Rules of
Department of Natural Resources
Division 60—Public Drinking Water Program
Chapter 5—Laboratory and Analytical Requirements

Title 10—DEPARTMENT OF NATURAL RESOURCES
Division 60—Public Drinking Water Program
Chapter 5—Laboratory and Analytical Requirements

10 CSR 60-5.010 Accepted and Alternate Procedures for Analyses

PURPOSE: This rule lists manuals containing acceptable analysis procedures for determination of contaminant levels.

Editor's Note: The secretary of state has determined that the publication of this rule in its entirety would be unduly cumbersome or expensive. The entire text of the material referenced has been filed with the secretary of state. This material may be found at the Office of the Secretary of State or at the headquarters of the agency and is available to any interested person at a cost established by state law.

(1) Inorganic and Radiological Contaminants. Unless substitute methods are approved by the department, analysis shall be conducted in accordance with the methods listed in the following table. Criteria for analyzing arsenic, barium, beryllium, cadmium, calcium, chromium, copper, lead, nickel, selenium, sodium, and thallium with digestion or directly without digestion, and other analytical test procedures are contained in "Technical Notes on Drinking Water Methods," EPA-600/R-94-173, October 1994. This document also contains approved analytical test methods which remain available for compliance monitoring until July 1, 1996. These methods will not be available for use after July 1, 1996. This document is available from the National Technical Information Service, NTIS PB95-104766, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800-553-6847.

Contaminant	Methodology (if appropriate)	Method (if appropriate)	Reference
Aluminum	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
		3120B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Platform Technique	200.9	22
		3113B	17
		3111D	17
Antimony	Atomic Absorption—Gaseous Hydride	D3697-92	19
	Atomic Absorption—Graphite Furnace	3113B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Platform Technique	200.9	22
Arsenic	Atomic Absorption—Graphite Furnace	D2972-93C	19
		3113B	17
	Atomic Absorption—Gaseous Hydride	D2972-93B	19
		3114B	17
	Atomic Absorption—Platform	200.9	22
	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
		3120B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Asbestos	Transmission Electron Microscopy	100.1	7
		100.2	27
Barium	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Graphite Furnace	3113B	17
	Atomic Absorption—Direct Aspiration	3111D	17
	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
		3120B	17
Beryllium	Atomic Absorption—Graphite Furnace	D3645-93B	19
		3113B	17
	Atomic Absorption—Platform	200.9	22
	Inductively Coupled Plasma—Emission Spectroscopy	3120B	17
		200.7	22
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Cadmium	Atomic Absorption—Graphite Furnace	3113B	17
	Atomic Absorption—Platform	200.9	22
	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Chloride		300.0	25
		D4327-91	19
		4500-Cl-D	17
Chromium	Atomic Absorption—Furnace Technique	3113B	17
	Atomic Absorption—Platform	200.9	22
	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
		3120B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Color		2120B	17
Copper	Atomic Absorption—Furnace Technique	D1688-90C	19
		3113B	17
	Atomic Absorption—Platform	200.9	22

Contaminant	Methodology (if appropriate)	Method (if appropriate)	Reference
Copper (cont.)	Atomic Absorption—Direct Aspiration	D1688-90A	19
		3111B	17
	Inductively Coupled Plasma	200.7	22
		3120B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Cyanide	Manual distillation followed by—	4500-CN-C	17
	1. Amenable Spectrophotometric	D2036-91B	19
		4500 CN-G	17
	2. Spectrophotometric—Manual	D2036-91A	19
		4500 CN-E	17
		1-3300-85	24
	3. Spectrophotometric— Manual—Semiautomated	335.4	25
	4. Selective Electrode	4500 CN-F	17
Fluoride	Colorimetric SPADNS, with distillation	4500 F-B&D	17
	Ion Chromatography	300.0	25
		D4327-91	19
		4110B	17
	Manual Electrode	D1179- 93B	19
		4500 F-C	17
	Automated Alizarin Fluoride Blue, with distillation (complexone)	4500 F-E	17
		129-71W	9
	Automated Ion Selective Electrode	380-75WE	10
Foaming Agents		5540C	17
Iron		200.7	22
		200.9	22
		3120B	17
		3111B	17
		3113B	17
Lead	Atomic Absorption—Furnace Technique	D3559-90D	19
		3113B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Platform Furnace	200.9	22
Manganese		200.7	22
		200.8	22
		200.9	22
		3120B	17
		3111B	17
		3113B	17
Mercury	Manual cold vapor technique	245.1	22
		D3223-91	19
		3112B	17
	Automated cold vapor technique	245.2	1
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Nickel	Atomic Absorption—Direct Aspiration	3111B	17
	Atomic Absorption—Platform Technique	200.9	22

Contaminant	Methodology (if appropriate)	Method (if appropriate)	Reference
Nickel (cont.)	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
		3120B	17
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Graphite Furnace	3113B	17
Nitrate	Manual Cadmium Reduction	D3867-90B	19
		4500 NO_3-E	17
	Automated Cadmium Reduction	353.2	25
		D3867-90A	19
		4500 NO_3-F	17
	Ion Selective Electrode	4500 NO_3-D	17
		601	26
	Ion Chromatography	300.0	25
		B1011	8
		4110B	17
		D4327-91	19
Nitrite	Spectrophotometric	4500-NO_3-B	17
	Automated Cadmium Reduction	353.2	25
		D3867-90A	19
		4500 NO_3-F	17
	Manual Cadmium Reduction	D3867-90B	19
		4500-NO_3-E	17
	Ion Chromatography	300.0	25
		B1011	8
		D4327-91	19
		4110B	17
Odor		2150B	17
Operational Monitoring—General			17
Operational Monitoring—pH	pH Value	150.1	1
		150.2	1
		D1293-84	19
		4500-H+-B	17
Operational Monitoring—Residual Disinfectant Monitoring—Chlorine Dioxide	Amperometric Titration	4500-ClO C	17
		4500-ClO E	17
	DPD Method	4500-ClO D	17
Operational Monitoring—Residual Disinfectant Monitoring—Free Chlorine	Amperometric Titration	4500-Cl D	17
	DPD Ferrous Titrimetric	4500-Cl F	17
	DPD Colorimetric	4500-Cl G	17
	Syringaldazine (FACTS)	4500-Cl H	17
Combined Chlorine	Amperometric Titration	4500-Cl D	17
	Amperometric Titration—low level measurement	4500-Cl E	17
	DPD Ferrous Titrimetric	4500-Cl F	17
	DPD Colorimetric	4500-Cl G	17
	Iodometric Electrode	4500-Cl I	17

Contaminant	Methodology (if appropriate)	Method (if appropriate)	Reference
Operational Monitoring—Lead and Copper Regulation	pH Electrometric	150.1	1
		150.2	1
		D1293-84	19
		4500-H+-B	17
	Conductivity	D1125-91A	19
		2510B	17
	Calcium (EDTA Titrimetric)	D511-93A	19
		3500-Ca-D	17
	(Inductively Coupled Plasma)	200.7	22
		3120B	17
	(Atomic Absorption—Direct Aspiration)	D511-93B	19
		3111B	17
	Alkalinity (Titrimetric)	D1067-92B	19
		2320B	17
	(Electrometric Titration)	I-1030-85	24
	Orthophosphate (unfiltered, no digestion or hydrolysis)		
	(Colorimetric, automated ascorbic acid)	365.1	25
		4500-P-F	17
	(Colorimetric, ascorbic acid single reagent)	D515-88A	19
		4500-P-E	17
	(Colorimetric, phosphomolybdate, automated-segmented flow, automated discrete)	I-1601-85	24
		I-2601-90	24
		I-2598-85	24
	(Ion Chromatography)	300.0	25
		D4327-91	19
		4110	17
	Silica (Colorimetric, molybdate blue, automated-segmented flow)	I-1700-85	24
		I-2700-85	24
	(Colorimetric)	D859-88	19
	(Molybdosilicate)	4500-Si-D	17
	(Heteropoly blue)	4500-Si-E	17
	(Automated method for molybdate-reatine silica)	4500-Si-F	17
	(Inductively Coupled Plasma)	200.7	22
		3120B	17
Operational Monitoring—Residual Disinfectant Monitoring—Ozone	Indigo	4500-O B	17
Operational Monitoring—Temperature	Thermometric	2550B	17
Radionuclides			11
			12
			13
	Examination of Water & Wastewater for Radioactivity	700	3

Contaminant	Methodology (if appropriate)	Method (if appropriate)	Reference
Radionuclides (cont.)	Gamma Spectrometry in Water	D2459	2
	Microquantities of Uranium in Water by Fluorometry	D2907	2
	Secondary Contaminants		3
Selenium	Atomic Absorption—Hydride Generation	D3859-93A	19
		3114B	17
	Atomic Absorption—Graphite Furnace	D385-93	19
		3113B	17
	Atomic Absorption—Platform	200.9	22
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
Silver	Atomic Absorption—Graphite Furnace	I-3720-85	24
	Inductively Coupled Plasma—Emission Spectroscopy	200.7	22
	Inductively Coupled Plasma—Mass Spectrometry	200.8	22
	Atomic Absorption—Platform Technique	200.9	22
		3120B	17
		3111B	17
		3113B	17
Sulfate	Colorimetric—Methylthymol blue	375.2	25
	Gravimetric	4500 SO_4-C, D	17
	Turbidimetric	4500 SO_4- F	17
	Ion Chromatography	300.0	25
		4110	17
		D4327-91	19
Thallium	Atomic Absorption—Platform Technique	200.9	22
	Inductively Coupled—Plasma-Mass Spectrometry	200.8	22
Total Dissolved Solids		2540C	17
Turbidity	Nephelometric	2130B	17
		180.1	25
Great Lakes Instruments	Method 2		18
Sodium	Inductively Coupled Plasma	200.7	22
	Atomic Absorption—Direct Aspiration	3111B	17
Zinc		200.8	22
		3111B	17

(A) References for analytical methods in 10 CSR 60-5.010(1):

1. "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268 (EPA-600/4-79-020), March 1983. Available from National Technical Information Service, PB84-128677. Methods 150.1, 150.2 and 246.2 are also available from U.S. EPA, EMSL, Cincinnati, OH 45268.

2. *Annual Book of ASTM Standards*, Vols. 11.01 and 11.02, 1991, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

3. *Standard Methods for the Examination of Water and Wastewater*, 16th edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1985.

4. "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," *Techniques of Water-Resources Investigations of the U.S. Geological Survey Books*, Book 5, Chapter A1, Third Edition, 1989. Available at Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

5. "Orion Guide to Water and Wastewater Analysis." Form WeWWG/5880, p. 5, 1985. Orion Research, Inc., Cambridge, MA 02139.

6. 200.7A "Inductively Coupled Plasma Atomic Emission Analysis of Drinking Water," Appendix to Method 200.7, March 1987, U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

7. "Analytical Method for Determination of Asbestos Fibers in Water," Method 100.1, EPA-600/4-83-043, September 1983. Available from National Technical Information Service, PB83-260471.

8. "Waters Test Method for the Determination of Nitrite/Nitrate in Water Using Single Column Ion Chromatography," Method B-1011, Millipore Corporation, Waters Chromatography Division, 34 Maple Street, Milford, MA 01757.

9. "Fluoride in Water and Wastewater," Industrial Method 129-71W, Technicon Industrial Systems, Tarrytown, NY 10591, December 1972.

10. "Fluoride in Water and Wastewater," Industrial Method No. 380-75WE, Technicon Industrial Systems, Tarrytown, NY 10591, February 1976.

11. "Radiochemical Methodology for Drinking Water," Environmental Monitoring Support Laboratory, EPA-600/4-75-008, U.S. EPA, Cincinnati, OH 45268.

12. "Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous Solutions," H.L. Krieger and S. Gold, EPA-R4-730014, U.S. EPA, Cincinnati, OH, May 1973.

13. *HASL Procedure Manual*, edited by John H. Harley, HASL 300, ERDA Health and Safety Laboratory, New York, NY, 1973.

14. "Determination of and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry," Method 200.8, version 4.3, August 1990, EPA, Environmental Monitoring and Systems Laboratory, Cincinnati, OH 45268. Available from ORD Publication, CERI, EPA, Cincinnati, OH 45268.

15. "Determination of Metals and Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry," Method 200.9, version 1.1, August 1990, EPA, Environmental Monitoring and Systems Laboratory, Cincinnati, OH 45268.

16. "Determination of Ozone in Water by the Indigo Method; A Submitted Standard Method," *Ozone Science and Engineering*, Volume 4, pages 169–176, Pergamon Press Ltd., 1982.

17. *Standard Methods for the Examination of Water and Wastewater*, 18th edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1992.

18. "Turbidity," GLI Method 2, November 2, 1992, Great Lakes Instruments, Inc., 8855 North 55 Street, Milwaukee, WI 53223.

19. *Annual Book of ASTM Standards*, Vols. 11.01 and 11.02, 1994, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

20. "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry" Method 200.8, August 1990, Revision 3.2 EPA EMSL. Available from U.S. EPA, EMSL Cincinnati, OH 45268.

21. "Determination of Inorganic Ions in Water by Ion Chromatography" Method 300.8, December 1989, U.S. EPA EMSL. Available from U.S. EPA, EMSL, Cincinnati, OH 45268.

22. "Methods for the Determination of Metals in Environmental Samples—Supplement I, EPA-600/R-94-111, May 1994." Available from National Technical Information Service (NTIS) NTIS PB 94-184942, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll free number is (800) 553-6847.

23. "Method 300. Determination of Inorganic Anions in Water by Ion Chromatography." Inorganic Chemistry Branch, Environmental Monitoring Systems Laboratory, August 1991.

24. Available from the Books and Open-File Reports Section, United States Geological Survey, Federal Center, Box 25425, Denver, CO 80225-0425.

25. "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA-600/R-93-100, August 1993. Available from National Technical Information Service, PB94-121811.

26. The procedure shall be done in accordance with the Technical Bulletin 601, "Standard Method of Test for Nitrate in Drinking Water," July 1994, PN 221890-001, Analytical Technology, Inc. Available from ATI, Orion, 529 Main Street, Boston, MA 02129.

27. "Determination of Asbestos Structure over 10-μm in Length in Drinking Water," Method 100.2, EPA-600/R-94-134, June 1994. Available from NTIS, PB94-201902.

(2) Organic Contaminants. Unless substitute methods are approved, the following table includes acceptable analysis procedures for organic contaminants:

Contaminant	Method
2,3,7,8-TCDD (Dioxin)	1613
2,4-D	515.1
	515.2
	555
2,4,5-TP (Silvex)	
515.1515.2,	555
3-Hydrodroxycarbofuran	531.1
	6610
Alachlor	505
	507
	525.2
	508.1
Aldicarb	531.1
	6610
Aldicarb sulfoxide	531.1
	6610
Aldicarb sulfone	531.1
	6610
Aldrin	505
	508
	525.2
	508.1
Atrazine	505
	507
	525.2
	508.1
Benzo(a)pyrene	525.2
	550
	550.1
Butachlor	507
	525.2
Carbaryl	531.1
	6610
Carbofuran	531.1
	6610
Chlordane	505
	508
	525.2
	508.1
Dalapon	515.1
	552.1
Di(2-ethylhexyl)adipate	506
	525.2
Di(2-ethylhexyl)phthalate	506
	525.2
Dibromochloropropane (DBCP)	504.1
	551
Dicamba	515.1
	515.2
	555
Dieldrin	505
	508
	508.1
	525.2
Dinoseb	515.1
	515.2
	555
Diquat	549.1
Endothall	548.1
Endrin	505
	508
	525.2
	508.1
Ethylene dibromide (EDB)	504.1
	551
Glyphosate	547
	6651
Heptachlor	505
	508
	525.2
	508.1
Heptachlor epoxide	505
	508
	525.2
	508.1
Hexachlorobenzene	505
	508
	525.2
	508.1
Hexachlorocyclopentadiene	505
	525.2
	508
	508.1
Lindane	505
	508
	525.2
	508.1
Methomyl	531.1
	6610
Methoxychlor	505
	525.2
	508.1
Metolachlor	507
	508.1
	525.2
Metribuzin	507
	508.1
	525.2
Oxamyl (vydate)	531.1
	6610
Pentachlorophenol	515.1
	515.2
	525.2
	555
Picloram	515.1
	515.2
	555
Polychlorinated biphenyls	505 (as Aroclors)
	508 (as Aroclors)
	508A (as decachlorobiphenyl)
Propachlor	508
	508.1
	525.2
Simazine	505
	507
	508.1
	525.2
Toxaphene	505
	508
	525.2
Total Trihalomethanes	502.2
	524.2
	551
Volatile Organic Chemicals (regulated and unregulated)	502.2
	524.2

Footnotes

1) A nitrogen-phosphorous detector should be substituted for the electron capture detector in Method 505 (or another approved method should be used) to determine alachlor, atrazine and simazine, if lower detection limits are required.

2) PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl. Each system which monitors for PCBs shall analyze each sample using either Method 505 or Method 508.

3) Analyses of total trihalomethanes shall be conducted in accordance with these methods and "Technical Notes on Drinking Water Methods," EPA-600/R94-173, October 1994, which is available at NTIS, PB95-104766.

4) In addition to Methods 502.2 and 524.2, analysis for bromodichloromethane, bromoform,* chlorodibromomethane, chloroform, carbon tetrachloride, tetrachlorethylene, 1,1,1-trichloroethane, and trichloroethylene may also be conducted by EPA Method 551. Analysis for 1,2,3-trichloropropane may be conducted by Methods 502.1, 524.2 and 504.1.

References for analytical methods in 10 CSR 60-5.010(2): Methods 502.2, 505, 507, 508, 508A, 515.1 and 531.1 are in "Methods for the Determination of Organic Compounds in Drinking Water," EPA-600/4-88-039, December 1988, revised July 1991. Methods 506, 547, 550, 550.1 and 551 are in "Methods for the Determination of Organic Compounds in Drinking Water—Supplement I," EPA-600-4-90-020, July 1990. Methods 515.2, 524.2, 548.1, 549.1, 552.1 and 555 are in "Methods for the Determination of Organic Compounds in Drinking Water—Supplement II," EPA-600/R-92-129, August 1992. Method 1613 is titled "Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope-Dilution HRGC/HRMS," EPA-821-B-94-005, October 1994. These documents are

available from National Technical Information Service (NTIS) NTIS PB91-231480, PB91-146027, PB92-207703, and PB95-104774, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll free number is (800) 553-6847. Method 6651 shall be followed in accordance with the 18th edition of "Standard Methods for the Examination of Water and Wastewater," 1992. Available from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005. Method 6610 shall be followed in accordance with the "Supplement to the 18th Edition of Standard Methods for the Examination of Water and Wastewater," 1994. Available from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005. EPA Methods 504.1, 508.1 and 525.2 are available from U.S. EPA EMSL, Cincinnati, OH 45268. The phone number is (513) 569-7586. Other analytical test procedures are contained in Technical Notes on Drinking Water Methods, EPA-600/R-94-173, October 1994, NTIS PB95-104766.

502.1 Volatile halogenated organic chemicals in water by purge and trap gas chromatography

502.2 Volatile organic compounds in water by purge and trap capillary column gas chromatography with photoionization and electrolytic conductivity detectors in series

503.1 Volatile aromatic and unsaturated organic compounds in water by purge and trap gas chromatography

504.1 1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-Trichloropropane (123TCP) in Water by Microextraction and Gas Chromatography

505 Analysis of organohalide pesticides and commercial polychlorinated biphenyl products (Aroclors) in water by microextraction and gas chromatography

506 Determination of phthalate and adipate esters in drinking water by liquid-liquid extraction or liquid-solid extraction and gas chromatography with photoionization detection

507 Determination of nitrogen- and phosphorus-containing pesticides in groundwater by gas chromatography with a nitrogen-phosphorus detector

508 Determination of chlorinated pesticides in water by gas chromatography with an electron capture detector

508.1 Determination of chlorinated pesticides, herbicides, and organohalides by liquid-solid extraction and electron capture gas chromatography

508A Screening for polychlorinated biphenyls by perchlorination and gas chromatography (for quantification if detected with Method 505 or 508)

515.1 Determination of chlorinated acids in water by gas chromatography with an electron capture detector, revision 5.0, May 1991

524.1 Measurement of purgeable organic compounds in water by purged column gas chromatography/mass spectrophotometry

524.2 Volatile organic chemicals in water by purge and trap capillary column gas chromatography/mass spectrophotometry

525.2 Determination of organic compounds in drinking water by liquid-solid extraction in capillary column gas chromatography/mass spectrometry

531.1 Measurement of N-methyl carbamoyloximes and N-methyl carbamates in water by direct aqueous injection HPLC with post-column derivatization

547 Analysis of glyphosate in drinking water by direct-aqueous-injection HPLC, with post-column derivatization

548 Determination of endothall in aqueous samples

549.1 Determination of diquat and paraquat in drinking water by liquid-solid extraction and high performance liquid chromatography with ultraviolet detection

550 Determination of polycyclic aromatic hydrocarbons in drinking water by liquid-liquid extraction and HPLC with coupled ultraviolet and fluorescence detection

550.1 Determination of polycyclic aromatic hydrocarbons in drinking water by liquid-solid extraction and HPLC with coupled ultraviolet and fluorescence detection

552 Determination of polycyclic aromatic hydrocarbons in drinking water by liquid-solid extraction and HPLC with coupled ultraviolet and fluorescence detection

551 Determination of chlorination disinfection byproducts and chlorinated solvents in drinking water by liquid-liquid extraction and gas chromatography with electron-capture detection

555 Determination of chlorinated acids in water by high performance liquid chromatography with a photodiode array ultraviolet detector

1613 "Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution." This method is available from U.S. EPA-OST, Sample Control Center, P.O. Box 1407, Alexandria, VA 22313.

6610 Carbamate pesticides

6651 Glyphosate herbicide

(3) Microbiological Contaminants. Unless substitute methods are approved, this section (3) lists acceptable analysis procedures for microbiological contaminants.

Contaminant	Approved Manual or Procedure
(A) *Escherichia coli*. the *(E. coli)*	Public water systems must conduct analysis of *Escherichia coli* in accordance with one (1) of following analytical methods: 1. EC medium supplemented with fifty (50) μ/ml of 4-methylumbelliferyl-beta-D-glucuronide (MUG) (final concentration). EC medium is described in *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition, Method 9221E, p. 9–52, paragraph 1a. MUG may be added to EC medium before autoclaving. EC medium supplemented with fifty (50) μ/ml of MUG is commercially available. At least ten (10) ml of EC medium supplemented with MUG must be used. The inner inverted fermentation tube may be omitted. The procedure for transferring a total coliform-positive culture to EC medium supplemented with MUG shall be as specified in 10 CSR 60-5.010(1)(B)2. for transferring a total coliform-positive culture to EC medium. Observe fluorescence with an ultraviolet light (366 nm) in the dark after incubating tube at 44.5 $\pm$ 0.2 degrees Celsius for 24 $\pm$ 2 hours; 2. Nutrient agar supplemented with 100 μ/ml MUG (final concentration). Nutrient agar is described in *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition, p. 9-47 to 9-48. This test is used to determine if a total coliform-positive sample, as determined by the membrane filter technique (MFT) or any other method in which a membrane filter is used, contains *E. coli*. Transfer the membrane filter containing a total coliform colony(ies) to nutrient agar supplemented with 100 μ/ml (final concentration) MUG. After incubating the agar plate at thirty-five degrees Celsius (35°C) for four (4) hours, observe the colony(ies) under ultraviolet light (366 nm) in the dark for fluorescence. If fluorescence is visible, *E. coli* are present; 3. Minimal Medium ONPG-MUG (MMO-MUG) Test, as set forth in the article "National Field Evaluation of a Defined Substrate Method for the Simultaneous Detection of Total Coliforms and *Escherichia coli* from Drinking Water: Comparison with Presence-Absence Techniques" (Edberg et al.), Applied and Environmental Microbiology, Volume 55, pp. 1003–1008, April 1989. (Note: The Autoanalysis Colilert System is an MMO-MUG test.) If the MMO-MUG test is total coliform-positive after a 24-hour incubation, test the medium for fluorescence with a 366-nm ultraviolet light (preferably with a 6-watt lamp) in the dark. If fluorescence is observed, the sample is *E. coli*-positive. If fluorescence is questionable (cannot be definitively read) after 24 hours incubation, incubate the culture for an additional four (4) hours (but not to exceed 28 hours total). And again test the medium for flouresecence. The MMO-MUG Test with hepes buffer in lieu of phosphate buffer is the only approved formulation for the detection of *E. coli*; 4. As an option to paragraph (3)(A)3. of this rule, a system with a total coliform-positive, MUG-negative, MMO-MUG test may further analyze the culture for the presence of *E. coli* by transferring a 0.1 ml 28-hour MMO-MUG culture to EC Medium + MUG. Observation of the results are described in 40 CFR 141.21(f)(6)(i); or 5. The Colisure test. A description of the Colisure test may be obtained from the Millipore Corporation, Technical Services Department, 80 Ashby Road, Bedford, MA 01730.
(B) Fecal Coliform.	1. Public water systems must conduct fecal coliform analysis in accordance with the following procedure: When the multiple-tube fermentation (MTF) technique or presence-absence (P-A) coliform test is used to test for total coliform, shake the lactose-positive presumptive tube or P-A vigorously and transfer the growth with a sterile three millimeter (3 mm) loop or sterile applicator stick into brilliant green lactose bile broth and EC medium to determine the presence of total and fecal coliforms, respectively. For EPA-approved analytical methods which use a membrane filter, transfer the total coliform-positive culture by one (1) of the following methods: Remove the membrane containing the total coliform colonies from the substrate with a sterile forceps and carefully curl and insert the membrane into a tube of EC medium (the laboratory may first remove a small portion of selected colonies for verification), swab the entire membrane filter surface with a sterile cotton swab and transfer the inoculum to EC medium (do not leave the cotton swab in the EC medium), or inoculate individual total coliform-positive colonies into EC medium. Gently shake the inoculated tubes of EC medium to insure adequate mixing and incubate in a waterbath at 44.5 $\pm$ 0.2 degrees Celsius for 24 $\pm$ 2 hours. Gas production of any amount in the inner fermentation tube of the EC medium indicates a positive fecal coliform test. The preparation of EC medium is described in *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition, Method 9221E, p. 9-2, paragraph 1a. Public water systems need only determine the presence or absence of fecal coliforms; a determination of fecal coliform density is not required. Only this method for fecal coliform is allowed for compliance with 10 CSR 60-4.020(5); or 2. *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition. A. Fecal coliform most probable number (MPN) procedures, method 9221E, pages 9-52 to 9-53 (Note: A-1 Broth may be held up to three (3) months in a tightly closed screwcap tube at four degrees Celsius (4°C);

Contaminant	Approved Manual or Procedure
	B. Fecal coliform membrane filter procedure, method 9222D, pages 9-60 to 9-61.
(C) Heterotrophic Bacteria.	*Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition, pour plate method, method 9215B. The time from sample collection to initiation of analysis may not exceed twenty-four (24) hours.
(D) Total Coliform.	1. The standard sample volume required for total coliform analysis, regardless of analytical method used, is one hundred milliliters (100 ml). The time from sample collection to initiation of analysis may not exceed forty-eight (48) hours. If the laboratory analyzes samples after thirty (30) hours and up to forty-eight (48) hours from sample collection, the laboratory shall indicate on the report of the analysis results that the data may be invalid because of excessive delay before sample processing. 2. *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association, 18th edition— A. Fermentation technique, method 9221A, B. (I) Lactose broth, as commercially available, may be used in lieu of lauryl tryptose broth, if the system conducts at least twenty-five (25) parallel tests between this medium and lauryl tryptose broth using the water normally tested, and this comparison demonstrates that the false-positive rate for total coliforms, using lactose broth, is less than ten percent (10%). (II) If inverted tubes are used to detect gas production, the media should cover these tubes at least one-half (1/2) to two-thirds (2/3) after the sample is added. (III) No requirement exists to run the completed phase on ten percent (10%) of all total coliform-positive confirmed tubes. B. Membrane filter (MF) technique, method 9222A, B, C. C. Presence-absence (P-A) coliform test, method 9221D. (I) Six-times formulation strength may be used if the medium is filter-sterilized rather than autoclaved. (II) No requirement exists to run the completed phase on ten percent (10%) of all total coliform-positive confirmed tubes. D. ONPG-MUG Test (also known as the Autoanalysis Colilert System, method 9223). E. Colisure test. The Colisure test must be incubated for twenty-eight (28) hours before examining the results. If an examination of the results at twenty-eight (28) hours is not convenient, then results may be examined at any time between twenty-eight (28) and forty-eight (48) hours. A description of the Colisure test may be obtained from the Millipore Corporation, Technical Service Department, 80 Ashby Road, Bedford, MA 01730.

(4) Sample collection for the contaminants listed in this rule must be conducted using the sample preservation, container and maximum holding time procedures specified in the following table. All other samples for contaminants in 10 CSR 60-5.010 shall be collected in accordance with procedures contained in the appropriate analytical method.

Contaminant	Preservative	Container	Holding Time
Antimony	Concentrated HNO_3 to pH < 2	P or G	6 months
Asbestos	Cool to 4°C	P or G	
Barium	Concentrated HNO_3 to pH < 2	P or G	6 months
Beryllium	Concentrated HNO_3 to pH < 2	P or G	6 months
Cadmium	Concentrated HNO_3 to pH < 2	P or G	6 months
Chromium	Concentrated HNO_3 to pH < 2	P or G	6 months
Copper			
Preserved	Concentrated HNO_3 to pH < 2	P or G	6 months
Unpreserved	NONE	P or G	14 days
Cyanide	Cool to 4°C, NaOH to pH > 12	P or G	
Fluoride	None	P or G	1 month
Lead			
Preserved	Concentrated HNO_3 to pH < 2	P or G	6 months
Unpreserved	NONE	P or G	14 days
Mercury	Concentrated HNO_3 to pH < 2	P or G	28 days

Contaminant	Preservative	Container	Holding Time
Nickel	Concentrated HNO_3 to pH < 2	P or G	6 months
Nitrate			
Chlorinated	Cool to 4°C	P or G	28 days
Nonchlorinated	Concentrated H_2SO_4 to pH < 2	P or G	14 days
Nitrite	Cool to 4°C	P or G	48 hours
Selenium	Concentrated HNO_3 to pH < 2	P or G	6 months
Thallium	Concentrated HNO_3 to pH < 2	P or G	6 months

(A) If HNO cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping to the laboratory. Upon receipt in the laboratory, the sample must be acidified with concentrated $HNO_3$to pH < 2 and held for sixteen (16) hours before analysis. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3; washings should be added to the sample.

(B) P = Plastic, hard or soft.

(C) G = Glass, hard or soft.

(D) In all cases samples should be analyzed as soon after collection as possible.

(E) For cyanide see method(s) for the information for preservation.

(F) The system shall take each sample at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

(G) If a system draws water from more than one (1) source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (that is, when water is representative of all sources being used).

(5) The department may reduce the total number of samples a system must analyze by allowing the use of compositing. Composite samples from a maximum of five (5) sampling points are allowed provided that the detection limit of the method used for analysis is less than one-fifth (1/5) of the MCL. For a lower number of composited samples the allowable relationship between the detection limit and MCL will change proportionally. Compositing of samples must be done in the laboratory and the composite sample must be analyzed within fourteen (14) days of collection. If the population served by the system is greater than thirty-three hundred (>3300) persons, then compositing is permitted only at sampling points within a single system. In systems serving less than or equal to thirty-three hundred (3300) persons, the department may permit compositing among different systems provided the five (5)-sample limit is maintained.

(A) Compositing of samples may be allowed for inorganic chemicals (IOCs) listed in 10 CSR 60-4.030(1) and synthetic organic chemicals (SOCs) listed in 10 CSR 60-4.040 and volatile organic chemicals (VOCs) listed in 10 CSR 60-4.010(2) and unregulated organic and inorganic chemicals listed in 10 CSR 60-4.110(2).

(B) If the concentration in the composite sample is greater than or equal to 0.0005 mg/l for any organic contaminant listed at 40 CFR 141.61(a), then a follow-up sample must be taken within fourteen (14) days at each sampling point included in the composite. These samples must be analyzed for the contaminants which exceeded 0.0005 mg/l in the composite sample. Resampling is not required for unregulated organic and inorganic chemicals listed in 10 CSR 60-4.110(2).

(C) If the concentration in the composite sample detects one (1) or more synthetic organic contaminants listed in paragraph (5)(D)2. of this rule, then a follow-up sample must be taken within fourteen (14) days at each sampling point included in the composite. The follow-up sample must be analyzed for the contaminant(s) detected.

(D) Detection limits.

1. Detection Limits for inorganic chemical analytical methods are the following:

Detection Limits for Inorganic Contaminants

Contaminant	Method	Detection Limit
		(mg/l)
Antimony	Atomic Absorption—Furnace Technique	0.003
	Atomic Absorption—Platform	0.0008
	Inductively Coupled Plasma—Mass Spectrometry	0.0004
	Atomic Absorption—Gaseous Hydride	0.001
Asbestos	Transmission Electron Microscopy	0.01 million fibers per liter
Barium	Atomic Absorption—Furnace Technique	0.0002
	Atomic Absorption—Direct Aspiration	0.1
	Inductively Coupled Plasma	0.002
	Inductively Coupled Plasma (EPA method 200.7)	0.001
	(mg/l)	
Beryllium	Atomic Absorption—Furnace Technique	0.0002
	Atomic Absorption—Platform	0.00002
	Inductively Coupled Plasma	0.0003
	Inductively Coupled Plasma—Mass Spectrometry	0.0003
Cadmium	Atomic Absorption—Furnace Technique	0.0001
	Inductively Coupled Plasma	0.001
Chromium	Atomic Absorption—Furnace Technique	0.001
	Inductively Coupled Plasma	0.007
	Inductively Coupled Plasma (EPA Method 200.7A)	0.001
Copper	All Methods Except Atomic Absorption	0.0002
	Atomic Absorption With Direct Aspiration	0.0002
Cyanide	Distillation, Spectrophotometric (screen)	0.02
	Distillation, Automated Spectrophotometric (screen)	0.005
	Distillation, Selective Electrode (screen)	0.02
	Distillation, Amenable, Spectrophotometric (free)	0.02
Lead	All Methods	0.001
Mercury	Manual Cold Vapor Technique	0.0002
	Automated Cold Vapor Technique	0.0002
Nickel	Atomic Absorption — Furnace Technique	0.001
	Atomic Absorption—Platform	0.0006
	Inductively Coupled Plasma	0.005
	Inductively Coupled Plasma—Mass Spectrometry	0.0005
Nitrate	Manual Cadmium Reduction	0.01
	Automated Hydrazine Reduction	0.01

Contaminant	Method	Detection Limit (mg/l)
	Automated Cadmium Reduction	0.05
	Ion Selective Electrode	1
	Ion Chromatography	0.01
Nitrite	Spectrophotometric	0.01
	Automated Cadmium Reduction	0.05
	Manual Cadmium Reduction	0.01
	Ion Chromatography	0.004
Selenium	Atomic Absorption—Furnace Technique	0.002
	Atomic Absorption—Gaseous Hydride	0.002
		(mg/l)
Thallium	Atomic Absorption—Furnace Technique	0.001
	Atomic Absorption—Platform	0.0007
	Inductively Coupled Plasma—Mass Spectrometry	0.0003

2. Detection limits for SOCs are the following:

Contaminant	Detection Limit (mg/l)
2,3,7,8-TCDD (Dioxin)	0.000000005
2,4-D	0.0001
2,4,5-TP (Silvex)	0.0002
Alachlor	0.0002
Aldicarb	0.0005
Aldicarb sulfoxide	0.0005
Aldicarb sulfone	0.0008
Atrazine	0.0001
Benzo(a)pyrene	0.00002
Carbofuran	0.0009
Chlordane	0.0002
Dalapon	0.001
Dibromochloropropane (DBCP)	0.00002
Di(2-ethylhexyl)adipate	0.0006
Di(2-ethylhexyl)phthalate	0.0006
Dinoseb	0.0002
Diquat	0.0004
Endothall	0.009
Endrin	0.00001
Ethylene dibromide (EDB)	0.00001
Glyphosate	0.006
Heptachlor	0.00004
Heptachlor epoxide	0.00002
Hexachlorobenzene	0.0001
Hexachlorocyclopentadiene	0.0001
Lindane	0.00002
Methoxychlor	0.0001
Oxamyl (Vydate)	0.002
Pentachlorophenol	0.00004
Picloram	0.0001
Polychlorinated biphenyls (PCBs) (as decachlorobiphenyl)	0.0001
Aroclor 1016	0.00008
Aroclor 1221	0.02
Aroclor 1232	0.0005
Aroclor 1242	0.0003
Aroclor 1248	0.0001
Aroclor 1254	0.0001
Aroclor 1260	0.0002
Simazine	0.00007
Toxaphene	0.001

3. The detection limit for VOCs is 0.0005 mg/l.

(E) If duplicates of the original sample from each sampling point used in the composite are available, the system may use these duplicates instead of resampling. The duplicate must be analyzed and the results reported to the department within fourteen (14) days of collection.

(F) Compositing Samples Prior to Gas Chromatograph (GC) Analysis.

1. Add five milliliter (5 ml) or equal larger amounts of each sample (up to five (5) samples are allowed) to a twenty-five milliliter (25 ml) glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

2. The samples must be cooled at four degrees Centigrade (4°C) during this step to minimize volatilization losses.

3. Mix well and draw out a five milliliter (5 ml) aliquot for analysis.

4. Follow sample introduction, purging and desorption steps described in the method.

5. If less than five (5) samples are used for compositing, a proportionately small syringe may be used.

(G) Compositing Samples Prior to GC/Mass Spectrophotometer (MS) Analysis.

1. Inject five milliliter (5 ml) or equal larger amounts of each aqueous sample (up to five (5) samples are allowed) into a twenty-five milliliter (25 ml) purging device using the sample introduction technique described in the method.

2. The total volume of the sample in the purging device must be twenty-five milliliters (25 ml).

3. Purge and desorb as described in the method.

(H) For lead and copper monitoring, composite samples from a maximum of five (5) sampling points per composite sample are allowed with prior approval of the department. The reportable value, as required in 10 CSR 60-7.020, for each of the samples in the composite is the concentration detected multiplied by the number of samples composited. If the concentration in the composite sample is less than the detection limit, the reportable value for each of the samples in the composite is the detection limit multiplied by the number of samples composited. If the ninetieth percentile concentration, calculated in accordance with 10 CSR 60-15.010(3)(C), exceeds the lead and copper action level established in 10 CSR 60-15.010(3)(A) and (B), each of the samples from which the composite was derived must be analyzed individually and reported as required in 10 CSR 60-7.020.

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**Original authority 1939, amended 1978, 1981, 1982, 1988, 1992, 1993, 1995.*

10 CSR 60-5.020 Laboratory Certification

PURPOSE: This rule establishes that required analyses must be done by laboratories certified by the department.

(1) For the purpose of determining compliance with this chapter, analytical results will be acceptable only if the samples have been analyzed by a laboratory certified by the department.

(2) To receive approval to conduct analyses for antimony, asbestos, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, nitrate, nitrite, selenium, and thallium the laboratory must—

(A) Analyze performance evaluation samples which include those substances provided by the Environmental Protection Agency (EPA) Environmental Monitoring and Support Laboratory or equivalent samples provided by the department; and

(B) Achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance Limit
Antimony	±30% at ≥0.006 mg/l
Asbestos	2 standard deviations based on study statistics

Contaminant	Acceptance Limit
Barium	±15% at ≥0.15 mg/l
Beryllium	±15% at ≥0.001 mg/l
Cadmium	±20% at ≥0.002 mg/l
Chromium	±15% at ≥0.01 mg/l
Copper	±10% at ≥0.50 mg/l
Fluoride	±10% at ≥ to 10 mg/l
Lead	±30% at ≥0.005 mg/l
Mercury	±30% at ≥0.0005 mg/l
Nickel	±15% at ≥0.01 mg/l
Nitrate	±10% at ≥0.4 mg/l
Nitrite	±15% at ≥0.4 mg/l
Selenium	±20% at ≥ 0.01 mg/l
Thallium	±30% at ≥ 0.002 mg/l

(3) To receive certification to conduct analyses for the contaminants in 10 CSR 60-4.100(2)(A)2–7. and (B)1.–13., the laboratory must—

(A) Analyze performance evaluation samples which include these substances provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the department;

(B) Achieve the quantitative acceptance limits in subsections (3)(C) and (D) of this rule for at least eighty percent (80%) of the regulated organic chemicals listed in 10 CSR 60-4.100(2)(A)2.–7. and (B)1.–13.;

(C) Achieve the quantitative results on the analyses performed under subsection (3)(A) of this rule that are within plus or minus twenty percent (±20%) of the actual amount of the substances in the performance evaluation sample when the actual amount is greater than or equal to 0.010 mg/l;

(D) Achieve quantitative results on the analyses performed under subsection (3)(A) of this rule that are within plus or minus forty percent (±40%) of the actual amount of the substances in the performance evaluation sample when the actual amount is less than 0.010 mg/l; and

(E) Achieve a method detection limit of 0.0005 mg/l.

(4) To receive certification for vinyl chloride, the laboratory must—

(A) Analyze performance evaluation samples provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the department;

(B) Achieve quantitative results on the analyses performed under subsection (4)(A) of this rule that are within plus or minus forty percent (±40%) of the actual amount of vinyl chloride in the performance evaluation sample;

(C) Achieve a method detection limit of 0.0005 mg/l; and

(D) Obtain certification for the contaminants listed in 10 CSR 60-4.100(2)(A)2.–7. and (B)1.–13.

(5) To receive certification to conduct analyses for the contaminants in 10 CSR 60-4.040(1), the laboratory must—

(A) Analyze performance evaluation samples which include those substances provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the department.

(B) Achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance Limit (percent)
2,3,7,8-TCDD (Dioxin)	2 standard
2,4-D	±50
2,4,5-TP	±50
Alachlor	±45
Aldicarb	2 standard deviations
Aldicarb sulfoxide	2 standard deviations
Aldicarb sulfone	2 standard deviations
Atrazine	±45
Benzo(a)pyrene	2 standard deviations
Carbofuran	±45
Chlordane	±45
Dalapon	2 standard deviations
Dibromochloropropane	±40
Di(2-ethylhexyl)adipate	2 standard deviations
Di(2-ethylhexyl)phthalate	2 standard deviations
Dinoseb	2 standard deviations
Diquat	2 standard deviations
Endothall	2 standard deviations
Endrin	±45
Ethylene dibromide	±40
Glyphosate	2 standard deviations
Heptachlor	±45
Heptachlor epoxide	±45
Hexachlorobenzene	2 standard deviations
Hexachlorocyclopentadiene	2 standard deviations
Lindane	±45
Methoxychlor	±45
Oxamyl	2 standard deviations
Polychlorinated biphenyls (PCBs) (as decachlorobiphenyl)	0—200
Picloram	2 standard deviations
Simazine	2 standard deviations
Toxaphene	±45
Pentachlorophenol	±50

(6) To receive approval to conduct analyses for copper and lead, the laboratory must—

(A) Analyze performance evaluation samples which include those substances provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the department;

(B) Achieve quantitative acceptance limits for copper plus or minus ten percent (±10%) of the actual amount in the performance evaluation sample when the actual amount is greater than or equal to 0.050 mg/l; lead plus or minus thirty percent (±30%) of the actual amount in the performance evaluation sample when the actual amount is greater than or equal to 0.005 mg/l; and

(C) Achieve a method detection limit of 0.001 mg/l.

(7) The department has the authority to allow the use of previously collected monitoring data for purposes of monitoring, if the data were collected and analyzed in accordance with the requirements of this rule.

(8) All lead levels measured between the Practical Quantification Level (PQL) and Method Detection Limit (MDL) must be either reported as measured or they can be reported as one-half (1/2) the PQL (0.0025 mg/l). All levels below the lead MDL must be reported as zero (0).

(9) All copper levels measured between the PQL and the MDL must be either reported as measured or they can be reported as one-half (1/2) the PQL (0.015 mg/l). All levels below the copper MDL must be reported as zero (0).

(10) Operational monitoring measurements required by 10 CSR 60-4.080(3) shall be performed on-site by persons acceptable to the department.

(11) The department will consider acceptance of analytical results from out-of-state laboratories upon written request.

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