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Part III

Environmental Protection Agency

40 CFR Part 122, 136, et al.

**Guidelines Establishing Test Procedures
for the Analysis of Pollutants Under the
Clean Water Act; National Primary
Drinking Water Regulations; and National
Secondary Drinking Water Regulations;
Analysis and Sampling Procedures; Final
Rule**

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 122, 136, 141, 143, 430, 455, and 465

[EPA-HQ-OW-2003-0070; FRL-8203-8]

RIN 2040-AD71

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This rule modifies the testing procedures approved for analysis and sampling under the Clean Water Act and Safe Drinking Water Act. EPA proposed these changes for public comment on August 18, 2003 and April 6, 2004. The Clean Water Act changes adopted in this final rule fall into the following categories: new vendor-developed methods as well as EPA and voluntary consensus standard bodies (VCSB) methods, updated versions of currently approved methods, revisions to method modification and analytical requirements, withdrawal of certain outdated methods, and changes to sample collection, preservation, and holding time requirements. This rule also changes regulations under the Safe Drinking Water Act that establish drinking water sampling and analysis procedures. The changes include approval of vendor-developed methods, new EPA and VCSB methods, updated VCSB methods, and approval of a modification to the test kit used with Syngenta Method AG-625 that restricts

its use in certain circumstances. The addition of new and updated methods to the wastewater and drinking water regulations provides increased flexibility to the regulated community and laboratories in the selection of analytical methods.

ADDRESSES: EPA has established a docket for this action under Docket ID No. EPA-OW-2003-0070. All documents in the docket are listed on the <http://www.regulations.gov> web site. Although listed in the index, some information is not publicly available, e.g., CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, is not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically through <http://www.regulations.gov> or in hard copy at the HQ Water Docket Center, EPA/DC, EPA West, Room 3334, 1301 Constitution Ave., NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number is (202) 566-2426 for the HQ Water Docket Center.

DATES: This regulation is effective April 11, 2007. The incorporation by reference of these methods is approved by the Director of the Federal Register on April 11, 2007. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. (Eastern time) on March 26, 2007 as provided at 40 CFR 23.2 and 23.7.

FOR FURTHER INFORMATION CONTACT: For information regarding the changes to wastewater regulations, contact Meghan Hessenauer, Engineering and Analysis Division (4303T), USEPA Office of

Science and Technology, 1200 Pennsylvania Ave., NW., Washington, DC 20460, 202-566-1040 (e-mail: hessenauer.meghan@epa.gov). For information regarding the changes to drinking water regulations, contact Patricia Snyder Fair, Technical Support Center (MS 140), USEPA, Office of Ground Water and Drinking Water, 26 West Martin Luther King Drive, Cincinnati, OH 45268, 513-569-7937 (e-mail: fair.pat@epa.gov).

SUPPLEMENTARY INFORMATION:

A. Potentially Regulated Entities

1. Clean Water Act

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits with conditions designed to ensure compliance with the technology-based and water quality-based requirements of the Clean Water Act (CWA). These permits may include restrictions on the quantity of pollutants that may be discharged as well as pollutant measurement and reporting requirements. If EPA has approved test procedures for analysis of a specific pollutant, the NPDES permittee must use an approved test procedure (or an approved alternate test procedure) for the specific pollutant when measuring the required waste constituent. Similarly, if EPA has established sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits will potentially be regulated by the actions in this rulemaking. Categories and entities that may potentially be subject to the requirements of today's rule include:

Category	Examples of potentially regulated entities
State, Territorial, and Indian Tribal Governments.	States, Territories, and Tribes authorized to administer the NPDES permitting program; States, Territories, and Tribes providing certification under Clean Water Act section 401.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	POTWs that must conduct monitoring to comply with NPDES permits.

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be regulated by this action. This table lists types of entities that EPA is now aware could potentially be regulated by this action. Other types of entities not listed in the table could also be regulated. To determine whether your facility is regulated by this action, you should carefully examine the applicability language at 40 CFR 122.1, (NPDES purpose and scope), 40 CFR 136.1

(NPDES permits and CWA), 40 CFR 403.1 (Pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

2. Safe Drinking Water Act

Public water systems are the regulated entities required to measure contaminants in drinking water

samples. In addition, EPA Regions, as well as States, and Tribal governments with authority to administer the regulatory program for public water systems under the Safe Drinking Water Act, may also measure contaminants in water samples. When EPA establishes a maximum contaminant level (MCL) for a given drinking water contaminant, the Agency also approves standardized test procedures for analysis of the contaminant. Public water systems required to test water samples must use

from sulfur, sulfide, sulfite, thiocyanate, and aldehydes. The recommended procedures may differ from those described in the older approved methods.

39. EPA is changing "Director of the Environmental Monitoring Systems Laboratory" and "Director, Analytical Methods Staff" to "Alternate Test Procedure Program Coordinator, Washington, DC" every place the phrases appear in the regulations. This reflects the current organizational structure and title for the head of EPA's Alternate Test Procedure (ATP) Program management. In addition, addresses for submission of ATPs are being updated to reflect the current location of the Alternate Test Procedure Program Coordinator.

40. The rule makes other minor editorial revisions to clarify existing regulations.

C. 40 CFR Parts 141 and 143

1. This rule amends the regulations at 40 CFR Part 141 and Part 143 to allow the use of 66 methods in "Standard Methods Online" (APHA 2003) (40 CFR 141.21, 141.23, 141.74, and 143.4).

2. This rule allows the use of 28 newer versions of methods published by ASTM International. The new versions are published in the 1999 "Annual Book of ASTM Standards," Vols. 11.01 and 11.02, in the 2000 "Annual Book of ASTM Standards," Vol. 11.02 and in individual standards published after 2000 (40 CFR 141.23).

3. This rule approves a new method submitted as an alternate test procedure for the determination of common anions—chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate, "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte" (D6508, Rev. 2) by Waters Corporation (40 CFR 141.23 and 143.4).

4. This rule approves two new methods for determination of available cyanide, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," Method OIA-1677, DW (January 2004), and ASTM D6888-04 (40 CFR 141.23).

5. This rule approves the use of EPA Method 300.1 (Revision 1.0, 1997) for compliance determinations of chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate (40 CFR 141.23 and § 143.4).

6. This rule approves the use of EPA Method 552.3 (Revision 1.0, 2003) for compliance determinations of dalapon (40 CFR 141.24).

7. This rule amends 40 CFR 141.25 to add a new method for determination of radium-226 and radium-228. This

method, "The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors" (Revision 1.2, December 2004), was developed by the Environmental Resources Center at the Georgia Institute of Technology, and was originally submitted to EPA as an alternate test procedure to the currently approved methods for determination of radium-226 and radium-228.

8. This rule allows States the option of approving ITS Free chlorine test strips as a test kit for the measurement of free chlorine using "Free Chlorine Species" (HOCl- and OCl-) by Test Strip," ITS Method D99-003 (Revision 3.0, November 21, 2003) by Industrial Test Systems, Inc. (40 CFR 141.74).

9. This rule approves EPA Method 327.0 (Revision 1.1, 2005) for measurement of chlorine dioxide residuals (40 CFR 141.74).

10. This rule approves the use of styrene divinyl benzene beads and stabilized formazin as alternatives to the presently approved formazin standard for determination of turbidity (40 CFR 141.74).

11. This rule revises footnote 17 to the table at 40 CFR 141.23 to allow the use of a 450-W UV lamp in the Kelada Method-01 for determination of cyanide.

12. This rule allows the use of Syngenta Method AG-625, with the modified immunoassay testing product manufactured by Beacon Analytical Systems, for the measurement of atrazine under certain conditions. It may only be used by those systems that do not use chlorine dioxide for drinking water treatment. In addition, the results of the analysis of samples with concentrations of atrazine more than one-half the atrazine MCL (*i.e.*, more than 1.5 µg/L) must be confirmed using another approved method (40 CFR 141.24).

13. This rule also revises footnote 8 to the table in paragraph (a)(1) to correct a long-standing discrepancy between the footnote and the specifications in Standard Method 9221 E. The table in question lists Standard Method 9221 E as one of two procedures that may be used for monitoring fecal coliforms. Footnote 8 simply notes the holding time for the A-1 broth used in this procedure. Today's action corrects the holding time specified in the footnote 8 to match the 7-day holding time that is specified in Standard Method 9221 E (40 CFR 141.74).

D. 40 CFR Part 430

This rule amends the Effluent Limitations Guidelines for the pulp, paper, and paperboard point source category at 40 CFR Part 430 to approve

a new method for determination of chlorinated phenolics in wastewaters generated by these industries. The rule adds a new section, 430.02(g), to allow the use of "Chlorinated Phenolics in Water by In situ Acetylation and GC/MS Determination" (Method CP-86.07) developed by the National Council for Air and Stream Improvement (NCASI) as an alternative to otherwise required Method 1653 in Part 430, Appendix A.

E. 40 CFR Part 455

This rule amends the regulations at 40 CFR Part 455 by moving Table 7 from 40 CFR Part 455 to 40 CFR Part 136.3(a) as new Table IG.

F. 40 CFR Part 465

This rule amends the Effluent Limitations Guidelines for the coil coating point source category at 40 CFR Part 465 to replace the method listed at section 465.03(c) for determination of oil and grease in wastewater samples from all subcategories of coil coating with EPA Method 1664A for determination of non-polar materials (NPM), which is generally equivalent to total petroleum hydrocarbons.

III. Changes Between the Proposed Rule and the Final Rule

Except as noted below, the content of the final rule is the same as that of the proposed rule. In some instances, EPA revised for clarity the language of the final rule from that in the proposed rule.

A. Silver Determinations

EPA received comments on the Agency's proposed withdrawal of EPA Method 272.1 which included information on how to keep silver in solution in samples with known or suspected high levels of silver by adding a solution containing cyanogen chloride. As a result, EPA has added a footnote to Table IB at § 136.3 to include procedures for preparation and addition of this reagent to digested samples of this type to keep the silver in solution for analysis by any of the approved methods. The Agency is withdrawing EPA Method 272.1, as proposed in April 2004 (69 FR 18183 April 6, 2004).

B. ASTM Method D5673-02 "Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry"

Based on comment received on the Agency's proposed approval of ASTM Method D5673-02, EPA is approving an updated version of this method ASTM D5673-03. EPA included the updated version of this method in a notice of data availability (70 FR 7909, February 16, 2005) and requested public

¹⁴“The Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors,” Revision 1.2, December 2004. Available from the Environmental Resources Center, Georgia Institute of Technology, 620 Cherry Street, Atlanta, GA 30332-0335, USA, Telephone: 404-894-3776. This method may be used to analyze for radium-226 and radium-228 in samples collected after January 1, 2005 to satisfy the radium-226 and radium-228 monitoring requirements specified at 40 CFR 141.26.

* * * * *

■ 16. Section 141.74 is amended as follows:

■ a. By revising the entry for turbidity in the table in paragraph (a)(1).

■ b. By revising footnotes 1, 8, 10, 11, and 12 to the table in paragraph (a)(1).

■ c. By adding footnote 13 to the table in paragraph (a)(1).

■ d. By revising paragraph (a)(2).

§ 141.74 Analytical and monitoring requirements.

(a) * * *

(1) * * *

Organism	Methodology	Citation ¹
Turbidity ¹³	Nephelometric Method	2130 B
	Nephelometric Method	180.1 ⁹
	Great Lakes Instruments	Method 2 ¹⁰
	Hach FilterTrak	10133 ¹²

¹ Except where noted, all methods refer to *Standard Methods for the Examination of Water and Wastewater*, 18th edition (1992), 19th edition (1995), or 20th edition (1998), American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005. The cited methods published in any of these three editions may be used. In addition, the following online versions may also be used: 2130 B-01, 9215 B-00, 9221 A, B, C, E-99, 9222 A, B, C, D-97, and 9223 B-97. Standard Methods Online are available at <http://www.standardmethods.org>. The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only Online versions that may be used.

⁸ A-1 broth may be held up to 7 days in a tightly closed screw cap tube at 4 °C.

⁹ “Methods for the Determination of Inorganic Substances in Environmental Samples,” EPA/600/R-93/100, August 1993. Available at NTIS, PB94-121811.

¹⁰ GLI Method 2, “Turbidity,” November 2, 1992, Great Lakes Instruments, Inc., 8855 North 55th Street, Milwaukee, WI 53223.

¹¹ A description of the SimPlate method, “IDEXX SimPlate TM HPC Test Method for Heterotrophs in Water,” November 2000, can be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092, telephone (800) 321-0207.

¹² A description of the Hach FilterTrak Method 10133, “Determination of Turbidity by Laser Nephelometry,” January 2000, Revision 2.0, can be obtained from; Hach Co., P.O. Box 389, Loveland, CO 80539-0389, telephone: 800-227-4224.

¹³ Styrene divinyl benzene beads (e.g. AMCO-AEPA-1 or equivalent) and stabilized formazin (e.g. Hach StablCal™ or equivalent) are acceptable substitutes for formazin.

(2) Public water systems must measure residual disinfectant concentrations with one of the analytical methods in the following table. If approved by the State, residual disinfectant concentrations for free chlorine and combined chlorine also may be measured by using DPD colorimetric test kits. In addition States may approve the use of the ITS free

chlorine test strip for the determination of free chlorine. Use of the test strips is described in Method D99-003, “Free Chlorine Species (HOCl- and OCl-) by Test Strip,” Revision 3.0, November 21, 2003, available from Industrial Test Systems, Inc., 1875 Langston St., Rock Hill, SC 29730. Free and total chlorine residuals may be measured continuously by adapting a specified

chlorine residual method for use with a continuous monitoring instrument provided the chemistry, accuracy, and precision remain the same. Instruments used for continuous monitoring must be calibrated with a grab sample measurement at least every five days, or with a protocol approved by the State.

Residual	Methodology	SM ¹	SM Online ²	Other
Free Chlorine	Amperometric Titration	4500-CI D	4500-CI D-00	D1253-03 ³
	DPD Ferrous Titrimetric	4500-CI F	4500-CI F-00.	
	DPD Colorimetric	4500-CI G	4500-CI G-00.	
	Syringaldazine (FACTS)	4500-CI H	4500-CI H-00.	
Total Chlorine	Amperometric Titration	4500-CI D	4500-CI D-00	D1253-03 ³
	Amperometric Titration (low level measurement).	4500-CI E	4500-CI E-00.	
	DPD Ferrous Titrimetric	4500-CI F	4500-CI F-00.	
	DPD Colorimetric	4500-CI G	4500-CI G-00.	
Chlorine Dioxide	Iodometric Electrode	4500-CI I	4500-CI I-00.	327.0, Revision 1.1 ⁴
	Amperometric Titration	4500-CIO ₂ C	4500-CIO ₂ C-00.	
	DPD Method	4500-CIO ₂ D.		
	Amperometric Titration	4500-CIO ₂ E	4500-CIO ₂ E-00.	
Ozone	Spectrophotometric			327.0, Revision 1.1 ⁴
	Indigo Method	4500-O ₃ B	4500-O ₃ B-97.	

¹ All the listed methods are contained in the 18th, 19th, and 20th editions of *Standard Methods for the Examination of Water and Wastewater*, 1992, 1995, and 1998; the cited methods published in any of these three editions may be used.

² Standard Methods Online are available at <http://www.standardmethods.org>. The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only Online versions that may be used.

³ *Annual Book of ASTM Standards*, Vol. 11.01, 2004 ; ASTM International; any year containing the cited version of the method may be used. Copies of this method may be obtained from ASTM International, 100 Barr Harbor Drive, P.O. Box C700 West Conshohocken, PA 19428-2959.

⁴ EPA Method 327.0, Revision 1.1, “Determination of Chlorine Dioxide and Chlorite Ion in Drinking Water Using Lissamine Green B and Horseradish Peroxidase with Detection by Visible Spectrophotometry,” USEPA, May 2005, EPA 815-R-05-008. Available online at <http://www.epa.gov/safewater/methods/sourcalt.html>.