Designation: D 2973 – 71 (Reapproved 1998)

Standard Test Method for Total Nitrogen in Peat Materials[1]

This standard is issued under the fixed designation D 2973; 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 is a chemical test method[2] for the determination of the weight percent of nitrogen in peat material.

1.2 *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 2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils[3]

3. Summary of Test Method

3.1 In this procedure the nitrogen is converted into ammonium salts by destructive digestion of the sample 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. Significance and Use

4.1 Nitrogen content is important as it is one of the primary plant food elements necessary for plant growth. 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. Apparatus

5.1 *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 water at 25°C to a rolling boil in about 5 min.

5.2 *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 ensure complete absorption of ammonia distilled over into the acid in the receiver.

5.3 *Erlenmeyer Flask*, 250 or 300-mL capacity.

6. Reagents

6.1 *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.[4] 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.

6.2 *Boric Acid Solution, 4 %* (40 g/L)—Dissolve 40 g of boric acid (H_3BO_3) in water and dilute to 1 L.

6.3 *Mercuric Oxide Tablets* (HgO).

6.4 *Mixed Indicator*—Dissolve 200 mg of methyl red in 100 mL of alcohol. Dissolve 200 mg methylene blue in 100 mL of alcohol. Mix in a ratio of 1 part methyl red to 2 parts methylene blue.

6.5 *Potassium Sulfate* (K_2SO_4).

6.6 *Potassium Sulfide Solution* (40 g/L)—Dissolve 40 g of potassium sulfide in water and dilute to 1 L.

6.7 *Sodium Hydroxide Solution* (450 g/L)—Dissolve 450 g of nitrate-free sodium hydroxide (NaOH) in water and dilute to 1 L. The specific gravity of the solution should be 1.36 or greater.

6.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

6.9 *Sulfuric Acid, Standard* (0.1 to 0.3 *N)*—Dissolve 3.0 to 9.0 mL of sulfuric acid (H_2SO_4) in water and dilute to 1 L.

6.10 *Zinc*, 30 mesh.

7. Preparation of Sample

7.1 Air-dry the sample in accordance with Method II of Test

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.06 on Physical-Chemical Interactions of Soil and Rock..

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[2] This method is currently undergoing an extensive review by ASTM Committee D-18 and additional alternative methods will be proposed.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."

Methods D 2974. Record the percentage of moisture removed by air-drying.

8. Procedure

8.1 Mix thoroughly the air-dried, ground sample and weigh to the nearest 1 mg the equivalent of 10.0 g of test specimen on the as-received basis. Determine the grams of air-dried sample equivalent to 10.0 g of as-received sample as follows:

equivalent sample weight, g = 10.0 − (10.0 × percent moisture removed)/100

Place the weighed sample in the digestion flask, add 0.7 g of HgO, 15 g of powdered K_2SO_4, and 35 mL of H_2SO_4. Place the flask in an inclined position and heat gradually. Then boil briskly until the solution clears. Continue boiling for an additional 30 min. Cool, add about 300 mL of water, cool below 25°C, add 25 mL of K_2S solution and mix to precipitate the mercury. 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. Immediately connect the flask to the digestion bulb or condenser. Have the tip of the condenser immersed in the H_3BO_3 solution (this need not be measured) in the receiver and then rotate the flask to mix the contents thoroughly. Heat until the ammonia has distilled (at least 150 mL of distillate). Titrate with standard acid using mixed indicator to violet end point.

9. Calculation

9.1 Calculate the percentage nitrogen as follows:

$$\text{Nitrogen (as–received), \%} = (A \times B) \times 0.14 \quad (1)$$

where:

A = millilitres of 0.1 N to 0.3 N H_2SO_4 required for titration of the solution, and

B = normality of the H_2SO_4.

10. Report

10.1 Report to the nearest 0.1 % the nitrogen content of the as-received sample.

11. Precision and Bias

11.1 *Precision*—Due to the nature of the soil or rock materials tested by this method it is either not feasible or too costly at this time to produce multiple specimens which 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. Subcommittee D18.18 welcomes proposals that would allow for development of a valid precision statement.

11.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

12. Keywords

12.1 chemical analysis; nitrogen compounds; peat