Designation: D982 – 05(Reapproved 2009)

Technical Association of Pulp
and Paper Industry
Standard Method T 418 os-61

Standard Test Method for Organic Nitrogen in Paper and Paperboard[1]

This standard is issued under the fixed designation D982; 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 the determination of nitrogenous organic materials in paper and paperboard, which typically are used to reduce the thermal degradation of the cellulose in the paper and paperboard.

1.2 The nitrogen determination by this method does not include the nitrogen in nitro compounds, nitrates, nitrites, azo, hydrazine, cyanide, or pyridine ring-type compounds, none of which are normally found in paper and paperboard. There is no known modification of the method that is applicable to all nitrogenous compounds.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product (Withdrawn 2010)[3]

D644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying (Withdrawn 2010)[3]

3. Summary of Test Method

3.1 This test method is a modification of the well-known Kjeldahl procedure. In this test method, compounds that yield their nitrogen as ammonia are digested with concentrated sulfuric acid, using sodium sulfate to raise the boiling point, and mercuric oxide as a catalyst. The organic matter is destroyed and the nitrogen is fixed as ammonium sulfate in the excess acid.

4. Significance and Use

4.1 The purpose of this test method is to determine the amount of organic nitrogen present within a sample of electrical insulation paper or paperboard. Nitrogen content is used to determine if the paper or paperboard has been chemically treated to withstand higher than normal operating temperatures. Such a paper or paperboard is referred to in the industry as "thermally upgraded." A paper or paperboard that is thermally upgraded can withstand higher operating temperatures and allow the electrical equipment to have a longer useful life span.

5. Apparatus

5.1 *Kjeldahl Apparatus,* with 500 or 800-mL flask and a digestion rack, an efficient bulb or scrubber type of trap to ensure that no nonvolatile alkali is carried over, the trap being connected to the flask with a rubber stopper and to the water-cooled condenser following, with rubber tubing. The condenser tube shall be made of alkali-resistant glass or block tin with the discharge end connected to a bent glass delivery tube, the lower end of which is drawn out to a bore of about 3 mm.

5.2 *Other Apparatus*—500-mL Erlenmeyer flask, 100-mL graduated cylinder, 50-mL buret, and mossy zinc or glass beads for the flask to prevent bumping.

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 are also acceptable, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.01 on Electrical Insulating Products.

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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] Reagents Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. 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, NY, and the "United States Pharmacopeia."

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

6.3 *Boric Acid and Indicator Solution*—Dissolve 43 g of boric acid (H_3BO_3) (free from borax), 6 mL of methyl red indicator, and 4 mL of methylene blue indicator, each 0.1 g in 100 mL of 95 % ethyl alcohol, per litre of freshly distilled water. Keep the solution in a borosilicate glass bottle. It is stable for at least 6 months.

NOTE 1—An alternative indicator mixture preferred by some for the boric acid solution is 2 mL of methyl red and 10 mL of bromcresol green, each 0.1 percent solution, in a 95 % ethanol (**1**)[5].

6.4 *Mercuric Oxide,* HgO.

6.5 *Sodium Hydroxide Solution (approximately 50 weight percent)*—Dissolve 1030 g of sodium hydroxide (NaOH) in 1 L of water.

6.6 *Sodium Sulfate,* anhydrous, powdered Na_2SO_4.

6.7 *Sodium Thiosulfate Solution (80 g/litreL)*—Dissolve 80 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 1 L of water.

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

6.9 *Sulfuric Acid, Standard Solution (0.1 N)*—Prepare a 0.1 *N* solution of H_2SO_4 and standardize.

NOTE 2—The substitution of a 0.1 *N* hydrochloric acid (HCl) solution is satisfactory.

7. Sampling

7.1 Obtain the sample to be tested in accordance with Practice D585.

8. Test Specimen

8.1 From the sample of the paper or paperboard, weigh, to the nearest 5 mg, a 2-g specimen consisting of small strips or pieces about 1 cm^2. At the same time, weigh a specimen for the determination of moisture in accordance with Test Method D644.

9. Procedure

9.1 Transfer the test specimen to the Kjeldahl flask and add 10 g of Na_2SO_4, 0.7 g of HgO, and 25 mL of H_2SO_4 (sp gr 1.84). It is convenient to premix the Na_2SO_4 and the HgO. Agitate gently until all the specimen is wet by the acid. Support the flask in an inclined position in a well-ventilated hood and heat the contents with a small flame or electric heater, taking care not to apply heat to the flask above the liquid level. An asbestos-cement board with a hole cut to the proper diameter for the flask is suggested. At first the mixture will froth and turn black. Heat cautiously until frothing has ceased; then increase the heat until the mixture boils gently. The black color gradually fades through brown to a colorless or almost colorless solution. Continue heating for about 1 h after the solution becomes colorless.

NOTE 3—To minimize the inevitable acid condensation in the hood with this procedure, remove most of the acid fumes through a glass tube connected to a water aspirator.

9.2 Allow the solution to cool. It usually will solidify upon cooling to room temperature. When the solution starts to solidify or has cooled to room temperature, *cautiously* add about 300 mL of water and 25 mL of the Na_2SO_4 solution to precipitate the mercury. Allow to stand 5 to 10 min with occasional shaking.

NOTE 4—It has been reported that mercury sometimes volatilizes and amalgamates with tin condenser tubes commonly used in the Kjeldahl apparatus. If the solution is allowed to stand a few minutes after the addition of the Na_2SO_4 solution, volatilization of the mercury is likely to be negligible (**2**)

9.3 Add 50 mL of the boric acid and indicator solution to a 500-mL Erlenmeyer flask, connect the glass delivery tube to the discharge end of the condenser, and adjust the assembly so the tube barely dips beneath the surface of the acid.

9.4 Add the anti-bumping material to the flask, be sure the bulb trap is properly connected to the condenser, and quickly and *carefully* pour 55 mL of cold NaOH solution down the side of the flask so that it will not mix at once with the acid nor subsequently lubricate the rubber stopper. (**Warning**—Cool the acid solution to or below room temperature before adding the NaOH solution. The addition of an ice cube, frozen from distilled water, to each flask in place of part of the 300 mL of water, is a convenient method of cooling.)

9.5 Immediately connect the flask to the bulb trap, forcing the rubber stopper on the bulb trap tightly into the neck of the flask. Swirl the flask slowly and then more rapidly in order to mix the acid with the alkali. Start heating the flask immediately and distill about 150 mL into the receiver. Keep the temperature of the receiver below 40 °C during the distillation.

NOTE 5—Fifty millilitres of the boric acid and indication solution will absorb about 95 mg of nitrogen as ammonia (**2**). Twenty-five millilitres would be sufficient to contain the nitrogen in a 2-g specimen of most papers.

9.6 Disconnect the delivery tube from the end of the condenser and remove the heat from the flask. Do not remove the heat before disconnecting the delivery tube because otherwise it is possible that some of the boric acid may be sucked back. Rinse the delivery tube into the flask, dilute the contents of the flask to about 250 mL, and titrate to a pink end point (pH about 4.9) with the 0.1 *N* acid. During the titration, the color changes from green to gray to pink, the intensity of the pink increasing to red with further addition of acid.

9.7 Make a *blank* determination, carrying through the entire procedure using 1 g of sucrose or dextrose in place of the paper specimen.

10. Calculation

10.1 Calculate the nitrogen content of the specimen as follows:

$$\text{Nitrogen, percent} = \left[(VN \times 0.014)/W \right] \times 100 \qquad (1)$$

where:

V = millilitres of standard acid (corrected for blank) required to titrate the distillate from the specimen,

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[5] The boldface numbers in parentheses refer to the list of references at the end of this method.