



## **Industrial Pretreatment**

### **ANALYTICAL METHODS FOR THE INDUSTRIAL PRETREATMENT PROGRAM**

The User is responsible for insuring that the analytical methods used for pretreatment compliance are one of the methods approved by the United States Environmental Protection Agency (EPA) or the Florida Department of Environmental Protection (DEP).

40 CFR Part 136 identifies analytical methods approved by EPA for industrial pretreatment activities. As indicated in 40 CFR 136.3(a), notices of changes to these procedures will be published in the *Federal Register*. If the User is a "Categorical User", there may also be additional methods referenced in 40 CFR Subchapter N.

Chapter 62-160 F.A.C. identifies additional methods approved by DEP for parameters not found in 40 CFR Part 136.

Attached are the following:

1. Tables 1B, 1C, 1D, and 1F from 40 CFR Part 136.
2. Table 2 from Chapter 62-160 F.A.C.
3. A table listing the analytical methods used by JEA for industrial pretreatment activities.  
Note: Some of the methods used by JEA are alternate methods approved by DEP in accordance with Chapter 62-160 F.A.C.

These attachments are provided for information only and may not be current. Please consult the applicable regulation(s) for further guidance.

**Table IB—List of Approved Inorganic Test Procedures**

Parameter	Methodology <sup>58</sup>	Reference (method number or page)					
		EPA <sup>35,52</sup>	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
1. Acidity, as CaCO <sub>3</sub> , mg/L	Electrometric endpoint or phenolphthalein endpoint		2310 B(4a)	2310 B(4a)	2310 B(4a)-97	D1067-92, 02	I-1020-85 <sup>2</sup>
2. Alkalinity, as CaCO <sub>3</sub> , mg/L	Electrometric or Colorimetric titration to pH 4.5, manual, or		2320 B	2320 B	2320 B-97	D1067-92, 02	973.43 <sup>3</sup> , I-1030-85 <sup>2</sup>
	automatic	310.2 (Rev. 1974) <sup>1</sup>					I-2030-85 <sup>2</sup>
3. Aluminum—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 D		3111 D-99		I-3051-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-9750
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	Direct Current Plasma (DCP) <sup>36</sup>					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Eriochrome cyanine R)		3500-A1 D	3500-A1 B	3500-A1 B-01		
4. Ammonia (as N), mg/L	Manual, distillation (at pH 9.5) <sup>6</sup> followed by:	350.1, Rev. 2.0 (1993)	4500-NH B3	4500-NH3 B	4500-NH3 B-97		973.49 <sup>3</sup>
	Nesslerization		4500-NH3 C (18th only)			D1426-98, 03 (A)	973.49 <sup>3</sup> , I-3520-85 <sup>2</sup>
	Titration		4500-NH3 C (19th) and 4500-NH3 E (18th)	4500-NH3 C	4500-NH3 C-97		
	Electrode		4500-NH3 D or E (19th) and 4500-NH3 F or G (18th)	4500-NH3 D or E	4500-NH3 D or E-97	D1426-98, 03 (B)	
	Automated phenate, or	350.1 <sup>60</sup> , Rev. 2.0 (1993)	4500-NH3 G (19th) and 4500-NH3 H (18th)	4500-NH3 G	4500-NH3 G-97		I-4523-85 <sup>2</sup>
	Automated electrode						See footnote 7
	Ion Chromatography					D6919-03	
5. Antimony—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						

	AA direct aspiration <sup>36</sup>		3111 B		3111 B-99		
	AA furnace		3113 B		3113 B-99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
6. Arsenic— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by	206.5 (Issued 1978) <sup>1</sup>					
	AA gaseous hydride		3114 B 4.d		3114 B 4.d-97	D2972-97, 03 (B)	I-3062-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D2972-97, 03 (C)	I-4063-98 <sup>49</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	Colorimetric (SDDC)		3500-As C	3500-As B	3500-As B-97	D2972-97, 03 (A)	I-3060-85
7. Barium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 D		3111 D-99		I-3084-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D4382-95, 02	
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP <sup>36</sup>						See footnote <sup>34</sup>
8. Beryllium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 D		3111 D-99	D3645-93 (88), 03 (A)	I-3095-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D3645-93 (88), 03 (B)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>

	DCP, or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (aluminon)		3500-Be D				
9. Biochemical oxygen demand (BOD <sub>5</sub> ), mg/L	Dissolved Oxygen Depletion		5210 B	5210 B	5210 B-01		973.44, <sup>3</sup> p. 17. <sup>9</sup> , I-1578-78 <sup>8</sup>
10. Boron—Total, <sup>37</sup> mg/L	Colorimetric (curcumin)		4500-B B	4500-B B	4500-B B-00		I-3112-85 <sup>2</sup>
	ICP/AES, or	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B99		I-4471-97 <sup>50</sup>
	DCP					D4190-94, 99	See footnote 34
11. Bromide, mg/L	Titrimetric					D1246-95, 99 (C)	p. S44. <sup>10</sup>
							I-1125-85 <sup>2</sup>
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
12. Cadmium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D3557-95, 02 (A or B)	974.27, <sup>3</sup> p. 37. <sup>9</sup> , I-3135-85 <sup>2</sup> or I-3136-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D3557-95, 02 (D)	I-4138-89 <sup>51</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-1472-85 <sup>2</sup> or I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP <sup>36</sup>					D4190-94, 99	See footnote <sup>34</sup>
	Voltametry <sup>11</sup> , or					D3557-95, 02 (C)	
	Colorimetric (Dithizone)		3500-Cd D				
13. Calcium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99	D511-93, 03(B)	I-3152-85 <sup>2</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	DCP, or						See footnote <sup>34</sup>
	Titrimetric (EDTA)		3500-Ca D	3500-Ca B	3500-Ca	D511-93,	

					B-97	03(A)	
	Ion Chromatography					D6919-03	
14. Carbonaceous biochemical oxygen demand (CBOD <sub>5</sub> ), mg/L <sup>12</sup>	Dissolved Oxygen Depletion with nitrification inhibitor		5210 B	5210 B	5210 B-01		
15. Chemical oxygen demand (COD), mg/L	Titrimetric	410.3 (Rev. 1978) <sup>1</sup>	5220 C	5220 C	5220 C-97	D1252-95, 00 (A)	973.46 <sup>3</sup> , p. 17 <sup>9</sup> I-3560-85 <sup>2</sup>
	Spectrophotometric, manual or automatic	410.4, Rev. 2.0 (1993)	5220 D	5220 D	5220 D-97	D1252-95, 00 (B)	See footnotes <sup>13,14</sup> . I-3561-85 <sup>2</sup>
16. Chloride, mg/L	Titrimetric: (silver nitrate) or		4500-CI-B	4500-CI-B	4500-CI-B-97	D512-89(99) (B)	I-1183-85 <sup>2</sup>
	(Mercuric nitrate)		4500-CI-C	4500-CI-C	4500-CI-C-97	D512-89(99) (A)	973.51 <sup>3</sup> , I-1184-85 <sup>2</sup>
	Colorimetric: manual or						I-1187-85 <sup>2</sup>
	Automated (Ferricyanide)		4500-CI-E	4500-CI-E	4500-CI-E-97		I-2187-85 <sup>2</sup>
	Potentiometric Titration		4500-CI-D	4500-CI-D	4500-CI-D-97		
	Ion Selective Electrode					D512-89(99)(C)	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
17. Chlorine—Total residual, mg/L; Titrimetric	Amperometric direct, or		4500-CI D	4500-CI D	4500-CI D-00	D1253-86(96), 03	
	Amperometric direct (low level)		4500-CI E	4500-CI E	4500-CI E-00		
	Iodometric direct		4500-CI B	4500-CI B	4500-CI B-00		
	Back titration ether endpoint <sup>15</sup> or		4500-CI C	4500-CI C	4500-CI C-00		
	DPD-FAS		4500-CI F	4500-CI F	4500-CI F-00		
	Spectrophotometric, DPD or		4500-CI G	4500-CI G	4500-CI G-00		
	Electrode						See footnote <sup>16</sup>
18. Chromium VI dissolved, mg/L	0.45-micron Filtration followed by:						
	AA chelation-extraction or		3111 C		3111 C-99		I-1232-85
	Ion Chromatography	218.6, Rev. 3.3 (1994)	3500-Cr E	3500-Cr C	3500-Cr C-01	D5257-97	993.23

	Colorimetric (Diphenyl-carbazide)		3500-Cr D	3500-Cr B	3500-Cr B-01	D1687-92, 02 (A)	I-1230-85
19. Chromium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B		3111 B-99	D1687-92, 02 (B)	974.27 <sup>3</sup> , I-3236-85 <sup>2</sup>
	AA chelation-extraction		3111 C		3111 C-99		
	AA furnace		3113 B		3113 B-99	D1687-92, 02 (C)	I-3233-93 <sup>46</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP, <sup>36</sup> or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Diphenyl-carbazide)		3500-Cr D	3500-Cr B	3500-Cr B-01		
20. Cobalt—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B or C		3111 B or C-99	D3558-94, 03 (A or B)	p. 37 <sup>9</sup> , I-3239-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D3558-94, 03 (C)	I-4243-89 <sup>51</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP					D4190-94, 99	See footnote <sup>34</sup>
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity	Colorimetric (ADMI), or		2120 E	2120 E			See footnote <sup>18</sup>
	(Platinum cobalt), or		2120 B	2120 B	2120 B-01		I-1250-85 <sup>2</sup>
	Spectrophotometric		2120 C	2120 C			
22. Copper—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D1688-95, 02 (A or B)	974.27 <sup>3</sup> p. 37 <sup>9</sup> I-3270-85 <sup>2</sup> or I-3271-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D1688-95,	I-4274-89 <sup>51</sup>

						02 (C)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP <sup>36</sup> or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Neocuproine) or		3500-Cu D	3500-Cu B	3500-Cu B-99		
	(Bicinchoninate)		3500-Cu E	3500-Cu C	3500-Cu C-99		See footnote <sup>19</sup>
23. Cyanide—Total, mg/L	Automated Distillation and Colorimetry, or						Kelada-01 <sup>55</sup>
	Manual distillation with MgCl <sub>2</sub> followed by:	335.4, Rev. 1.0 (1993) <sup>57</sup>	4500-CN <sup>-</sup> C	4500-CN <sup>-</sup> C		D2036-98(A)	10-204-00-1-X <sup>56</sup>
	Titrimetric or		4500-CN <sup>-</sup> D	4500-CN <sup>-</sup> D	4500-CN <sup>-</sup> D-99		p. 22 <sup>9</sup>
	Spectrophotometric, manual or		4500-CN <sup>-</sup> E	4500-CN <sup>-</sup> E	4500-CN <sup>-</sup> E-99	D2036-98(A)	I-3300-85
	Automated <sup>20</sup> or	335.4, Rev. 1.0 (1993) <sup>57</sup>					10-204-00-1-X <sup>56</sup> , I-4302-85 <sup>2</sup>
	Ion Selective Electrode		4500-CN <sup>-</sup> F	4500-CN <sup>-</sup> F	4500-CN <sup>-</sup> F-99	D2036-98(A)	
24. Available Cyanide, mg/L	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl <sub>2</sub> followed by Titrimetric or Spectrophotometric		4500-CN <sup>-</sup> G	4500-CN <sup>-</sup> G	4500-CN <sup>-</sup> G-99	D2036-98(B)	
	Flow injection and ligand exchange, followed by amperometry <sup>61</sup>					D6888-04	OIA-1677 <sup>44</sup>
	Automated Distillation and Colorimetry						Kelada-01 <sup>55</sup>
25. Fluoride—Total, mg/L	Manual distillation <sup>6</sup> followed by:		4500-F B	4500-F B	4500-F B-97		
	Electrode, manual or		4500-F B	4500-F B	4500-F C-97	D1179-93, 99 (B)	
	Automated						I-4327-85 <sup>2</sup>
	Colorimetric, (SPADNS) or		4500-F D	4500-F D	4500-F D-97	D1179-93, 99 (A)	
	Automated complexone		4500-F E	4500-F E	4500-F E-97		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0	4110 B	4110 B	4110 B-00	D4327-97,03	993.30 <sup>3</sup>

		(1997)					
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
26. Gold— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration, or		3111 B		3111 B-99		
	AA furnace, or	231.2 (Rev. 1978) <sup>1</sup>					
	DCP						See footnote <sup>34</sup>
27. Hardness—Total, as CaCO <sub>3</sub> , mg/L	Automated colorimetric,	130.1 (Issued 1971) <sup>1</sup>					
	Titrimetric (EDTA) or		2340 B or C	2340 B or C	2340 B or C-97	D1126-86(92), 02	973.5 2B <sup>3</sup> , I-1338-85 <sup>2</sup>
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).						
28. Hydrogen ion (pH), pH units	Electrometric measurement or		4500-H <sup>+</sup> B	4500-H <sup>+</sup> B	4500-H <sup>+</sup> B-00	D1293-84(90), 99 (A or B)	973.41. <sup>3</sup> , I-1586-85 <sup>2</sup>
	Automated electrode	150.2 (Dec. 1982) <sup>1</sup>					See footnote <sup>21</sup> , I-2587-85 <sup>2</sup>
29. Iridium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration or		3111 B		3111 B-99		
	AA furnace	235.2 (Issued 1978) <sup>1</sup>					
30. Iron— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D1068-96, 03 (A or B)	974.27 <sup>3</sup> , I-3381-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D1068-96, 03 (C)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	DCP <sup>36</sup> or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Phenanthroline)		3500-Fe D	3500-Fe B	3500-Fe B-97	D1068-96, 03 (D)	See footnote <sup>22</sup>
31. Kjeldahl Nitrogen <sup>5</sup> —Total, (as N), mg/L	Digestion and distillation followed by: <sup>20</sup>		4500-Norg B or C and 4500-NH <sub>3</sub> B	4500-Norg B or C and 4500-NH <sub>3</sub> B	4500-Norg B or C-97 and 4500-NH <sub>3</sub> B-97	D3590-89, 02 (A)	
	Titration or		4500-NH <sub>3</sub>	4500-NH <sub>3</sub>	4500-NH <sub>3</sub>	D3590-89,	973.48 <sup>3</sup>



			C (19th) and 4500-NH3 E (18th)	C	C-97	02 (A)	
	Nesslerization or		4500-NH3 C (18th Only)			D3590-89, 02 (A)	
	Electrode		4500-NH3 F or G (18th) and 4500-NH3 D or E (19th)	4500-NH3 D or E	4500-NH3 D or E-97		
	Automated phenate colorimetric	351.1 (Rev. 1978) <sup>1</sup>					I-4551-78 <sup>8</sup>
	Semi-automated block digester colorimetric	351.2, Rev. 2.0 (1993)				D3590-89, 02 (B)	I-4515-91 <sup>45</sup>
	Manual or block digester potentiometric					D3590-89, 02 (A)	
	Block digester, followed by Auto distillation and Titration, or						See footnote <sup>39</sup>
	Nesslerization, or						See footnote <sup>40</sup>
	Flow injection gas diffusion						See footnote <sup>41</sup>
32. Lead— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D3559-96, 03 (A or B)	974.27 <sup>3</sup> , I-3399-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D3559-96, 03 (D)	I-4403-89 <sup>51</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP <sup>36</sup>					D4190-94, 99	See footnote <sup>34</sup>
	Voltametry <sup>11</sup> or					D3559-96, 03 (C)	
	Colorimetric (Dithizone)		3500-Pb D	3500-Pb B	3500-Pb B-97		
33. Magnesium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99	D511-93, 03(B)	974.27 <sup>3</sup> , I-3447-85 <sup>2</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	DCP or						See footnote <sup>34</sup>

	Gravimetric		3500—Mg D				
	Ion Chromatography					D6919-03	
34. Manganese— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B		3111 B-99	D858-95, 02 (A or B)	974.27 <sup>3</sup> , I-3454-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D858-95, 02 (C)	
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP36, or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Persulfate), or		3500—Mn D	3500—Mn B	3500—Mn B-99		920.203 <sup>3</sup>
	(Periodate)						See footnote <sup>23</sup>
35. Mercury—Total <sup>4</sup> , mg/L	Cold vapor, manual or	245.1, Rev. 3.0 (1994)	3112 B		3112 B-99	D3223-97, 02	977.22 <sup>3</sup> , I-3462-85 <sup>2</sup>
	Automated	245.2 (Issued 1974)					
	Cold vapor atomic fluorescence spectrometry (CVAFS)	245.7 Rev. 2.0 (2005) <sup>59</sup>					
	Purge and Trap CVAFS	1631E <sup>43</sup>					
36. Molybdenum— Total <sup>4</sup> , mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 D		3111 D-99		I-3490-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99		I-3492-96 <sup>47</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP						See footnote <sup>34</sup>
37. Nickel— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D1886-90, 94 (98) (A or B)	I-3499-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99	D1886-90, 94 (98) (C)	I-4503-89 <sup>51</sup>
	STGFAA	200.9, Rev. 2.2					

		(1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP <sup>36</sup> , or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (heptoxime)		3500-Ni D (17th Edition)				
38. Nitrate (as N), mg/L	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
	Ion Selective Electrode		4500-NO <sub>3</sub> <sup>-</sup> -D	4500-NO <sub>3</sub> <sup>-</sup> -D	4500-NO <sub>3</sub> <sup>-</sup> -D-00		
	Colorimetric (Brucine sulfate), or	352.1 <sup>1</sup>					973.50 <sup>3</sup> , 419D <sup>1,7</sup> , p. 28 <sup>9</sup>
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).						
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction, manual or		4500-NO <sub>3</sub> <sup>-</sup> -E	4500-NO <sub>3</sub> <sup>-</sup> -E	4500-NO <sub>3</sub> <sup>-</sup> -E-00	D3867-99(B)	
	Automated, or	353.2, Rev. 2.0 (1993)	4500-NO <sub>3</sub> <sup>-</sup> -F	4500-NO <sub>3</sub> <sup>-</sup> -F	4500-NO <sub>3</sub> <sup>-</sup> -F-00	D3867-99(A)	I-4545-85 <sup>2</sup>
	Automated hydrazine		4500-NO <sub>3</sub> <sup>-</sup> -H	4500-NO <sub>3</sub> <sup>-</sup> -H	4500-NO <sub>3</sub> <sup>-</sup> -H-00		
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
40. Nitrite (as N), mg/L	Spectrophotometric: Manual or		4500-NO <sub>2</sub> <sup>-</sup> -B	4500-NO <sub>2</sub> <sup>-</sup> -B	4500-NO <sub>2</sub> <sup>-</sup> -B-00		See footnote <sup>25</sup>
	Automated (Diazotization)						I-4540-85 <sup>2</sup>
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)	4500-NO <sub>3</sub> <sup>-</sup> -F	4500-NO <sub>3</sub> <sup>-</sup> -F	4500-NO <sub>3</sub> <sup>-</sup> -F-00	D3867-99(A)	I-4545-85 <sup>2</sup>
	Manual (*bypass cadmium reduction)		4500-NO <sub>3</sub> <sup>-</sup> -E	4500-NO <sub>3</sub> <sup>-</sup> -E	4500-NO <sub>3</sub> <sup>-</sup> -E-00	D3867-99(B)	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev.2 <sup>54</sup>
41. Oil and grease—Total recoverable, mg/L	Hexane extractable material (HEM): n-Hexane extraction and gravimetry	1664A <sup>42</sup>		5520 B <sup>38</sup>	5520 B-01 <sup>38</sup>		

	Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry.	1664A <sup>42</sup>					
42. Organic carbon—Total (TOC), mg/L	Combustion or oxidation		5310 B, C, or D	5310 B, C, or D	5310 B, C, or D-00	D2579-93 (A or B)	973.47, <sup>3</sup> p. 14 <sup>24</sup>
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, Rev. 2.0 (1993)	4500-P F	4500-P F			973.56 <sup>3</sup> , I-4601-85 <sup>2</sup>
	Manual single reagent		4500-P E	4500-P E		D515-88(A)	973.55 <sup>3</sup>
	Manual two reagent	365.3 (Issued 1978) <sup>1</sup>					
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
45. Osmium—Total <sup>4</sup> , mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration, or		3111 D		3111 D-99		
	AA furnace	252.2 (Issued 1978) <sup>1</sup>					
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or		4500-O C	4500-O C	4500-O C-01	D888-92, 03 (A)	973.4 5B <sup>3</sup> , I-1575-78 <sup>8</sup>
	Electrode		4500-O G	4500-O G	4500-O G-01	D888-92, 03 (B)	I-1576-78 <sup>8</sup>
47. Palladium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration, or		3111 B		3111 B-99		p. S27 <sup>10</sup>
	AA furnace	253.2 <sup>1</sup> (Issued 1978)					p. S28 <sup>10</sup>
	DCP						See footnote <sup>34</sup>
48. Phenols, mg/L	Manual distillation <sup>26</sup> Followed by:	420.1 <sup>1</sup> (Rev. 1978)					See footnote <sup>27</sup>
	Colorimetric (4AAP) manual, or	420.1 <sup>1</sup> (Rev. 1978)					See footnote <sup>27</sup>
	Automated	420.4 Rev. 1.0 (1993)					
49. Phosphorus (elemental), mg/L	Gas-liquid chromatography						See footnote <sup>28</sup>
50. Phosphorus—Total, mg/L	Persulfate digestion followed by: <sup>20</sup>		4500-P B.5	4500-P B.5			973.55 <sup>3</sup>

	Manual or	365.3 <sup>1</sup> (Issued 1978)	4500-P E	4500-P E		D515-88(A)	
	Automated ascorbic acid reduction	365.1 Rev. 2.0 (1993)	4500-P F	4500-P F			973.56 <sup>3</sup> , I-4600-85 <sup>2</sup>
	Semi-automated block digester	365.4 <sup>1</sup> (Issued 1974)				D515-88(B)	I-4610-91 <sup>48</sup>
51. Platinum— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99		
	AA furnace	255.2 <sup>1</sup>					
	DCP						See footnote <sup>34</sup>
52. Potassium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99		973.53 <sup>3</sup> , I-3630-85 <sup>2</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	Flame photometric, or		3500-K D	3500-K B	3500-K B-97		
	Colorimetric						317 B <sup>17</sup>
	Ion Chromatography					D6919-03	
53. Residue—Total, mg/L	Gravimetric, 103-105°		2540 B	2540 B	2540 B-97		I-3750-85 <sup>2</sup>
54. Residue— filterable, mg/L	Gravimetric, 180°		2540 C	2540 C	2540 C-97		I-1750-85 <sup>2</sup>
55. Residue—non- filterable (TSS), mg/L	Gravimetric, 103-105 °C post washing of residue		2540 D	2540 D	2540 D-97		I-3765-85 <sup>2</sup>
56. Residue— settleable, mg/L	Volumetric, (Imhoff cone), or gravimetric		2540 F	2540 F	2540 F-97		
57. Residue— Volatile, mg/L	Gravimetric, 550 °C	160.4 <sup>1</sup>					I-3753-85 <sup>2</sup>
58. Rhodium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration, or		3111 B		3111 B-99		
	AA furnace	265.2 <sup>1</sup>					
59. Ruthenium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration, or		3111 B		3111 B-99		
	AA furnace	267.2 <sup>1</sup>					
60. Selenium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA furnace		3113 B		3113 B-99	D3859-98, 03 (B)	I-4668-98 <sup>49</sup>
	STGFAA	200.9, Rev. 2.2					

		(1994)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	AA gaseous hydride		3114 B		3114 B-97	D3859-98, 03 (A)	I-3667-85 <sup>2</sup>
61. Silica— Dissolved, <sup>37</sup> mg/L	0.45 micron filtration followed by:						
	Colorimetric, Manual or		4500-Si D	4500-SiO2 C	4500-SiO2C-97	D859-94, 00	I-1700-85 <sup>2</sup>
	Automated (Molybdosilicate), or						I-2700-85 <sup>2</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
62. Silver—Total, <sup>4, 31</sup> mg/L	Digestion <sup>4, 29</sup> followed by:						
	AA direct aspiration		3111 B or C		3111 B or C-99		974.27 <sup>3</sup> , p. 37 <sup>9</sup> , I-3720-85 <sup>2</sup>
	AA furnace		3113 B		3113 B-99		I-4724-89 <sup>51</sup>
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP						See footnote <sup>34</sup>
63. Sodium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99		973.54 <sup>3</sup> , I-3735-85 <sup>2</sup>
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	DCP, or						See footnote <sup>34</sup>
	Flame photometric		3500-Na D	3500-Na B	3500-Na B-97		
	Ion Chromatography					D 6919-03	
64. Specific conductance, micromhos/cm at 25 °C	Wheatstone bridge	120.1 <sup>1</sup> (Rev. 1982)	2510 B	2510 B	2510 B-97	D1125-95 (99) (A)	973.40 <sup>3</sup> , I-2781-85 <sup>2</sup>
65. Sulfate (as SO4), mg/L	Automated colorimetric	375.2, Rev. 2.0 (1993)					
	Gravimetric		4500-SO4 <sup>2-</sup> C or D	4500-SO4 <sup>2-</sup> C or D			925.54 <sup>3</sup>

	Turbidimetric					D516-90, 02	426C <sup>30</sup>
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B-00	D4327-97, 03	993.30 <sup>3</sup>
	CIE/UV						D6508, Rev. 2 <sup>54</sup>
66. Sulfide (as S), mg/L	Titrimetric (iodine), or		4500-S <sup>2</sup> -F (19th) 4500-S <sup>2</sup> -E (18th)	4500-S <sup>2</sup> -F	4500-S <sup>2</sup> -F-00		I-3840-85 <sup>2</sup>
	Colorimetric (methylene blue)		4500-S <sup>2</sup> -D	4500-S <sup>2</sup> -D	4500-S <sup>2</sup> -D-00		
	Ion Selective Electrode		4500-S <sup>2</sup> -G	4500-S <sup>2</sup> -G	4500-S <sup>2</sup> -G-00	D4658-03	
67. Sulfite (as SO <sub>3</sub> ), mg/L	Titrimetric (iodine-iodate)		4500-SO <sub>3</sub> <sup>2-</sup> -B	4500-SO <sub>3</sub> <sup>2-</sup> -B	4500-SO <sub>3</sub> <sup>2-</sup> -B-00		
68. Surfactants, mg/L	Colorimetric (methylene blue)		5540 C	5540 C	5540 C-00	D2330-88, 02	
69. Temperature, °C	Thermometric		2550 B	2550 B	2550 B-00		See footnote <sup>32</sup>
70. Thallium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99		
	AA furnace	279.2 <sup>1</sup> (Issued 1978)					
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
71. Tin—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 B		3111 B-99		I-3850-78 <sup>8</sup>
	AA furnace, or		3113 B		3113 B-99		
	STGFAA	200.9, Rev. 2.2 (1994)					
	ICP/AES	200.7, Rev. 4.4 (1994)					
72. Titanium—Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 D		3111 D-99		
	AA furnace	283.2 <sup>1</sup> (Issued 1978)					
	DCP						See footnote <sup>34</sup>
73. Turbidity, NTU <sup>53</sup>	Nephelometric	180.1, Rev. 2.0 (1993)	2130 B	2130 B	2130 B-01	D1889-94, 00	I-3860-85 <sup>2</sup>

74. Vanadium— Total, <sup>4</sup> mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration		3111 D		3111 D-99		
	AA furnace					D3373-93, 03	
	ICP/AES	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B-99		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP, or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Gallic Acid)		3500-V D	3500-V B	3500-V B- 97		
75. Zinc –Total <sup>4</sup> , mg/L	Digestion <sup>4</sup> followed by:						
	AA direct aspiration <sup>36</sup>		3111 B or C		3111 B or C-99	D1691-95, 02 (A or B)	974.27 <sup>3</sup> , p. 37 <sup>9</sup> , I- 3900-85 <sup>2</sup>
	AA furnace	289.2 <sup>1</sup> (Issued 1978)					
	ICP/AES <sup>36</sup>	200.7, Rev. 4.4 (1994)	3120 B	3120 B	3120 B- 99 <sup>59</sup>		I-4471-97 <sup>50</sup>
	ICP/MS	200.8, Rev. 5.4 (1994)				D5673-03	993.14 <sup>3</sup>
	DCP, <sup>36</sup> or					D4190-94, 99	See footnote <sup>34</sup>
	Colorimetric (Dithizone) or		3500-Zn E				
	(Zincon)		3500-Zn F	3500-Zn B	3500-Zn B-97		See footnote <sup>33</sup>

**Table 1B Notes:**

<sup>1</sup>“Methods for Chemical Analysis of Water and Wastes,” Environmental Protection Agency, Environmental Monitoring Systems Laboratory–Cincinnati (EMSL-CI), EPA-600/4-79-020 (NTIS PB 84-128677), Revised March 1983 and 1979 where applicable.

<sup>2</sup>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.

<sup>3</sup>“Official Methods of Analysis of the Association of Official Analytical Chemists,” Methods Manual, Sixteenth Edition, 4th Revision, 1998.

<sup>4</sup>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 EPA 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 EPA 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 EPA 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.

<sup>5</sup>Copper sulfate may be used in place of mercuric sulfate.

<sup>6</sup>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.

<sup>7</sup>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.

<sup>8</sup>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).

<sup>9</sup>American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd st., New York, NY 10036.

<sup>10</sup>"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).

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

<sup>12</sup>Carbonaceous biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional BOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required can the permittee report data using a nitrification inhibitor.

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

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

<sup>15</sup>The back titration method will be used to resolve controversy.

<sup>16</sup>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.

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

<sup>18</sup>National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

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

<sup>20</sup>When using a method with block digestion, this treatment is not required.

<sup>21</sup>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.

<sup>22</sup>Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>23</sup>Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2–113 and 2–117, Hach Chemical Company, Loveland, CO 80537.

<sup>24</sup>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.

<sup>25</sup>Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>26</sup>Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

<sup>27</sup>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 spectrometric procedure.

<sup>28</sup>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.

<sup>29</sup>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<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

<sup>30</sup>The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition.

<sup>31</sup>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 cyanogen iodide solution by adding 4.0 mL of concentrated NH<sub>4</sub>OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I<sub>2</sub> 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 NH<sub>4</sub>OH. 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.

<sup>32</sup>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.

<sup>33</sup>Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2–231 and 2–333, Hach Chemical Company, Loveland, CO 80537.

<sup>34</sup>"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

<sup>35</sup>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."

<sup>36</sup>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, P.O. Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.

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

<sup>38</sup>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.

<sup>39</sup>Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

<sup>40</sup>Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

<sup>41</sup>Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.

<sup>42</sup>Method 1664, Revision A “ *n* -Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n* -Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry” EPA-821-R-98-002, February 1999. Available at NTIS, PB-121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.

<sup>43</sup>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.

<sup>44</sup>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.

<sup>45</sup>“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.

<sup>46</sup>“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.

<sup>47</sup>“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.

<sup>48</sup>“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.

<sup>49</sup>“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.

<sup>50</sup>“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.

<sup>51</sup>“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.

<sup>52</sup>All EPA methods, excluding EPA 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. EPA Method 300.1 is available from <http://www.epa.gov/safewater/methods/pdfs/met300.pdf>.

<sup>53</sup>Styrene divinyl benzene beads (e.g., AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g., Hach StabCal™ or equivalent) are acceptable substitutes for formazin.

<sup>54</sup>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, 34 Maple St., Milford, MA, 01757, Telephone: 508/482-2131, Fax: 508/482-3625.

<sup>55</sup>Kelada-01, “Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate,” EPA 821-B-01-009, Revision 1.2, August 2001, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001-108275]. The toll free telephone number is: 800-553-6847. 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.

<sup>56</sup>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 6645 W. Mill Road, Milwaukee, WI 53218, Telephone: 414-358-4200.

<sup>57</sup>When using sulfide removal test procedures described in Method 335.4, reconstitute particulate that is filtered with the sample prior to distillation.

<sup>58</sup>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.

<sup>59</sup>Method 245.7, Rev. 2.0, “Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry,” February 2005, EPA-821-R-05-001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304, Telephone: 703-461-2100, Fax: 703-461-8056.

<sup>60</sup>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.

<sup>61</sup>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.

**Table IC—List of Approved Test Procedures for Non-Pesticide Organic Compounds**

Parameter <sup>1</sup>	EPA method number <sup>2,7</sup>			Other approved methods			
	GC	GC/MS	HPLC	Standard Methods [Edition(s)]	Standard Methods Online	ASTM	Other
1. Acenaphthene	610	625, 1625B	610	6440 B [18th, 19th, 20th]		D4657-92 (99)	See footnote <sup>9</sup> , p. 27
2. Acenaphthylene	610	625, 1625B	610	6410 B, 6440 B, [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
3. Acrolein	603	624 <sup>4</sup> , 1624B					
4. Acrylonitrile	603	624 <sup>4</sup> , 1624B					
5. Anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
6. Benzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th]	6200 B and C-97		
7. Benzidine		625 <sup>5</sup> , 1625B	605				See footnote <sup>3</sup> , p.1
8. Benzo(a)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
9. Benzo(a)pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
10. Benzo(b)fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
11. Benzo(g,h,i) perylene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
12. Benzo(k) fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
13. Benzyl chloride							See footnote <sup>3</sup> , p. 130: See footnote <sup>6</sup> , p. S102

14. Benzyl butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
15. Bis(2-chloroethoxy) methane	611	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
16. Bis(2-chloroethyl) ether	611	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
17. Bis(2-ethylhexyl) phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
18. Bromodichloro-methane	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th]	6200 B and C-97		
19. Bromoform	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th]	6200 B and C-97		
20. Bromomethane	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th], 6200 B [20th] and 6210 B [18th, 19th]	6200 B and C-97		
21. 4-Bromophenyl phenyl ether	611	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
22. Carbon tetrachloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		See footnote <sup>3</sup> , p. 130
23. 4-Chloro-3-methyl phenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
24. Chlorobenzene	601, 602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		See footnote <sup>3</sup> , p. 130
25. Chloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
26. 2-Chloroethylvinyl ether	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
27. Chloroform	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		See footnote <sup>3</sup> , p. 130
28. Chloromethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
29. 2-Chloronaphthalene	612	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
30. 2-Chlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B(00, 6420 B-00		See footnote <sup>9</sup> , p. 27
31. 4-Chlorophenyl phenyl ether	611	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
32. Chrysene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
33. Dibenzo(a,h)anthracene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
34. Dibromochloro-methane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th] 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		

35. 1,2-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		See footnote <sup>9</sup> , p. 27
36. 1,3-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		See footnote <sup>9</sup> , p. 27
37. 1,4-Dichloro-benzene	601, 602	624, 1625B		6200 C [20th] and 6220 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		See footnote <sup>9</sup> , p. 27
38. 3,3-Dichloro-benzidine		625, 1625B	605	6410 B [18th, 19th, 20th]	6410 B-00		
39. Dichlorodifluoro-methane	601			6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		
40. 1,1-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
41. 1,2-Dichloroethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
42. 1,1-Dichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
43. trans-1,2-Dichloro-ethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
44. 2,4-Dichlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
45. 1,2-Dichloro-propane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
46. cis-1,3-Dichloro-propene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
47. trans-1,3-Dichloro-propene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		
48. Diethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
49. 2,4-Dimethylphenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
50. Dimethyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
51. Di-n-butyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
52. Di-n-octyl phthalate	606	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
53. 2,3-Dinitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		
54. 2,4-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
55. 2,6-Dinitrotoluene	609	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27

56. Epichlorohydrin							See footnote <sup>3</sup> , p. 130; See footnote <sup>6</sup> , p. S102
57. Ethylbenzene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th]	6200 B and C-97		
58. Fluoranthene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
59. Fluorene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
60. 1,2,3,4,6,7,8-Heptachloro-dibenzofuran		1613B <sup>10</sup>					
61. 1,2,3,4,7,8,9-Heptachloro-dibenzofuran		1613B <sup>10</sup>					
62. 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin		1613B <sup>10</sup>					
63. Hexachlorobenzene	612	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
64. Hexachloro-butadiene	612	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
65. Hexachlorocyclo-pentadiene	612	625 <sup>5</sup> , 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
66. 1,2,3,4,7,8-Hexachlorodibenzofuran		1613B <sup>10</sup>					
67. 1,2,3,6,7,8-Hexachlorodibenzofuran		1613B <sup>10</sup>					
68. 1,2,3,7,8,9-Hexachlorodibenzofuran		1613B <sup>10</sup>					
69. 2,3,4,6,7,8-Hexachlorodibenzofuran		1613B <sup>10</sup>					
70. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin		1613B <sup>10</sup>					
71. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin		1613B <sup>10</sup>					
72. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin		1613B <sup>10</sup>					
73. Hexachloroethane	612	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
74. Ideno(1,2,3-cd) pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
75. Isophorone	609	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
76. Methylene chloride	601	624, 1624B		6200 C [20th] and 6230 B [18th, 19th]	6200 C-97		See footnote <sup>3</sup> , p. 130
77. 2-Methyl-4,6-dinitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
78. Naphthalene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27

79. Nitrobenzene	609	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
80. 2-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
81. 4-Nitrophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
82. N-Nitrosodimethylamine	607	6255, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
83. N-Nitrosodi-n-propylamine	607	6255, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
84. N-Nitrosodiphenylamine	607	6255, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>9</sup> , p. 27
85. Octachlorodibenzofuran		1613B <sup>10*</sup>					
86. Octachlorodibenzo-p-dioxin		1613B <sup>10</sup>					
87. 2,2'-Oxybis(2-chloropropane) [also known as bis(2-chloroisopropyl) ether]	611	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00		
88. PCB-1016	608	625		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 43; See footnote <sup>8</sup>
89. PCB-1221	608	625		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 43; See footnote <sup>8</sup>
90. PCB-1232	608	625		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 43; See footnote <sup>8</sup>
91. PCB-1242	608	625		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 43; See footnote <sup>8</sup>
92. PCB-1248	608	625					
93. PCB-1254	608	625		6410 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 43; See footnote <sup>8</sup>
94. PCB-1260	608	625		6410 B, 6630 B [18th, 19th, 20th]	6410 B-00		See footnote 3, p. 43; See footnote 8
95. 1,2,3,7,8-Pentachloro- dibenzofuran		1613B <sup>10</sup>					
96. 2,3,4,7,8-Pentachloro- dibenzofuran		1613B <sup>10</sup>					
97. 1,2,3,7,8,-Pentachlorodibenzo- <i>p</i> -dioxin		1613B <sup>10</sup>					
98. Pentachlorophenol	604	625, 1625B		6410 B, 6630 B [18th, 19th, 20th]	6410 B-00		See footnote <sup>3</sup> , p. 140; See footnote <sup>9</sup> , p. 27
99. Phenanthrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27
100. Phenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00		See footnote <sup>9</sup> , p. 27
101. Pyrene	610	625, 1625B	610	6410 B, 6440 B [18th, 19th, 20th]	6410 B-00	D4657-92 (99)	See footnote <sup>9</sup> , p. 27



102. 2,3,7,8-Tetra-chlorodibenzofuran		1613B <sup>10</sup>				
103. 2,3,7,8-Tetra-chlorodibenzo-p-dioxin		613, 625 <sup>5a</sup> , 1613B <sup>10</sup>				
104. 1,1,2,2-Tetra-chloro ethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	See footnote <sup>3</sup> , p. 130
105. Tetrachloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	See footnote <sup>3</sup> , p. 130
106. Toluene	602	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6220 B [18th, 19th]	6200 B and C-97	
107. 1,2,4-Trichloro-benzene	612	625, 1625B		6410 B [18th, 19th, 20th]	6410 B-00	See footnote <sup>3</sup> , p. 130; See footnote <sup>9</sup> , p. 27
108. 1,1,1-Trichloro-ethane	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	
109. 1,1,2-Trichloro-ethane	601	624, 1624B	6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97		See footnote <sup>3</sup> , p. 130
110. Trichloroethene	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	
111. Trichlorofluoro-methane	601	624		6200 B [20th] and 6210 B [18th, 19th], 6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	
112. 2,4,6-Trichlorophenol	604	625, 1625B		6410 B, 6420 B [18th, 19th, 20th]	6410 B-00, 6420 B-00	See footnote <sup>9</sup> , p. 27
113. Vinyl chloride	601	624, 1624B		6200 B [20th] and 6210 B [18th, 19th], >6200 C [20th] and 6230 B [18th, 19th]	6200 B and C-97	

<sup>1</sup>All parameters are expressed in micrograms per liter (µg/L) except for Method 1613B in which the parameters are expressed in picograms per liter (pg/L).

<sup>2</sup>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.

<sup>3</sup>“Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,” U.S. Environmental Protection Agency, September, 1978.

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

<sup>5</sup>Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.

<sup>5a</sup>625, screening only.

<sup>6</sup>“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).

<sup>7</sup>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 on-going 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.

<sup>8</sup>“Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk” 3M Corporation Revised 10/28/94.

<sup>9</sup>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.

<sup>10</sup>Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

**Table ID—List of Approved Test Procedures for Pesticides<sup>1</sup>**

Parameter	Method	EPA <sup>2,7</sup>	Standard Methods 18th, 19th, 20th Ed.	Standard Methods Online	ASTM	Other
1. Aldrin	GC	608	6630 B & C		D3086–90, D5812–96 (2002)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B–00		
2. Ametryn	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68
3. Aminocarb	TLC					See footnote <sup>3</sup> , p. 94; See footnote <sup>6</sup> , p. S16
4. Atraton	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68
5. Atrazine	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68; See footnote <sup>9</sup>
6. Azinphos methyl	GC					See footnote <sup>3</sup> , p. 25; See footnote <sup>6</sup> , p. S51
7. Barban	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
8. α-BHC	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B–00		
9. β-BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B–00		
10. δ-BHC	GC	608	6630 C		D3086–90, D5812–96(02)	See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B–00		
11. γ-BHC (Lindane)	GC	608	6630 B & C		D3086–90, D5812–96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B–00		

12. Captan	GC		6630 B		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7
13. Carbaryl	TLC					See footnote <sup>3</sup> , p. 94, See footnote <sup>6</sup> , p. S60
14. Carbo-phenothion	GC					See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73
15. Chlordane	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
16. Chloro-propham	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64.
17. 2,4-D	GC		6640 B			See footnote <sup>3</sup> , p. 115; See footnote <sup>4</sup> , p. 40
18. 4,4'-DDD	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
19. 4,4'-DDE	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
20. 4,4'-DDT	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
21. Demeton-O	GC					See footnote <sup>3</sup> , p. 25; See footnote <sup>6</sup> , p. S51
22. Demeton-S	GC					See footnote <sup>3</sup> , p. 25; See footnote <sup>6</sup> , p. S51
23. Diazinon	GC					See footnote <sup>3</sup> , p. 25; See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S51
24. Dicamba	GC					See footnote <sup>3</sup> , p. 115
25. Dichlofen-thion	GC					See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73
26. Dichloran	GC		6630 B & C			See footnote <sup>3</sup> , p. 7
27. Dicofol	GC				D3086-90, D5812-96(02)	
28. Dieldrin	GC	608	6630 B & C			See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
29. Dioxathion	GC					See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73
30. Disulfoton	GC					See footnote <sup>3</sup> , p. 25; See footnote <sup>6</sup> , p. S51
31. Diuron	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
32. Endosulfan I	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B-00		
33. Endosulfan II	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B-00		

34. Endosulfan Sulfate	GC	608	6630 C			See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
35. Endrin	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625 <sup>5</sup>	6410 B	6410 B-00		
36. Endrin aldehyde	GC GC/MS	608 625				See footnote <sup>8</sup>
37. Ethion	GC					See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73
38. Fenuron	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
39. Fenuron-TCA	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
40. Heptachlor	GC GC/MS	608 625	6630 B & C 6410 B	6410 B-00	D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
41. Heptachlor epoxide	GC GC/MS	608 625	6630 B & C 6410 B	6410 B-00	D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73; See footnote <sup>8</sup>
42. Isodrin	GC					See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S73
43. Linuron	GC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
44. Malathion	GC		6630 C			See footnote <sup>3</sup> , p. 25; See footnote <sup>4</sup> , p. 27; See footnote <sup>6</sup> , p. S51
45. Methiocarb	TLC					See footnote <sup>3</sup> , p. 94; See footnote <sup>6</sup> , p. S60
46. Methoxy-chlor	GC		6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
47. Mexacar-bate	TLC					See footnote <sup>3</sup> , p. 94; See footnote <sup>6</sup> , p. S60
48. Mirex	GC		6630 B & C			See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27
49. Monuron	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
50. Monuron-TCA	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
51. Nuburon	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
52. Parathion methyl	GC		6630 C			See footnote <sup>3</sup> , p. 25; See footnote <sup>4</sup> , p. 27
53. Parathion ethyl	GC		6630 C			See footnote <sup>3</sup> , p. 25; See footnote <sup>4</sup> , p. 27
54. PCNB	GC		6630 B & C			See footnote <sup>3</sup> , p. 7
55. Perthane	GC				D3086-90, D5812-96(02)	See footnote <sup>4</sup> , p. 27
56. Prometon	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68; See footnote <sup>9</sup>
57. Prometryn	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68; See footnote <sup>9</sup>
58. Propazine	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68; See footnote <sup>9</sup>
59. Propham	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
60. Propoxur	TLC					See footnote <sup>3</sup> , p. 94; See footnote <sup>6</sup> , p. S60

61. Sebumeton	TLC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68
62. Siduron	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
63. Simazine	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68; See footnote <sup>9</sup>
64. Strobane	GC		6630 B & C			See footnote <sup>3</sup> , p. 7
65. Swep	TLC					See footnote <sup>3</sup> , p. 104; See footnote <sup>6</sup> , p. S64
66. 2,4,5-T	GC		6640 B			See footnote <sup>3</sup> , p. 115; See footnote <sup>4</sup> , p. 40
67. 2,4,5-TP (Silvex)	GC		6640 B			See footnote <sup>3</sup> , p. 115; See footnote <sup>4</sup> , p. 40
68. Terbutylazine	GC					See footnote <sup>3</sup> , p. 83; See footnote <sup>6</sup> , p. S68
69. Toxaphene	GC	608	6630 B & C		D3086-90, D5812-96(02)	See footnote <sup>3</sup> , p. 7; See footnote <sup>4</sup> , p. 27; See footnote <sup>8</sup>
	GC/MS	625	6410 B	6410 B-00		
70. Trifluralin	GC		6630 B			See footnote <sup>3</sup> , p. 7; See footnote <sup>9</sup>

<sup>1</sup>Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

<sup>2</sup>The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

<sup>3</sup>"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.

<sup>4</sup>"Methods for Analysis of Organic Substances in Water and Fluvial Sediments," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).

<sup>5</sup>The method may be extended to include  $\alpha$ -BHC,  $\gamma$ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

<sup>6</sup>"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).

<sup>7</sup>Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an 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. 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.

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

<sup>9</sup>USGS Method 0-3106-93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94-37.

**Table IF—List of Approved Methods for Pharmaceutical Pollutants**

Pharmaceuticals pollutants	CAS registry No.	Analytical method number
----------------------------	------------------	--------------------------

acetonitrile	75-05-8	1666/1671/D3371/D3695.
n-amyl acetate	628-63-7	1666/D3695.
n-amyl alcohol	71-41-0	1666/D3695
benzene	71-43-2	D4763/D3695/502.2/524.2.
n-butyl-acetate	123-86-4	1666/D3695.
tert-butyl alcohol	75-65-0	1666.
chlorobenzene	108-90-7	502.2/524.2.
chloroform	67-66-3	502.2/524.2/551.
o-dichlorobenzene	95-50-1	1625C/502.2/524.2.
1,2-dichloroethane	107-06-2	D3695/502.2/524.2.
diethylamine	109-89-7	1666/1671.
dimethyl sulfoxide	67-68-5	1666/1671.
ethanol	64-17-5	1666/1671/D3695.
ethyl acetate	141-78-6	1666/D3695.
n-heptane	142-82-5	1666/D3695.
n-hexane	110-54-3	1666/D3695.
isobutyraldehyde	78-84-2	1666/1667.
isopropanol	67-63-0	1666/D3695.
isopropyl acetate	108-21-4	1666/D3695.
isopropyl ether	108-20-3	1666/D3695.
methanol	67-56-1	1666/1671/D3695.
Methyl Cellosolve Δ	109-86-4	1666/1671
methylene chloride	75-09-2	502.2/524.2
methyl formate	107-31-3	1666.
4-methyl-2-pentanone (MIBK)	108-10-1	1624C/1666/D3695/D4763/524.2.
phenol	108-95-2	D4763.
n-propanol	71-23-8	1666/1671/D3695.
2-propanone (acetone)	67-64-1	D3695/D4763/524.2.
tetrahydrofuran	109-99-9	1666/524.2.
toluene	108-88-3	D3695/D4763/502.2/524.2.
triethylamine	121-44-8	1666/1671.
xylenes	(Note 1)	1624C/1666.

Table 1F note:

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

**62-160, Table 2**  
**APPROVED WATER AND WASTEWATER PROCEDURES, CONTAINERS,**  
**PRESERVATION AND HOLDING TIMES**  
**FOR PARAMETERS NOT FOUND IN 40 CFR 136 \*\***

PARAMETER	METHOD	REFERENCE <sup>i</sup>	CONTAINER <sup>ii</sup>	PRESERVATION <sup>iii</sup>	MAXIMUM HOLDING TIME <sup>iv</sup>
Bromine	DPD Colorimetric <sup>v</sup>	SM 408E SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA-300.0 B <sup>vi</sup>	P, G	Cool, 4C	30 days
Chlorophylls	Spectrophotometric	SM 1002G SM 10200H	P, G <sup>vii</sup>	14d in dark	30 days <sup>g</sup>
Corrosivity	Calculated (CaCO <sub>3</sub> Stability, Langelier Index)	SM 203 SM 2330 ASTM D513-82	P, G	Cool, 4C <sup>viii</sup>	7 days <sup>h</sup>
Odor	Human Panel	SM 207 SM 2150	G only	Cool, 4C	6 hours
Salinity	Electrometric <sup>i</sup> Hydrometric Argentometric	SM 210 A SM 2420 B SM 210 B SM 2520 C SM 210C	G, wax seal	Analyze immediately or use wax seal	30 days <sup>ix</sup>
Taste	Human Panel	SM 211 A,B SM 2160 B SM 2160 C SM 2160 D ASTM 1292-86	G only	Cool, 4C	24 hours
Total Dissolved Gases	Direct-Sensing Membrane-Diffusion Method	SM 2810	---	---	Analyze in-situ
Transparency	Irradiometric <sup>x</sup>	17-3.021(6), FAC	---	---	Analyze in-situ
Un-ionized Ammonia	Calculated <sup>xi</sup>	DER-SOP <sup>xii</sup>	P, G	Cool, 4C Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	8 hours unpreserved 28 days preserved <sup>k</sup>
Organic Pesticides <sup>xiii</sup>	GC and HPLC	EPA (600-series) <sup>m</sup>	<sup>xiv</sup>	<sup>n</sup>	<sup>n</sup>

\*\*Reference: 17-160.700, F.A.C., Table 4

## 62-160, Table 2

<sup>i</sup> SM XXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 16th Edition, 1985 (except Chlorophylls, SM 1002G).  
SM XXXX = procedures from Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 17th Edition, 1989 (Except Chlorophylls, SM 10200H).

ASTM XXXX-YY= procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1988.

<sup>ii</sup> P= plastic, G= glass.

<sup>iii</sup> When specified, sample preservation should be performed immediately upon sample collection.

<sup>iv</sup> The times listed are the maximum times that samples may be held before analysis and still be considered valid.

<sup>v</sup> The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e. negligible interference).

<sup>vi</sup> "The Determination of Inorganic Anions in Water Ion Chromatography", EPA Method 300.0 B, Revised August 1991, by John D. Pfaff, Carol A. Brockoff and James W. O'Dell, U.S. EPA, Cincinnati, Ohio 45268.

<sup>vii</sup> Collect samples in opaque bottles and process under reduced light. Samples on filter taken from water having pH 7 or higher may be placed in airtight plastic bags and stored frozen for up to three weeks. Samples from acidic water must be processed promptly to prevent chlorophyll degradation.

<sup>viii</sup> Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.

<sup>ix</sup> The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

<sup>x</sup> Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e. the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DER Rule 17-3 FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.

<sup>xi</sup> The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H<sub>2</sub>SO<sub>4</sub> to pH between 1.5 and 2. Acid-preserved samples, stored at 4C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

<sup>xii</sup> DER Central Analytical Laboratory, Tallahassee, FL, Revision No. 1, October 3, 1983. The 1983 draft is available from the DER QA Section.

<sup>xiii</sup> Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) which are not included in Table ID of 40 CFR Part 136 (July 1989).

<sup>xiv</sup> Container, preservation and holding time as specified in each individual method shall be followed.