ENVIRONMENTAL PROTECTION AGENCY REGULATIONS ON TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

(40 CFR 136; 38 FR 28758, October 16, 1973)

PART 136—TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

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AUTHORITY: Sec. 304(g) of Federal Water Pollution Control Act Amendments of 1972 86 Stat. 816, et seq., Pub. L. 92–500).

§ 136.1 Applicability.

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

(a) An application submitted to the Administrator, or to a State having an approved NPDES program, for a permit under section 402 of the Federal Water Pollution Control Act as amended (FWPCA), and,

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

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

§ 136.2 Definitions.

As used in this part, the term:

(a) "Act" means the Federal Water Pollution Control Act, as amended, 33 U.S.C. 1314, et seq.

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

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

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

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

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 13th Edition, 1971. This publication is available from the American Public Health Association, 1015 18th St. NW., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 23, Water, Atmospheric Analysis, 1972*. This publication is available from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Wastes*, 1971, Environmental Protection Agency, Analytical Quality Control Laboratory, Cincinnati, Ohio. This publication is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (Stock Number 5501–0067).

§ 136.3 Identification of test procedures.

Every parameter or pollutant for which an effluent limitation is now specified pursuant to sections 401 and 402 of the Act is named together with test descriptions and references in Table I. The discharge parameter values for which reports are required must be determined by one of the standard analytical methods cited and described in Table I, or under certain circumstances by other methods that may be more advantageous to use when such other methods have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing that the Director of the State in which such discharge will occur does not object to the use of such alternate test procedures.

Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or Director upon the recommendation of the Director of the Methods Development and Quality Assurance Research Laboratory.

§ 136.4 Application for alternate test procedures.

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

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

(c) Unless and until printed applica tion forms are made available, an appli cation for an alternate test procedur may be made by letter in triplicate. An application for an alternate test proce dure under this subchapter shall:

(1) Provide the name and address o the responsible person or firm makin the discharge (if not the applicant) an the applicable ID number of the existin or pending permit, issuing agency, an type of permit for which the alternat test procedure is requested, and the dis charge serial number.

(2) Identify the pollutant or parame ter for which approval of an alternat testing procedure is being requested.

(3) Provide justification for usin testing procedures other than thos specified in Table I.

(4) Provide a detailed description o the proposed alternate test procedure together with references to publishe studies of the applicability of the alter nate test procedure to the effluents i question.

§ 136.5 Approval of alternate test pro cedures.

(a) The Regional Administrator o the region in which the discharge wil occur has final responsibility for ap proval of any alternate test procedure

(b) Within thirty days of receipt o an application, the Director will forwar such application, together with his rec ommendations, to the Regional Admin istrator. Where the Director recommend rejection of the application for scien tific and technical reasons which he pro vides, the Regional Administrator shal deny the application, and shall forwar a copy of the rejected application an his decision to the Director of the Stat Permit Program and to the Director o the Methods Development and Qualit Assurance Research Laboratory.

(c) Before approving any applicatio for an alternate test procedure, the Re gional Administrator shall forward a copy of the application to the Directo of the Methods Development and Qual ity Assurance Laboratory for review an recommendation.

(d) Within ninety days of receipt b the Regional Administrator of an appli cation for an alternate test procedure the Regional Administrator shall notif the applicant and the appropriate State agency of approval or rejection, or shal specify the additional information which is required to determine whether to ap prove the proposed test procedure. Prio

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TABLE I—LIST OF APPROVED TEST PROCEDURES

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
General analytical methods:				
1. Alkalinity as $CaCO_3$, mg $CaCO_3$/liter.	Titration: electrometric, manual or automated method—methyl orange.	p. 370	p. 143	p. 6. p. 8.
2. B.O.D. five day mg/liter.	Modified winkler or probe method	p. 489		
3. Chemical oxygen demand (C.O.D.) mg/liter.	Dichromate reflux	p. 495	p. 219	p. 17.
4. Total solids mg/liter	Gravimetric 103-105° C	p. 535		p. 280.
5. Total dissolved (filterable) solids mg/liter.	Glass fiber filtration 180° C			p. 275.
6. Total suspended (nonfilterable) solids mg/liter.	Glass fiber filtration 103-105° C	p. 537		p. 278.
7. Total volatile solids mg/liter.	Gravimetric 550° C	p. 536		p. 282.
8. Ammonia (as N) mg/liter.	Distillation—nesslerization or titration automated phenolate.			p. 134. p. 141.
9. Kjeldahl nitrogen (as N) mg/liter.	Digestion + distillation—nesslerization or titration utomated digestion phenolate.	p. 469		p. 149. p. 157.
10. Nitrate (as N) mg/liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.	p. 458 p. 461	p. 124	p. 170. p. 175. p. 185.
11. Total phosphorus (as P) mg/liter.	Persulfate digestion and single reagent (ascorbic acid), or manual digestion, and automated single reagent or stannous chloride.	p. 526 p. 532	p. 42	p. 235. p. 246. p. 259.
12. Acidity mg $CaCO_3$/liter	Electrometric end point or phenolphthalein end point.		p. 148	
13. Total organic carbon (TOC) mg/liter.	Combustion—infrared method [1]	p. 257	p. 702	p. 221.
14. Hardness—total mg $CaCO_3$/liter.	EDTA titration; automated colorimetric atomic absorption.	p. 179	p. 170	p. 76. p. 78.
15. Nitrite (as N) mg/liter.	Manual or automated colorimetric diazotization.			p. 185. p. 195.
Analytical methods for trace metals:				
16. Aluminum—total [2] mg/liter.	Atomic absorption	p. 210		p. 98.
17. Antimony—total [2] mg/liter.	Atomic absorption [4]			
18. Arsenic—total mg/liter.	Digestion plus silver diethyldithiocarbamate; atomic absorption.[3]	p. 65 p. 62		p. 13.
19. Barium—total [2] mg/liter.	Atomic absorption [4]	p. 210		
20. Beryllium—total [2] mg/liter.	Aluminon; atomic absorption	p. 67 p. 210		
21. Boron—total mg/liter	Curcumin	p. 69		
22. Cadmium—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 422	p. 692	p. 101.
23. Calcium—total [2] mg/liter.	EDTA titration; atomic absorption	p. 84	p. 692	p. 102.
24. Chromium VI mg/liter.	Extraction and atomic absorption; colorimetric.	p. 429		p. 94.
25. Chromium—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 426	p. 692 p. 403	p. 104.
26. Cobalt—total [2] mg/liter.	Atomic absorption [4]		p. 692	
27. Copper—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 430	p. 692 p. 410	p. 106.
28. Iron—total [2] mg/liter	do	p. 210 p. 433	p. 692 p. 152	p. 108.
29. Lead—total [2] mg/liter	do	p. 210 p. 436	p. 692	p. 110.
30. Magnesium—total [2] mg/liter.	Atomic absorption; Gravimetric	p. 210 p. 416 p. 201	p. 692	p. 112.
31. Manganese—total [2] mg/liter.	Atomic absorption	p. 210	p. 692	p. 114.
32. Mercury—total mg/liter.	Flameless atomic absorption [5]			
33. Molybdenum—total [2] mg/liter.	Atomic absorption [4]			
34. Nickel—total [2] mg/liter.	Atomic absorption; colorimetric [4]	p. 443	p. 692	
35. Potassium—total [2] mg/liter.	Atomic absorption; colorimetric; flame photometric.	p. 283 p. 285	p. 326	p. 115.
36. Selenium—total mg/liter.	Atomic absorption [3]			
37. Silver—total [2]	Atomic absorption [4]	p. 210		
38. Sodium—total [2] mg/liter.	Flame photometric; atomic absorption	p. 317	p. 326	p. 118.
39. Thallium—total [2] mg/liter.	Atomic absorption [4]			
40. Tin—total [2] mg/liter	do			
41. Titanium—total mg/liter.	do			
42. Vanadium—total [2] mg/liter.	Atomic Absorption; [4] Colorimetric	p. 157		
43. Zinc—total [2] mg/liter	Atomic Absorption; Colorimetric	p. 210 p. 444	p. 692	p. 120.
Analytical methods for nutrients, anions, and organics:				
44. Organic nitrogen (as N) mg/liter.	Kjeldahl nitrogen minus ammonia nitrogen.	p. 468		p. 149.
45. Ortho-phosphate (as P) mg/liter.	Direct single reagent; automated single reagent or stannous chloride.	p. 532	p. 42	p. 235. p. 246. p. 259.
46. Sulfate (as SO_4) mg/liter.	Gravimetric; turbidimetric; automated colorimetric—barium chloranilate.	p. 331 p. 334	p. 51 p. 52	p. 286. p. 288.
47. Sulfide (as S) mg/liter.	Titrimetric—iodine	p. 551		p. 294.
48. Sulfite (as SO_3) mg/liter.	Titrimetric; iodide-iodate	p. 537	p. 261	
49. Bromide mg/liter	do		p. 216	
50. Chloride mg/liter	Silver nitrate; mercuric nitrate; automated colorimetric-ferricyanide.	p. 96 p. 97	p. 23 p. 21	p. 29. p. 31.
51. Cyanide—total mg/liter.	Distillation—silver nitrate titration or pyridine pyrazolone colorimetric.	p. 307	p. 556	p. 41.
52. Fluoride mg/liter	Distillation—SPADNS	p. 171 p. 174	p. 191	p. 64.
53. Chlorine—total residual mg/liter.	Colorimetric; amperometric titration	p. 382	p. 223	
54. Oil and grease mg/liter.	Liquid-Liquid extraction with trichlorotrifluoroethane.	p. 254		

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TABLE I—LIST OF APPROVED TEST PROCEDURES

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
55. Phenols mg/liter	Colorimetric,[4] AAP	p. 502	p. 445	p. 232.
56. Surfactants mg/liter	Methylene blue colorimetric	p. 339	p. 619	p. 181.
57. Algicides mg/liter	Gas chromatography [6]			
58. Benzidine mg/liter	Diazotization—colorimetric [7]			
59. Chlorinated organic compounds (except pesticides) mg/liter.	Gas chromatography [6]			
60. Pesticides mg/liter	Gas chromatography [6]			
Analytical methods for physical and biological parameters:				
61. Color platinum-cobalt units or dominant wave-length, hue, luminance, purity.	Colorimetric; spectrophotometric	p. 160 p. 392		p. 38.
62. Specific conductance mho/cm at 25° C.	Wheatstone bridge	p. 323	p. 163	p. 284.
63. Turbidity jackson units.	Turbidimeter	p. 350	p. 467	p. 308.
See Note at end of Table I				
64. Fecal streptococci bacteria number/100 ml.	MPN; membrane filter; plate count	p. 689 p. 690 p. 691		
65. Coliform bacteria (fecal) number/100 ml.	MPN: Membrane filter	p. 669 p. 684		
66. Coliform bacteria (total) number/100 ml.	do	p. 664 p. 679		
Radiological parameters:				
67. Alpha—total pCi/liter	Proportional counter; scintillation counter	p. 598	p. 509	
68. Alpha—counting error pCi/liter.	do	p. 598	p. 512	
69. Beta—total pCi/liter	Proportional counter†	p. 598	p. 478	
70. Beta—counting error pCi/liter.	do	p. 598	p. 478	
71. Radium—total pCi/liter.	Proportional counter; scintillation counter	p. 611 p. 617	p. 674	

[1] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on Combustion-Methane Detection, is also acceptable.

[2] For the determination of total metals the sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. If much suspended material is present, as little as 50–100 ml of well-mixed sample will most probably be sufficient. (The sample volume required may also vary proportionally with the number of metals to be determined.)

Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated distilled HNO_3. Place the beaker on a hotplate and evaporate to dryness making certain that the sample does not boil. Cool the beaker and add another 3 ml portion of distilled concentrated HNO_3. Cover the beaker with a watch glass and return to the hotplate. Increase the temperature of the hotplate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary until the digestion is complete, generally indicated by a light colored residue. Add (1:1 with distilled water) distilled concentrated HCl in an amount sufficient to dissolve the residue upon warming. Wash down the beaker walls and the watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis. Concentrations so determined shall be reported as "total".

[3] See D. C. Manning, "Technical Notes", Atomic Absorption Newsletter, Vol. 10, No. 6 p. 123, 1971. Available from Perkin-Elmer Corporation, Main Avenue, Norwalk, Connecticut 06852.

[4] Atomic absorption method available from Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

[5] For updated method, see: Journal of the American Water Works Association 64, No. 1, pp. 20–26 (Jan. 1972) or ASTM Method D 3223–73, American Society for Testing and Materials Headquarters, 1916 Race St., Philadelphia, Pa. 19103.

[6] Interim procedures for algicides, chlorinated organic compounds, and pesticides can be obtained from the Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

[7] Benzidine may be estimated by the method of M.A. El-Dib, "Colorimetric Determination of Aniline Derivatives in Natural Waters", El-Dib, M.A., Journal of the Association of Official Analytical Chemists, Vol. 54, No. 6, Nov., 1971, pp. 1383–1387.

†As a prescreening measurement.

to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Methods Development and Quality Assurance Research Laboratory. A copy of all approval and rejection notifications will be forwarded to the Director, Methods Development and Quality Assurance Research Laboratory for the purposes of national coordination

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