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 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

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D2974 Test Methods for Moisture, Ash, and Organic Matter 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 in Geotechnical Data

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

- 3.1 Definitions:
- 3.1.1 For definitions of common technical terms in this standard, refer to Terminology D653.

4. Summary of Test Method

4.1 In this method, 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.

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.

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.

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 Soil as a Medium for Plant Growth.

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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.

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*—Thermostatically controlled oven capable of maintaining a uniform temperature of $110 \pm 5^{\circ}\text{C}$ throughout the drying chamber. Specification E145 provides information regarding the specification of ovens.
- 6.5 Balance—Balances shall conform to the requirements of Specification D4753 and having a readability without estimation to 0.01 g for moisture 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 Cylinder—A glass cylinder with an indication mark at $1,000 \pm 5$ mL used for mixing reagents.
- 6.8 *Miscellaneous Items*—Items such as spatulas, dishes, stirring rods, 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 (H_2SO_4) in Type III water and dilute to 1 L.
 - 7.13 *Zinc*—30 mesh.

8. Hazards

8.1 Warning—Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury containing products. See the applicable product Safety Data Sheet (SDS) for additional. Users should be aware that selling mercury or mercury containing products into your state or country may be prohibited by law.

9. Sampling and Test Specimens

- 9.1 Obtain a sample as outlined in Section 7 of Test Methods D2974.
- 9.2 Air-dry the sample in accordance with Method B, 8.1.3.2 of Test Methods D2974 (air dried portion only). Determine and record the moisture removed during air-drying as a percentage of the as-received mass to the nearest 0.1 %.
- 9.3 Thoroughly mix the air-dried specimen and obtain the equivalent of 10.0 g of test specimen on the as-received basis. Determine and record this mass to the nearest 0.001 g. Calculate the grams 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)

10. Procedure

- 10.1 Place the air-dried equivalent specimen as obtained in 9.3 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

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.

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 (H_3BO_3) 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 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 1.4.
- 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 Technician name or initials and date of testing.
- 12.3 Record as a minimum the following test specimen data:
 - 12.3.1 Record the moisture 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 compounds; peat

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition $(2010^{\epsilon 1})$ that may impact the use of this standard. (July 1, 2016)

- (1) Added reference to D6026 in Section 1 and 11.
- (2) Added clarification to Section 5.
- (3) Added Note 1.
- (4) Updated the Apparatus section to include items needed to perform the test.
- (5) Added the Hazards section about mercury.

- (6) Change section from "Preparation of Sample" to "Sampling and Test Specimens" and included more details on obtaining the test specimen.
- (7) Broke the procedure into line items instead of one long paragraph.
- (8) Updated the Report and Precision and Bias sections.

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