

Designation: D2973 - 22

Standard Test Method for Total Nitrogen in Peat Materials¹

This standard is issued under the fixed designation D2973; 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 a chemical test method for the determination of the mass percent of nitrogen in peat material.
- 1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.
- 1.3.1 The procedures used to specify how data are collected/recorded or calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.
- 1.4 Warning—Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.
- 1.5 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.
- 1.6 This international standard was developed in accordance with internationally recognized principles on standard-

¹ This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.22 on Media for Plant Growth.

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ization 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

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D2974 Test Methods for Determining the Water (Moisture) Content, Ash Content, and Organic Material of Peat and Other Organic Soils

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits and Data Records in Geotechnical Data

3. Terminology

- 3.1 Definitions: 3ed22f68214b/astm-d2973-2
- 3.1.1 For definitions of common technical terms used in this standard, refer to Terminology D653.

4. Summary of Test Method

- 4.1 In general terms, the nitrogen is converted into ammonium salts by destructive digestion of the specimen with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts are subsequently decomposed in a hot alkaline solution from which the ammonia is recovered by distillation and finally determined by acidimetric titration.
- 4.2 Specifically, a representative specimen is obtained from a representative sample and is air dried following Method B of D2974. A portion of the air dried specimen is put in a flask along with mercuric oxide, potassium sulfate, and sulfuric acid. The flask is gradually heated to a boil. After boiling and

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.

cooling, water, potassium sulfide solution, zinc, and sodium hydroxide solution are added. The flask is connected to the digestion bulk/condenser and the flask is heated. Finally, sulfuric acid mixed with an indicator is titrated into the flask until the end point is reached.

5. Significance and Use

- 5.1 Nitrogen content is important as it is one of the primary plant food elements necessary for plant growth. Nitrogen content of peat is necessary to make sure an adequate, but not excessive amount of fertility is supplied to the target plant. Nitrogen is present in peat as organic nitrogen, and therefore, does not release nitrogen to plants as quickly as chemical fertilizers. However, nitrogen from peat continues to be released for several years as the organic matter decomposes.
- 5.2 This information is particularly useful to people working in industries where plant health, sustainability, and viability are important.

Note 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

- 6.1 For Digestion—Use Kjeldahl flasks of hard, moderately thick, well-annealed glass with total capacity of about 800 mL. Conduct digestion over a heating device adjusted to bring 250 mL of Type III water at 25°C to a rolling boil in about 5 min.
- 6.2 For Distillation—Use 800-mL Kjeldahl flasks fitted with rubber stoppers through which passes the lower end of an efficient scrubber bulk or trap to prevent mechanical carry-over of sodium hydroxide solution during distillation. Connect the upper end of the bulb tube to the condenser tube by rubber tubing. Trap the outlet of the condenser in such a way as to make sure complete absorption of ammonia distilled over into the acid in the receiver.
- 6.3 Erlenmeyer Flask—A glass flask having a capacity of 250 or 300 mL.
- 6.4 *Oven*—A thermostatically controlled oven capable of maintaining a uniform temperature of 110 ± 5 °C throughout the drying chamber.
- 6.5 Balance—Balances shall conform to the requirements of Guide D4753 and having a readability without estimation to 0.01 g for water content determinations and 0.001 g for mass determinations. The capacity of the balance shall exceed the mass of the container plus specimen.
- 6.6 Mortar and Pestle (Optional)—Apparatus suitable for crushing soft lumps.
- 6.7 Glass Cylinder—A cylinder with an indication mark at $1,000 \pm 5$ mL.
- 6.8 *Miscellaneous Items*—Items such as spatulas, dishes, stirring rods, glassware of appropriate sizes with graduations, gloves, eye protection, protective clothing, and wash bottle may be useful.

7. Reagents

- 7.1 Purity of Reagents—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.³ 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.
- 7.2 *Type III Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D1193.
- 7.3 Boric Acid Solution (H_3BO_3), 4 %, (40 g/L)—Dissolve 40 g of boric acid in Type III water and dilute to 1 L.
 - 7.4 Mercuric Oxide Tablets (HgO).
- 7.5 Methyl Red—Dissolve 200 mg of methyl red in 100 mL of alcohol.
- 7.6 Methyl Blue—Dissolve 200 mg of methyl blue in 100 mL of alcohol.
- 7.7 *Mixed Indicator*—Mix in a ratio of 1 part methyl red to 2 parts methylene blue.
 - 7.8 Potassium Sulfate (K₂SO₄).
- 7.9 Potassium Sulfide Solution (K_2S), (40 g/L)—Dissolve 40 g of potassium sulfide in Type III water and dilute to 1 L.
- 7.10 Sodium Hydroxide Solution (NaOH), (450 g/L)—Dissolve 450 g of nitrate-free sodium hydroxide in Type III water and dilute to 1 L. The specific gravity of the solution should be 1.36 or greater.
- 7.11 Sulfuric Acid (H_2SO_4), (sp gr 1.84)—Concentrated sulfuric acid.
- 7.12 Sulfuric Acid, Standard (0.1 to 0.3 N)—Dissolve 3.0 to 9.0 mL of sulfuric acid (H₂SO₄) in Type III water and dilute to 1 L. When making this standard, the normality must be known without estimation as this value is used in the calculation of the percent nitrogen.
 - 7.13 *Zinc*—30 mesh.

8. Hazards

8.1 Chemicals used in this test can be harmful. It is recommended to wear gloves, eye protection, and protective clothing when working with these chemicals. Adequate ventilation should also be considered. Review of the Safety Data Sheets (SDS) that accompany the chemicals is recommended prior to their use.

9. Sampling and Test Specimens

9.1 Following Section 7 of Test Methods D2974, first obtain the representative sample and then the representative specimen.

³ 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.



- 9.2 Air-dry the test specimen following Method B, 8.2.1 through 8.2.4 of Test Methods D2974. Measure and record the water removed during air-drying as a percentage of the as-received mass to the nearest 0.1 %.
- 9.3 Calculate the amount of air-dried equivalent specimen mass as follows to the nearest 0.1 g:

air-dried equivalent specimen mass,
$$g = 10.0 - (10.0 \times moisture removed (%))/100$$
 (1)

9.4 Thoroughly mix the air-dried specimen and obtain the equivalent of 10.0 g of test specimen on the as-received basis. Measure and record this mass to the nearest 0.001 g.

10. Procedure

- 10.1 Place the air-dried equivalent specimen as obtained in 9.4 in the digestion flask.
- 10.2 Add 0.7 g of HgO, 15 g of powdered K_2SO_4 , and 35 mL of H_2SO_4 .
- 10.3 Place the flask in an inclined position and heat gradually. Then boil briskly until the solution clears. Continue boiling for an additional 30 min.
- 10.4 Cool, then add about 300 mL of Type III water. Cool below 25°C, and then add 25 mL of K_2S solution and mix to precipitate the mercury.
- 10.5 Add a pinch of zinc to prevent bumping, tilt the flask, and add a layer of NaOH solution sufficient to make the contents strongly alkaline. Do not agitate the mixture until the flask is connected to the digestion bulb or condenser.
- 10.6 Immediately connect the flask to the digestion bulb or condenser. Have the tip of the condenser immersed in the boric acid (H₃BO₃) solution (the amount of boric acid need not be measured) in the receiver and then rotate the flask to mix the contents thoroughly.
- 10.7 Heat until the ammonia has distilled (150 mL or more of distillate).
- 10.8 Titrate with standard sulfuric acid using mixed indicator to violet end point. Record the amount of standard sulfuric acid used during titration to the nearest 1 mL.

11. Calculation

11.1 Calculate the percentage nitrogen to the nearest 1 % as follows:

Nitrogen (as – received), $\% = (A \times B) \times 0.14$ (2)

where:

 $A = \text{millilitres of } 0.1 \text{ to } 0.3 \text{ N H}_2\text{SO}_4 \text{ used for titration and } B = \text{normality of the H}_2\text{SO}_4.$

12. Report: Test Data Sheet(s)/Form(s)

- 12.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.3 and in Practice D6026.
- 12.2 Record as a minimum the following general information (data):
- 12.2.1 Sample/Specimen identification information, such as, description and manufacturer of the peat.
 - 12.2.2 Any special selection and preparation processes.
- 12.2.3 Test number, if any, testing dates and the initials of the person(s) who performed the test.
- 12.3 Record as a minimum the following test specimen data:
 - 12.3.1 Record the water removed during air-drying.
- 12.3.2 Record the mass of the air-dried equivalent specimen
- 12.3.3 Record the amount of standard sulfuric acid used during titration.
- 12.3.4 Record the percent nitrogen of the as-received specimen to the nearest 0.1 %.

13. Precision and Bias

- 13.1 Precision—Test data precision is not presented due to the nature of the materials tested. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Also, it is not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.
- 13.1.1 Subcommittee D18.22 is seeking any data from the users of this test method that might be used to make a limited statement on precision.
- 13.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

14. Keywords

14.1 chemical analysis; nitrogen; nitrogen compounds; peat; peat products; percent nitrogen