Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

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

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76.

NR 219.02 Applicability. 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:

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

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

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76.

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.

(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 inspec-

tion 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 recr. January, 1978, No. 265, eff. 2-1-78.

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.

and cautiously add a 5 ml portion of aqua regia. (Agua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3). 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.

[23]As 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.

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

[25]Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/1 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/1, 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/1 the recommended procedure is satisfactory.

[26]The 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.