a Rotating oven to rotate the samples while aging..
4. Set the oven timer for 16 hours, and adjust the temperature setting to 350F. Turn on the rotator, and start oven.
5. After the aging period is complete, turn off the sample rotator, and the oven. Remove aging cells, and place in a sink of running water to cool the aging cells. The water level should not be above the lower edge of the aging cell cap. Cool the samples to room temperature.
6. Open the aging cells and collect the supernatant solution using a suitably sized syringe.
7. Filter the supernatant using Whatman 40 filter paper with a 4" glass filter funnel. After adding the supernatant, cover the funnel with a watch glass. Collect the filtrate in sealable sample vials. Label each vial.
8. Collect approximately 30 ml of each sample into separate vials, one to be used for the carbonate determination, and the other to be used for the sulfide.
9. Give the samples to Analytical for the Garret Gas Train evaluation, as per API RP 13B-1, Appendix A, current edition.
10. Results are reported in mg/kg of barite, corrected for background values by the blank.

    i.e. mg/kg CO_3 / S = (mg/l CO_3/S Sample - mg/l CO_3/S Blank) / 2

SUBCHAPTER D—WATER PROGRAMS (Continued)

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

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

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 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	p. 132 [3]	9221C E [4]	.....	
	3 dilution, or Membrane filter (MF) [2], single step	p. 124 [3]	9222D [4]		B–0050–85 [5]
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132 [3]	9221C E [4]	.....	
	MF, single step [6]	p. 124 [3]	9222D [4]		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]	.....	
	MF [2] single step or two step	p. 108 [3]	9222B [4]		B–0025–85 [5]
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]	.....	
	MF [2] with enrichment	p. 111 [3]	9222(B+B.5c) [4]		
5. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution	p. 139 [3]	9230B [4]	.....	
	MF [2], or	p. 136 [3]	9230C [4]		B–0055–85 [5]
	Plate count	p. 143 [3]			
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				
	Selenastrum growth	1002.0 [8]			
		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				
	Mysidopsis bahia survival, growth, and fecundity	1006.0 [9]			
	Arbacia punctulata fertilization	1007.0 [9]			
	Champia parvula reproduction	1008.0 [9]			
		1009.0 [9]			

Notes to Table IA:

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

[2] A 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.

[3] 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.

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

[5] 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 Interior, Reston, Virginia.

[6] 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.

[7] USEPA. 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.

[8] USEPA. 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).
[9] Short-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.

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 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 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_3B			973.49.[3]
Nesslerization	350.2	4500–NH_3C	D1426–93(A)	I–3520–85	973.49.[3]
Titration	350.2	4500–NH_3E			
Electrode	350.3	4500–NH_3F or G	D1426–93(B)		
Automated phenate, or	350.1	4500–NH_3H		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.

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 ..	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 ..... I–3562–85	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	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	974.27.[3]
AA chelation-extraction	218.3	3111 C			
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			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. 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	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.
31. Kjeldahl Nitrogen—Total, (as N), mg/L:					
Digestion and distillation followed by:	351.3	4500–NH_3B or C	D3590–89(A)		
Titration	351.3	4500–NH_3E	D3590–89(A)		973.48$_3$.
Nesslerization	351.3	4500–NH_3C	D3590–89(A)		
Electrode	351.3	4500–NH_3F 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					Note 39.
Nesslerization					Note 40.
Flow injection gas diffusion					Note 41.
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		3500–Mn D			920.203.[3]
(Periodate)					Note 23.
35. Mercury—Total,[4] mg/L:					
Cold vapor, manual, or	245.1	3112 B	D3223–91	I–3462–85	[3]977.22
Automated	245.2				

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: Gravimetric (extraction)	413.1	5520 B[38].			
Oil and grease and non-polar material, mg/L: Hexane extractable material (HEM): *n*-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]

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 digestor	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	[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] 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]	[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, Techniques of Water—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 ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures ''total BOD''. The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data 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, Biocinchoinate 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 mainfold 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 spectophotometric 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 $Na_2S_2O_3$ 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 Extractablke 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]					
	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	Other
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	