

Attachment 1

TABLE 1– LIST OF CHEMICAL AND BIOLOGICAL TEST PROCEDURES REMOVED AND ALTERNATE APPROVED REPLACEMENT METHODS

Parameter	Methodology	EPA	Std. Methods (18 th , 19 th)	Std. Methods (20 th)	Std. Methods Online	ASTM	USGS / AOAC / Other
Effectuated Parameters From the March 12, 2007 USEPA Method Update Rule (MUR) for Chemistry Table 1B							
1. Acidity , as CaCO ₃ , mg/L	Electrometric endpoint or phenolphthalein endpoint	305.1-Removed	2310 B(4a)	2310 B(4a)	2310 B(4a)-97	D1067-92, 02	I-1020-85 ²
2. Alkalinity , as CaCO ₃ , mg/L	Electrometric or Colorimetric titration to pH 4.5, manual	310.1-Removed	2320 B	2320 B	2320B-97	D1067-92, 02	973.43 ³ I-1030-85 ²
3. Aluminum--Total , ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	202.1-Removed	3111 D		3111 D-99		I-3051-85 ²
	AA furnace	202.2-Removed	3113 B		3113 B-99		
4. Ammonia (as N), mg/L	Manual, distillation (at pH 9.5), ⁶ followed by	350.1, Rev 2.0	4500-NH ₃ B		4500-NH ₃ B-97		973.49 ³
	Nesslerization	350.2-Removed	4500-NH ₃ C (18 th only)			D1426-98, 03 (A)	973.49 ³ I-3520-85 ²
	Titration	350.2-Removed	4500-NH ₃ C (19 th) and 4500-NH ₃ E (18 th)	4500-NH ₃ C	4500-NH ₃ C-97		973.49 ³
	Electrode	350.3-Removed	4500-NH ₃ D or E (19 th) and 4500-NH ₃ F or G (18 th)	4500-NH ₃ D or E	4500-NH ₃ D or E-97	D1426-98, 03 (B)	
5. Antimony--Total , ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	204.1-Removed	3111 B		3111 B -99		
	AA furnace	204.2-Removed	3113 B		3113 B -99		
6. Arsenic--Total , ⁴ mg/L	Digestion ⁴ followed by	206.5 (Issued 1978) ¹					
	AA gaseous hydride	206.3-Removed	3114 B 4.d		3114 B 4.d - 97	D2972-97, 03 (B)	I-3062-85 ²
	AA furnace	206.2-Removed	3113 B		3113 B -99	D2972-97, 03 (C)	I-4063-98 ⁴⁹

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18 th , 19 th)	Std. Methods (20 th)	Std. Methods Online	ASTM	USGS / AOAC / Other
6. Arsenic-Total, ⁴ mg/L (cont'd)	Colorimetric (SDDC)	206.4-Removed	3500-As C	3500-As B	3500-As B-97	D2972-97, 03 (A)	I-3060-85 ²
7. Barium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ¹⁴	208.1-Removed	3111 D		3111D-99		I-3084-85 ²
	AA furnace	208.2-Removed	3113 B		3113 B-99	D4382-95, 02	
8. Beryllium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	210.1-Removed	3111 D		3111D-99	D3645-93 (88), 03 (A)	I-3095-85 ²
	AA furnace	210.2-Removed	3113 B		3113 B-99	D3645-93 (88), 03 (B)	
9. Biochemical oxygen demand (BOD₅), mg/L	Dissolved Oxygen Depletion	405.1-Removed	5210 B	5210 B	5210 B-01		973.44, ³ p. 17. ⁹ I-1578-78 ⁸
10. Boron ³⁷ --Total, mg/L	Colorimetric (curcumin)	212.3-Removed	4500-B B	4500-B B	4500-B B-00		I-3112-85 ²
11. Bromide, mg/L	Titrimetric	320.1-Removed				D1246-95 (C), 99	p. S44. ¹⁰ I-1125-85 ²
12. Cadmium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	213.1-Removed	3111 B or C		3111 B or C-99	D3557-95, 02 (A or B)	974.27 ³ p.37 ⁹ I-3135-85 ² , I-3136-85 ²
	AA furnace	213.2-Removed	3113 B		3111 B-99	D3557-95, 02 (D)	I-4138-89 ⁵¹
13. Calcium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	215.1-Removed	3111 B		3111 B-99	D511-93, 03 (B)	I-3152-85 ²
	Titrimetric (EDTA)	215.2-Removed	3500 Ca D	3500-Ca B	3500-Ca B-97	D511-93, 03 (A)	
14. Carbonaceous biochemical oxygen demand (CBOD₅), mg/L ¹²	Dissolved Oxygen Depletion with nitrification inhibitor	405.1-Removed	5210 B	5210 B	5210 B-01		
15. Chemical oxygen demand (COD), mg/L	Titrimetric	410.1 and 410.2-Removed 410.3 (Rev. 1978)	5220 C	5220 C	5220 C-97	D1252-95, 00 (A)	973.46 ³ p 17 ⁹ I-3560-85 ²
16. Chloride, mg/L	(Mercuric nitrate)	325.3-Removed	4500-Cl ⁻ C	4500-Cl ⁻ C	4500-Cl ⁻ C-97	D512-89, 99 (A)	973.51. ³ I-1184-85 ²
	Automated (Ferricyanide)	325.1 and 325.2-Removed	4500-Cl ⁻ E	4500-Cl ⁻ E	4500-Cl ⁻ E-97		I-2187-85 ²

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
17. Chlorine--Total residual, mg/L	Amperometric direct	330.1-Removed	4500-Cl ⁻ D	4500-Cl ⁻ D	4500-Cl ⁻ D-00	D1253-86 (96), 03	
	Iodometric direct	330.3-Removed	4500-Cl ⁻ B	4500-Cl ⁻ B	4500-Cl ⁻ B-00		
	Back titration either endpoint ¹⁵	330.2-Removed	4500-Cl ⁻ C	4500-Cl ⁻ C	4500-Cl ⁻ C-00		
	DPD-FAS	330.4-Removed	4500-Cl ⁻ F	4500-Cl ⁻ F	4500-Cl ⁻ F-00		
	Spectrophotometric, DPD	330.5-Removed	4500-Cl ⁻ G	4500-Cl ⁻ G	4500-Cl ⁻ G-00		
18. Chromium VI dissolved, mg/L	0.45 micron filtration followed by:						
	AA chelation-extraction or	218.4-Removed	3111 C		3111 C -99		I-1232-85 ²
19. Chromium--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	218.1-Removed	3111 B		3111 B -99	D1687-92, 02 (B)	974.27 ³ I-3236-85 ²
	AA chelation-extraction	218.3-Removed	3111 C		3111 C-99		
	AA furnace	218.2-Removed	3113 B		3113 B-99	D1687-92, 02 (C)	I-3233-93 ⁴⁶
20. Cobalt--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	219.1-Removed	3111B or C		3111B or C-99	D3558-94, 03 (A or B)	p. 37. ⁹ I-3239-85 ²
	AA furnace	219.2-Removed	3113B		3113B-99	D3558-94, 03 (C)	I-4323-89 ⁵¹
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:	Colorimetric (ADMI)	110.1-Removed	2120 E	2120 E			See footnote 18.
	(Platinum cobalt)	110.2-Removed	2120 B	2120 B	2120 B-01		I-1250-85 ²
	Spectrophotometric	110.3-Removed	2120 C	2120 C			
22. Copper--Total,⁴mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	220.1-Removed	3111B or C		3111B or C-99	D1688-95, 02 (A or B)	974.27 ³ p. 37 ⁹ I-3270-85 ² I-3271-85 ²
	AA furnace	220.2-Removed	3113 B		3113 B-99	D1688-95, 02 (C)	I-4274-89 ⁵¹

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
23. Cyanide--Total, mg/L	Manual distillation with MgCl ₂ followed by:						
	Spectrophotometric, manual or	335.2-Removed	4500-CN E	4500-CN E	4500-CN E-99	D2036-98 (A)	I-3300-85 ²
	Automated ²⁰	335.3-Removed 335.4, Rev. 1.0 (1993) ⁵⁷					10-204-00-1-X ⁵⁶ I-4302-85 ²
24. Available Cyanide, mg/L	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl ₂ followed by titrimetric or spectrophotometric	335.1-Removed	4500-CN G	4500-CN G	4500-CN G-99	D2036-98 (B)	
25. Fluoride--Total, mg/L	Manual distillation ⁶ followed by						
	Electrode, manual	340.2-Removed	4500-F C	4500-F C	4500-F C-97	D1179-93, 99 (B)	
	Colorimetric (SPADNS)	340.1-Removed	4500-F D	4500-F D	4500-F D-97	D1179-93, 99 (A)	
	Automated complexone	340.3-Removed	4500-F E	4500-F E	4500-F E-97		
26. Gold--Total,⁴mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	231.1-Removed	3111 B		3111 B-99		
27. Hardness--Total, as CaCO₃, mg/L	Titrimetric (EDTA)	130.2-Removed	2340 B or C	2340 B or C	2340 B or C-97	D1126-92, 02	973.52B. ³ I-1338-85 ²
28. Hydrogen ion (pH), pH units	Electrometric measurement	150.1-Removed	4500-H ⁺ B	4500-H ⁺ B	4500-H ⁺ B-00	D1293-84 (90), 99 (A or B)	973.41. ³ I-1586-85 ²
29. Iridium--Total,⁴mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	235.1-Removed	3111 B		3111 B-99		
30. Iron--Total,⁴mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	236.1-Removed	3111 B or C		3111 B or C-99	D1068-96, 03 (A or B)	974.27. ³ I-3381-85 ²
	AA furnace	236.2-Removed	3113 B		3113 B-99	D1068-96, 03 C	
31. Kjeldahl Nitrogen--Total, (as N), mg/L	Digestion and distillation followed by: ²⁰	351.3-Removed	4500-N _{org} B or C and 4500-NH ₃ B	4500-N _{org} B or C and 4500-NH ₃ B	4500-N _{org} B or C-97 and 4500-NH ₃ B-97	D3590-89, 02 (A)	

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
31. Kjeldahl Nitrogen-- Total, (as N), mg/L (cont'd)	Titration	351.3-Removed	4500-NH ₃ C (19 th) and 4500-NH ₃ E (18 th)	4500-NH ₃ C	4500-NH ₃ C-97	D3590-89, 02 (A)	973.48. ³
	Nesslerization	351.3-Removed	4500-NH ₃ C (18 th only)			D3590-89, 02 (A)	
	Electrode	351.3-Removed	4500-NH ₃ F or G (18 th) and 4500-NH ₃ D or E (19 th)	4500-NH ₃ D or E	4500-NH ₃ D or E-97		
	Manual or block digester potentiometric	351.4-Removed				D3590-89, 02 (A)	
32. Lead--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	239.1-Removed	3111 B or C		3111 B or C-99	D3559-96, 03 (A or B)	974.27. ³ I-3399-85 ²
	AA furnace	239.2-Removed	3113 B		3113 B-99	D3559-96, 03 (D)	I-4403-89 ⁵¹
33. Magnesium-- Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	242.1-Removed	3111 B		3111 B-99	D511-93, 03 (B)	974.27 ³ I-3447-85 ²
34. Manganese-- Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	243.1-Removed	3111 B		3111 B-99	D858-95, 02 (A or B)	974.27. ³ I-3454-85 ²
	AA furnace	243.2-Removed	3113 B		3113 B-99	D858-95, 02 (C)	
36. Molybdenum--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	246.1-Removed	3111 D		3111 D-99		I-3490-85 ²
	AA furnace	246.2-Removed	3113 B		3113 B-99		I-3492-96 ⁴⁷
37. Nickel--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	249.1-Removed	3111 B or C		3111 B or C-99	D1886-90, 94 (98) (A or B)	I-3499-85 ²
	AA furnace	249.2-Removed	3113 B		3113 B-99	D1886-90, 94 (98)(C)	I-4503-89 ⁵¹

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction, Manual	353.3-Removed	4500-NO ₃ E	4500-NO ₃ E	4500-NO ₃ E-00	D3867-99 (B)	
	Automated hydrazine	353.1-Removed	4500-NO ₃ H	4500-NO ₃ H	4500-NO ₃ H-00		
40. Nitrite (as N), mg/L	Spectrophotometric: Manual	354.1-Removed	4500-NO ₂ B	4500-NO ₂ B	4500-NO ₂ B-00		See footnote ²⁵
41. Oil and grease--Total recoverable , mg/L	Gravimetric (extraction): Freon	413.1-Removed	5520 B³⁸-Removed	5520 B³⁸-Removed	5520 B³⁸-Removed		
	Hexane extractable material (HEM): n-Hexane extraction and gravimetry.	1664A ⁴²	5520 B ³⁸	5520 B ³⁸	5520 B-01 ³⁸		
	Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry.	1664A ⁴²					
42. Organic carbon--Total (TOC) , mg/L	Combustion or oxidation	415.1-Removed	5310 B, C, or D	5310 B, C, or D	5310 B, C, or D-00	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)	Methods removed and replacements are listed with items 31 and 4					
44. Orthophosphate (as P), mg/L	Ascorbic acid method						
	Manual single reagent	365.2-Removed	4500-P E	4500-P E		D515-88(A)	973.55 ³
45. Osmium--Total⁴ , mg/L	Digestion ⁴ followed by:						
	AA direct aspiration, or	252.1-Removed	3111 D		3111 D-99		
46. Oxygen , dissolved, mg/L	Winkler (Azide modification), or	360.2-Removed	4500-O C	4500-O C	4500-O C-01	D888-92, 03 (A)	973.4 5B ⁻³ I-1575-78 ⁸
	Electrode	360.1-Removed	4500-O G	4500-O G	4500-O G-01	D888-92, 03 (B)	I-1576-78 ⁸
47. Palladium--Total⁴ , mg/L	Digestion ⁴ followed by:						
	AA direct aspiration, or	253.1-Removed	3111 B		3111 B-99		p. S27 ¹⁰
48. Phenols , mg/L:	Manual distillation ²⁶ Followed by:						
	Automated ¹⁹	420.2-Removed 420.4					

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
50. Phosphorus--Total, mg/L	Persulfate digestion followed by	365.2-Removed	4500-P B.5	4500-P B.5			973.55. ³
	Manual	365.2-Removed 365.3	4500-P E	4500-P E		D515-88(A)	
51. Platinum--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	255.1-Removed	3111 B		3111 B-99		
52. Potassium--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	258.1-Removed	3111 B		3111 B-99		973.53. ³ I-3630-85 ²
53. Residue--Total, mg/L	Gravimetric, 103-105°	160.3-Removed	2540 B	2540 B	2540 B-97		I-3750-85 ²
54. Residue--filterable, mg/L	Gravimetric, 180°	160.1-Removed	2540 C	2540 C	2540 C-97		I-1750-85 ²
55. Residue—non- filterable (TSS), mg/L	Gravimetric, 103-105° post washing of residue	160.2-Removed	2540 D	2540 D	2540 D-97		I-3765-85 ²
56. Residue--settleable, mg/L	Volumetric, (Imhoff cone), or gravimetric	160.5-Removed	2540 F	2540 F	2540 F-97		
58. Rhodium--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration, or	265.1-Removed	3111 B		3111 B-99		
59. Ruthenium--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration, or	267.1-Removed	3111 B		3111 B-99		
60. Selenium--Total,⁴ mg/L	Digestion ⁴ followed by:						
	AA furnace	270.2-Removed	3113 B		3113 B-99	D3859-98, 03 (B)	I-4668-98 ⁴⁹
61. Silica³⁷--Dissolved, mg/L	0.45 micron filtration followed by:						
	Colorimetric, Manual or	370.1-Removed	4500-Si D	4500-SiO ₂ C	4500-SiO ₂ C-97	D859-94, 00	I-1700-85 ²
62. Silver--Total,⁴ mg/L	Digestion ^{4,29} followed by:						
	AA direct aspiration	272.1-Removed	3111 B or C		3111 B or C-99		974.27 ³ p. 37 ⁹ I-3720-85 ²
	AA furnace	272.2-Removed	3113 B		3113 B-99		I-4724-89 ⁵¹

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18 th , 19 th)	Std. Methods (20 th)	Std. Methods Online	ASTM	USGS / AOAC / Other
63. Sodium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	273.1-Removed	3111 B		3111 B-99		973.54 ³ I-3735-85 ²
65. Sulfate (as SO ₄), mg/L	Automated colorimetric	375.1-Removed 375.2					
	Gravimetric	375.3-Removed	4500-SO ₄ ²⁻ C or D	4500-SO ₄ ²⁻ C or D			925.54 ³
65. Sulfate (as SO ₄), mg/L (cont'd)	Turbidimetric	375.4-Removed				D516-90, 02	426C ³⁰
66. Sulfide (as S), mg/L	Titrimetric (iodine), or	376.1-Removed	4500-S ²⁻ F (19 th) or 4500-S ²⁻ E (18 th)	4500-S ²⁻ F	4500-S ²⁻ F- 00		I-3840-85 ²
	Colorimetric (methylene blue)	376.2-Removed	4500-S ²⁻ D	4500-S ²⁻ D	4500-S ²⁻ D- 00		
67. Sulfite (as SO ₃), mg/L	Titrimetric (iodine-iodate)	377.1-Removed	4500-SO ₃ ²⁻ B	4500-SO ₃ ²⁻ B	4500-SO ₃ ²⁻ B-00		
68. Surfactants , mg/L	Colorimetric (methylene blue)	425.1-Removed	5540 C	5540 C	5540 C-00	D2330-88, 02	
69. Temperature , °C	Thermometric	170.1-Removed	2550 B	2550 B	2550 B-00		See footnote 32.
70. Thallium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	279.1-Removed	3111 B		3111 B -99		
71. Tin--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	282.1-Removed	3111 B		3111 B-99		I-3850-78 ⁸
	AA furnace, or	282.2-Removed	3113 B		3113 B-99		
72. Titanium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	283.1-Removed	3111 D		3111 D-99		
74. Vanadium--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration	286.1-Removed	3111 D		3111 D-99		
	AA furnace	286.2-Removed				D3373-93, 03	
75. Zinc--Total, ⁴ mg/L	Digestion ⁴ followed by:						
	AA direct aspiration ³⁶	289.1-Removed	3111 B or C		3111 B or C-99	D1691-95, 02 (A or B)	974.27 ³ p. 37. ⁹ I-3900-85 ²

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18th, 19th)	Std. Methods (20th)	Std. Methods Online	ASTM	USGS / AOAC / Other
Petroleum Hydrocarbons, Total	See TABLE 2- REPLACEMENT OPTIONS FOR PETROLEUM HYDROCARBONS	*418.1-No Longer Approved					
Effected Parameters From the March 12, 2007 USEPA Method Update Rule (MUR) for Chemistry Table 1C							
35. 1,2-Dichlorobenzene	Methodology identified in the next column →	612 and 625- Removed 601, 602-GC 624, 1625B- GC/MS	6410B- Removed 6220 B [18th, 19th] and 6230 B [18th, 19th].	6410B- Removed 6200 C	6200 C-97		See footnote 9, pg. 27
36. 1,3-Dichlorobenzene	Methodology identified in the next column →	612 and 625- Removed 601, 602-GC 624, 1625B- GC/MS	6410B- Removed 6220 B [18th, 19th] and 6230 B [18th, 19th].	6410B- Removed 6200 C	6200 C-97		See footnote 9, pg. 27
37. 1,4-Dichlorobenzene	Methodology identified in the next column →	612 and 625- Removed 601, 602-GC 624, 1625B- GC/MS	6410B- Removed 6220 B [18th, 19th] and 6230 B [18th, 19th].	6410B- Removed 6200 C	6200 C-97		See footnote 9, pg. 27
From the March 26, 2007 USEPA MUR for Biological Pollutants in Wastewater and Sewage Sludge							

Attachment 1

Parameter	Methodology	EPA	Std. Methods (18 th , 19 th)	Std. Methods (20 th)	Std. Methods Online	ASTM	USGS / AOAC / Other
7. Enterococci, number per 100ml ²⁰	MPN ^{7,9} , multiple tube/multiple well		9230B-Removed			D6503-99 ¹⁰	Enterolert ^{® 13,23}
	MF ^{2,6,7,8,9} single step	1600 ²⁴	9230C-Removed				

Note:* Total Petroleum Hydrocarbon testing is not addressed in earlier versions of the Federal Register. Certification for EPA Method 418.1 was specific to NJ testing and reporting only.

March 12, 2007 MUR Table 1B Chemistry Footnotes:

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, Sixteenth Edition, 4th Revision, 1998.

4 For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/111, May, 1994, and is reproduced in Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) use Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.

5 Copper sulfate may be used in place of mercuric sulfate.

6 Manual distillation is not required if comparability data on representative effluent samples are on 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.

Attachment 1

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, April 2, 1975.

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 method 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 a nitrification inhibitor.

13 OIC Chemical Oxygen Demand Method, Oceanography International Corporation.

14 Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company.

15 The back titration method will be used to resolve controversy. Orion Research Instruction Manual, Residual Chlorine Electrode Model 97 - 70, 1977, Orion Research Incorporated. 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, Biocinchonate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company.

20 When using a method with block digestion, this treatment is not required.

21 Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378 - 75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc.

22 Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company.

23 Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117.

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.

Attachment 1

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 For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogeniodide solution by adding 4.0 mL of concentrated NH₄OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I₂ to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH>7 with NH₄OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.

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.

34 "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986–Revised 1991, Thermo Jarrell Ash Corporation, 27 Forge Parkway, Franklin, MA 02038

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 Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", 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 use *n*-hexane extraction solvent when determining Oil and Grease parameters – Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). Use of other extraction solvents (e.g., those in the 18th and 19th editions) is prohibited.

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.

43 USEPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA-821-R-02-024). 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.

Attachment 1

- 44 Available Cyanide, Method OIA-1677, "Available Cyanide by Flow Injection, Logan Exchange, and Amperometry," ALPKEM.
- 45 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method," Open File Report (OFR) 00-170.
- 46 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory –Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 93-449.
- 47 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 97-198.
- 48 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis" Open File Report (OFR) 92-146.
- 49 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry" Open File Report (OFR) 98-639.
- 50 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry," Open File Report (OFR) 98-165.
- 51 "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory –Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment," Open File Report (OFR) 93-125.
- 52 All methods, excluding Method 300.1, are published in "Methods for the Determination of Metals in Environmental Samples," Supplement I, National Exposure Risk Laboratory-Cincinnati (NERL-CI), EPA/600/R-94/111, May 1994; and "Methods for the Determination of Inorganic Substances in Environmental Samples," NERL-CI, EPA/600/R-93/100, August, 1993.
- 53 Styrene divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StablCal™ or equivalent) are acceptable substitutes for formazin.
- 54 Method D6508, Rev. 2, "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte," available from Waters Corp.
- 55 Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821-B-01-009, Revision 1.2, August 2001. Note: A 450-W UV lamp may be used in this method instead of the 550-W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.
- 56 QuikChem Method 10-204-00-1-X, "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis" is available from Lachat Instruments.
- 57 When using sulfide removal test procedures described in Method 335.4, reconstitute particulate that is filtered with the sample prior to distillation.

Attachment 1

58 Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.

59 Method 245.7, Rev. 2.0, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry," February 2005, EPA-821-R-05-001.

60 The use of EDTA may decrease method sensitivity in some samples. Analysts may omit EDTA provided that all method specified quality control acceptance criteria are met.

61 Samples analyzed for available cyanide using Methods OIA-1677 or D6888-04 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample analysis to no more than 30 minutes to preclude settling of materials in samples.

March 12, 2007 MUR Table 1C Chemistry Footnotes:

2 The full text of Methods 601–613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B 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 Procedure for the Determination of the Method Detection Limit," of this Part 136.

7 Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624B, and 1625B (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 ongoing basis must spike and analyze 10% (5% for methods 624 and 625 and 100% for methods 1624B and 1625B) 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. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

9 USGS Method 0–3116–87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments," U.S. Geological Survey, Open File Report 93–125.

March 26, 2007 MUR Biological Footnotes:

2 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.

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 Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

8 When the MF method has been used previously to test waters with high turbidity, large numbers of non-coliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

Attachment 1

⁹ To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

¹⁰ ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

¹³ These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme b-glucuronidase produced by *E. coli*.

²⁰ Recommended for enumeration of target organism in wastewater effluent.

²³ A description of the Enterolert[®] test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

²⁴ USEPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-b-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-009.

TABLE 2-REPLACEMENT OPTIONS FOR PETROLEUM HYDROCARBONS

Petroleum Type	Technology	Method
Gasoline	GC/FID P&T	SW846 8015B GRO
Kerosene	GC/FID	OQA-QAM-025
Diesel Fuel/ Fuel Oil #2	GC/FID Gravimetric Gravimetric Gravimetric- SPE	SW846 8015B DRO 1664A SGT-HEM 45°C 1664A SGT-HEM 85°C 1664A SGT-HEM-SPE
Fuel Oil #4 - #6	GC/FID Gravimetric	OQA-QAM-025 1664A SGT-HEM
Lubricating Oil	Gravimetric	1664A SGT-HEM
Oil exposed to air and heat	Gravimetric	1664A SGT-HEM

Rev. 7/07