Designation: D1426 – 08

StandardTest Methods for Ammonia Nitrogen In Water[1]

This standard is issued under the fixed designation D1426; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of ammonia nitrogen, exclusive of organic nitrogen, in water. Two test methods are included as follows:

	Sections
Test Method A—Direct Nesslerization	7 to 15
Test Method B—Ion Selective Electrode	16 to 24

1.2 Test Method A is used for the routine determination of ammonia in steam condensates and demineralizer effluents.

1.3 Test Method B is applicable to the determination of ammonia nitrogen in the range from 0.5 to 1000 mg NH_3N/L directly in reagent and effluent waters. Higher concentrations can be determined following dilution. The reported lower range is based on multiple-operator precision. Lower limits have been obtained by two of the twelve laboratories participating in the round robin.

1.4 Both test methods A and B are applicable to surface and industrial waters and wastewaters following distillation. The test method for distillation given in Appendix X1 has been used in the past to meet requirements for predistillation of samples being analyzed for ammonia.

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

1.6 *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.*

1.7 The distillation method now appears as Appendix X1 and is provided as nonmandatory information only. The automated colorimetric phenate method has been discontinued.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1066 Practice for Sampling Steam
D1129 Terminology Relating to Water
D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)[3]
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

2.2 *APHA Standard*[4]:

Standard Methods for the Examination of Water and Waste Water

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D1129.

4. Significance and Use

4.1 Nitrogen is a nutrient in the environment and is necessary to sustain growth of most organisms. It exists in several forms such as nitrate, nitrite, organic nitrogen such as proteins or amino acids, and ammonia.

4.2 Ammonia is a colorless, gaseous compound with a sharp distinctive odor. It is highly soluble in water where it exists in a molecular form associated with water and in an ionized form as NH_4^+. The extent of association or ionization is dependent

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[1] These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents 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 last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American Public Health Association, 800 I St. N.W., Washington, DC 20001, http://www.apha.org.

***A Summary of Changes section appears at the end of this standard**

on the temperature and pH. It may also be toxic to aquatic life. The extent of toxicity is dependent upon species and extent of dissociation.[5] Ammonia may occur in water as a product of anaerobic decomposition of nitrogen containing compounds or from waste streams containing ammonia.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used in all tests. 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, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I. In addition, this water shall be free of ammonia nitrogen. Such water is best prepared by the passage of distilled water through an ion-exchange resin. These resins should also be selected so that organic compounds which might subsequently interfere with the ammonia determination will be removed. Regeneration of the ion-exchange materials should be carried out in accordance with the instructions of the manufacturer.

6. Sampling

6.1 Collect the sample in accordance with Practice D1066, Specification D1192, and Practices D3370, as applicable.

6.2 Preserve the samples by the addition of 1 mL of concentrated sulfuric acid per litre and store at 4°C. The pH should be 2.0 or less. Analyze the samples within 24 h of sampling. Do not use mercuric chloride as a preservative.

TEST METHOD A—DIRECT NESSLERIZATION

7. Scope

7.1 This test method is suitable for the rapid routine determination of ammonia nitrogen in steam condensates and demineralized water. See Appendix X1 for the distillation test method.

8. Summary of Test Method

8.1 A sample aliquot is Nesslerized directly and the ammonia content determined colorimetrically.

9. Interferences

9.1 Glycine, urea, glutamic acid, cyanates, and acetamide hydrolyze very slowly in solution on standing, but, of these, only urea and cyanates will hydrolyze on distillation at a pH of 9.5. Glycine, hydrazine, and some amines will react with Nessler's reagent to give the characteristic yellow color in the time required for the test. Similarly, volatile alkaline compounds such as hydrazine and the amines will influence titrimetric results. Some organic compounds such as ketones, aldehydes, alcohols, and some amines may cause an off color on Nesslerization. Some of these, such as formaldehyde may be eliminated by boiling off at a low pH prior to Nesslerization. Residual chlorine must be removed prior to the ammonia determination by pretreatment of the sample.

9.2 Turbid samples may be clarified with $ZnSO_4$ and NaOH solution; the precipitated $Zn(OH)_2$ is filtered off, discarding the first 25 mL of filtrate, and the ammonia is determined on an aliquot of the remaining clear filtrate by direct Nesslerization. Ammonia can be lost in basic conditions. Check procedure with a standard solution.

10. Apparatus

10.1 *Nessler Tubes*— Matched Nessler tubes[4] about 300 mm long, 17-mm inside diameter, and marked for 50 mL at 225 ± 1.5 mm from inside the bottom.

10.2 *Photometer*—Filter photometer or spectrophotometer suitable for absorbance measurements at 425 nm. Filter photometers and photometric practices used in this test method shall conform to Practice E60. Spectrophotometers shall conform to Practice E275.

10.3 *Stoppers*—Rubber, size No. 2, to fit Nessler tubes. These stoppers shall be boiled in H_2SO_4 (1 + 99), rinsed, boiled in NaOH solution (1 g/L), rinsed, allowed to stand in dilute Nessler reagent for 30 min, and then rinsed again.

11. Reagents

11.1 *Ammonia Nitrogen Solution, Standard* (1 mL = 0.01 mg N)—Dry reagent grade ammonium sulfate $((NH_4)_2SO_4)$ for 1 h at 100°C. Accurately weigh 4.718 g and dissolve in water. Dilute to 1 L in a volumetric flask. Pipet 10 mL of this stock solution to a 1-L volumetric flask and dilute to volume with water.

11.2 *Disodium Dihydrogen Ethylenediamine Tetraacetate Solution* (500 g/L)—Dissolve 500 g of disodium dihydrogen ethylenediamine tetraacetate dihydrate in water containing 100 g of NaOH. Gently heat to complete dissolution. Cool and dilute to 1 L.

11.3 *Nessler Reagent*—Dissolve 100 g of anhydrous mercuric iodide (HgI_2) and 70 g of anhydrous potassium iodide (KI) in a small volume of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of sodium hydroxide (NaOH) in 500 mL of water. Dilute the mixture to 1 L. Store the solution in the dark for five days and filter twice, either through a fritted glass crucible or glass fiber filter before using. If this reagent is stored in a chemically resistant bottle out of direct sunlight, it will remain stable up to a period of 1 year.

NOTE 1—This reagent should give the characteristic color with ammonia within 10 min after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50-mL volume). The solution may be used without 5-day storage if it is filtered through a 0.45 µm membrane (previously rinsed with reagent water Type I (see Specification D1193)) shortly before use.

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[5] *Quality Criteria for Water*, USEPA-440/9-76-023, July 26, 1976, pp. 16–24.

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