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Standard Test Method for Total Chemically Bound Nitrogen in Water by Pyrolysis and Chemiluminescence Detection[1]

This standard is issued under the fixed designation D5176; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the total nitrogen content of water in concentrations from 0.5 to 1000 mg/L. Higher nitrogen concentrations may be determined by making the proper dilutions.

1.2 This test method does not determine molecular nitrogen (N_2).

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

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3. Terminology

3.1 ~~*Definitions—*~~*Definitions:* ~~For definitions of terms used in this test method, refer to Terminology D1129.~~

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total chemically bound* ~~*nitrogen—*~~*nitrogen, n—*all inorganic and organic nitrogen in the sample, except molecular nitrogen (N_2).

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4. Summary of Test Method

4.1 The sample of water is introduced into a stream of oxygen or inert/oxygen mix flowing through a quartz pyrolysis tube. Oxidative pyrolysis converts chemically bound nitrogen to nitric oxide (NO). The gas stream is dried and the NO is contacted with ozone (O_3) producing metastable nitrogen dioxide (NO_2*). As the NO_2* decays, light is emitted and detected by a photomultiplier tube. The resulting signal is a measure of the total chemically bound nitrogen in the sample.

5. Significance and Use

5.1 This test method is useful for the determination of total chemically bound nitrogen in wastewaters and other waters.

6. Apparatus[3]

6.1 *Pyrolysis Furnace*—An electric tube furnace capable of achieving a temperature of 1100°C. The furnace may be single or multizoned and may have common or separate and independent temperature controls.

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[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The apparatus described in 6.1 – 6.7 is manufactured by Antek Instruments, Inc., Houston, TX and Dohrmann Division of Rosemount Analytical Inc., Santa Clara, CA, and was used in the validation study of this test method.

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6.2 *Pyrolysis Tube*—The pyrolysis tube must be fabricated from quartz and should be designed to ensure complete pyrolysis of a wide variety of samples.

6.3 *Chemiluminescence ~~Detector —~~Detector*—The detector shall have a photomultiplier tube capable of sensing the light emission of the decaying NO_2*. The detector shall have digital display, onboard ozone generator and analog output for data system or strip chart recorder.

6.4 *Recorder* (optional)—The recorder shall be able to accept a 1 V full scale signal and to provide a chart speed of 1 cm/min.

6.5 *Microlitre Syringe*—Any standard series of microlitre syringes with stainless steel needles is acceptable. See manufacturer's instructions for appropriate syringe sizes.

6.6 *Syringe Drive Mechanism*—The syringe drive shall be capable of driving the sample from a microlitre syringe at a controlled, reproducible rate.

6.7 *Sample Boat*—Samples with high concentrations of suspended matter or dissolved nonvolatile compounds may tend to plug the syringe needle upon injection into the pyrolysis tube. In this case a sample boat of quartz or platinum, with or without quartz wool, should be used, in conjunction with the appropriate pyrolysis tube. The pyrolysis tube shall allow the introduction of the sample into the boat by microlitre syringe without interrupting the gas flow system.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. [4] Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets the purity specifications of Type I or Type II water, presented in Specification D1193.

7.3 *Inert Gas,* Argon (minimum purity 99.99 %).

7.4 *Oxygen* (minimum purity 99.6 %).

7.5 *Stock Solution, Pyridine* (10 000 mg N/L)—Prepare by accurately weighing 5.647 g of pyridine into a 100 mL volumetric flask and dilute to 100 mL with water.

7.6 *Pyridine Solutions, Standard* (1000, 500, 100, 50, 10, 5, 1, and 0.5 mg N/L)—Dilute ten volumes of the stock solution (see 6.5) with 90 volumes of water to prepare a 1000 mg N/L standard. Similarly, by serial dilution with water, prepare 500, 100, 50, 10, 5, 1, and 0.5 mg N/L standards.

8. Preparation of Apparatus

8.1 Assemble apparatus according to manufacturer's instructions.

9. Calibration and Standardization

9.1 Use the water that was used to prepare the standards as a zero blank standard.

9.2 A sample size of 5 to 10 µL is sufficient to cover the concentration range of this test method. The volume of the sample shall be accurately determined.

9.3 *Syringe Injection*—Fill the syringe to the 5 µL mark and retract the plunger so that the liquid meniscus is at the 1 µL mark. Note the position of the plunger. Insert the syringe needle through the inlet septum up to the syringe barrel and allow the furnace to burn all nitrogen bearing residue off the syringe needle. Reset the detector and inject the sample at a controlled rate of 1 to 2 µL/s. A syringe drive mechanism (see 6.6) is strongly recommended. When all sample has been injected, withdraw the syringe needle. Retract the plunger so that the sample meniscus is again at the 1 µL point and note the plunger position. The true amount injected is the difference between the two plunger positions.

NOTE 1—If water samples contain high concentrations of suspended matter or dissolved nonvolatile compounds, the syringe needle may tend to plug or the precision and bias of the test method may be degraded. In such a case, the sample boat system should be used (see 6.7).

9.4 *Boat Injection*—Fill the microlitre syringe to the mark and inject the sample directly into the boat while holding the needle in contact with the side of the boat or with the quartz wool.

9.5 Determine each calibration standard and the zero blank three times and record the net response from the average of each set of standard responses.

[4] *Reagent Chemicals, American Chemical Society ~~Specifications~~ Specifications, ~~,~~*American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.