



Designation: D1426 – 15 (Reapproved 2021)^{ε1}

Standard Test Methods for Ammonia Nitrogen In Water¹

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 U.S. Department of Defense.

^{ε1} NOTE—The WTO caveat was editorially added in November 2021.

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:

Test Method A—Direct Nesslerization	Sections
Test Method B—Ion Selective Electrode	7 – 16
	17 – 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 NH₃N/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, health, and environmental 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.

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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1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- D1066 Practice for Sampling Steam
 - 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
 - D3370 Practices for Sampling Water from Flowing Process Streams
 - 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:³
- Standard Methods for the Examination of Water and Waste Water

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, 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

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 Available from American Public Health Association, 800 I St. NW, Washington, DC 20001, <http://www.apha.org>.

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 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.⁴ 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.⁵ 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 Practices **D1066** and **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.

NOTE 1—This preservation procedure will convert cyanate to ammonia. The user must be cautioned not to acidify samples if they contain cyanates. The preservation can extend the holding time to 28 days; however, the user will need to confirm the actual holding time.

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 tubes³ 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 and Materials

11.1 *Ammonia Nitrogen Solution, Standard* (1 mL = 0.01 mg N)—Dry reagent grade ammonium sulfate ($(\text{NH}_4)_2\text{SO}_4$) 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. A purchased ammonia nitrogen stock solution of appropriate known purity is also acceptable.

NOTE 2—Ammonia standards should be stored in an area free from ambient ammonia vapors.

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 *Filter Paper*—Purchase suitable filter paper. Typically the filter papers have a pore size of 0.45- μm membrane.

⁴ *Quality Criteria for Water*, USEPA-440/9-76-023, July 26, 1976, pp. 16–24.
⁵ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Anal. 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.