Designation: D 5176 – 91 (Reapproved 2003)

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

This standard is issued under the fixed designation D 5176; 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 the standard. The values given in parentheses are for information only.

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:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

3.2 *Definition of Term Specific to This Standard:*

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

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.

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*—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

[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] *Annual Book of ASTM Standards*, Vol 11.01.

[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.