

Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

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NR 219.01 Purpose. The purpose of this chapter is to establish analytical test methods, requirements for laboratories, and procedures applicable to effluent limitations for discharges from point sources as authorized by ss. 147.04 (5) and 144.95, Stats.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86.

NR 219.02 Applicability. (1) The procedures prescribed herein shall, except as provided in s. NR 219.05, be used in the determination of concentrations and quantities of pollutant parameters as required for:

(a) An application submitted to the department for a permit under ch. 147, Stats.

(b) Reports required to be submitted by dischargers in accordance with the conditions of issued permits.

(2) Section NR 219.07 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149, HSS 157, or HSS 165.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86.

NR 219.03 Definitions. As used in this chapter:

(1) **Standard Methods** - means "Standard Methods for the Examination of Water and Waste Water," 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street NW, Washington, D.C. 20036.

(2) **ASTM** - means "Annual Book of Standards, Part 31, Water, 1975." This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(3) **EPA methods** - means "Methods for Chemical Analysis of Water and Waste, 1974", Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

(4) **Regional Administrator** - the term "Regional Administrator" means the Regional Administrator of Region V, U.S. Environmental Protection Agency.

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(4m) Copies of the publications identified above, and of the publications referred to in footnotes 1 through 3, 5 through 10, 12, 13, 15 through 17, and 22 through 24 of s. NR 219.06 are available for inspection at the offices of the department of natural resources, the secretary of state and the revisor of statutes.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. (1), (2), (3) and (4m), Register, January, 1978, No. 265, eff. 2-1-78.

NR 219.04 Application for alternate test procedures. (1) Any person may apply to the regional administrator for approval of an alternate test procedure for a specific discharge. Such application shall be made in the following manner:

(a) The applicant shall submit an application to the regional administrator through the department.

(b) The application for an alternate test procedure shall be made by letter in triplicate, and

1. Provide the name and address of the responsible person or firm making the discharge (if not the applicant), the number of the existing or pending permit, the name of the issuing agency, and the discharge serial number,

2. Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested,

3. Provide justification for using testing procedures other than those specified in ch. NR 219, and

4. Provide a detailed description of the proposed alternate test procedure, together with references to published studies on the applicability of the alternate test procedure to the effluents in question.

(2) Any person may apply to the director, environmental monitoring and support laboratory, Cincinnati, Ohio 45268 for approval of an alternate test procedure for nationwide use. Such application shall be made in the following manner:

(a) The application for an alternate test procedure shall be made by letter, in triplicate, and

1. Provide the name and address of the responsible person or firm making the request,

2. Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested,

3. Provide a detailed description of the proposed alternate test procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in wastewater from representative or specified industrial or other categories, and

4. Provide comparability data for the performance of the proposed alternate test procedure compared to the approved test procedures.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; r. and rec. January, 1978, No. 265, eff. 2-1-78.

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NR 219.05 Approval of alternate test procedures. (1) The regional administrator has final responsibility for approval of any alternate test procedure proposed by responsible person or firm making the discharge.

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1. The first part of the document discusses the importance of maintaining accurate records of all transactions. It emphasizes that this is crucial for ensuring the integrity of the financial statements and for providing a clear audit trail.

2. The second part of the document outlines the specific procedures that should be followed when recording transactions. This includes details on how to handle receipts, invoices, and other supporting documents, as well as the timing and frequency of record-keeping.

3. The third part of the document addresses the role of the accounting department in the overall financial management of the organization. It highlights the need for close communication and collaboration between the accounting department and other departments to ensure that all financial data is captured and recorded accurately.

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and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO₃). Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 minutes. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker and 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 volume based on the expected metal concentration. The sample is now ready for analysis.

²³As the various furnace devices (flameless A.A.) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

²⁴See "Atomic Absorption Newsletter," vol. 13,75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

²⁵Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as 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 a 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 2M Na₂S₂O₃ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended procedure is satisfactory.

²⁶The method found on page 75 measures only the dissolved portion while the method on page 78 measures only suspended. Therefore the two results must be added together to obtain "total."

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; r. and recr. Register, January, 1978, No. 265, eff. 2-1-78.

NR 219.07 Laboratory certification or registration. Bacteriological analyses of groundwater samples, and all radiological analyses shall be performed by the state laboratory of hygiene or at a laboratory certified or approved by the department of health and social services. Other laboratory test results submitted to the department under this chapter shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

- (1) Temperature,
- (2) Turbidity,
- (3) Bacteria tests in wastewater effluent,
- (4) pH,
- (5) Chlorine residual,
- (6) Specific conductance,
- (7) Physical properties of soils and sludges,
- (8) Nutrient tests of soils and sludges,
- (9) Flow measurements.

Note: The requirement in this section to submit data from a certified or registered laboratory is effective on August 28, 1986.

History: Cr. Register, April, 1986, No. 364, eff. 8-28-86.