Designation: D 1013 – 93 (Reapproved 1998)

Standard Test Method for Determining Total Nitrogen in Resins and Plastics[1]

This standard is issued under the fixed designation D 1013; 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 Department of Defense.

1. Scope

1.1 This test method covers the determination of total nitrogen in nitrogen-containing plastics, resins, and resin solutions. This test method is not applicable for use on materials containing nitro-groups.

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 1193 Specification for Reagent Water[2]

3. Significance and Use

3.1 Total nitrogen content gives an indication of the level or purity of a nitrogen-containing material such as an amine resin. It is important for the quality control of amine resins and is often used to determine whether the proper amounts of many types of nitrogen-containing materials are present in formulated products.

4. Apparatus

4.1 *Kjeldahl Flasks*, for digestion and distillation, 800-mL capacity, moderately heavy wall, and made of hard glass.

4.2 *Connecting Bulbs*, of the Davisson type, or a bulb equally effective in preventing mechanical carry-over of the contents of the distillation flask to the condenser.

4.3 *Digestion and Distillation Equipment*—A suitable Kjeldahl digestion and distillation apparatus, such as any of the well-known commercial units for multiple work. The units may be heated either electrically or by gas burner.

4.4 *Connecting Tubes* made of moderately heavy-wall glass tubing, 6 to 8 in. (150 to 200 mm) in length, for conducting the distillate from the condenser to the receiver.

4.5 *Weighing Tube* (*for Liquid Resins*)—Any convenient device for weighing a few grams of sample in a matter such that no loss of volatile constituents will be sustained during the weighing operation.

5. Reagents

5.1 *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.

5.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5.3 *Hydrochloric or Sulfuric Acid*, (standard 0.5 *N*)—Dilute 43 mL of HCl (sp gr 1.19) or 14 mL of H_2SO_4 (sp gr 1.84) to 1 L with water. Standardize against the 0.5 *N* NaOH solution (3.7), using the same indicator as will be used in titration of the specimen (4.4).

5.4 *Metallic Mercury or Mercuric Oxide*.

5.5 *Methyl Purple Indicator Solution*.

NOTE 1—Methyl purple has recently met with considerable favor, and is to be recommended because of the abruptness of its color change in the presence of ammonium salts. This indicator may be purchased in solution form ready for use.

5.6 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

5.7 *Potassium Sulfate*.

5.8 *Sodium Hydroxide Solution* (760 g/L)—Dissolve 1000 g of technical grade NaOH in 1 L of water.

5.9 *Sodium Hydroxide, Standard Solution* (0.5 *N*)—Prepare a 0.5 *N* NaOH solution free of carbonates, using reagent grade NaOH, and standardize against the National Bureau of Standards standard reference material No. 84 of potassium acid

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved April 15, 1993. Published June 1993. Originally published as D 1013 – 49. Last previous edition D 1013 – 88.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[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 *Analar 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.

phthalate, using phenolphthalein indicator. Protect the solution against CO_2 absorption.

5.10 *Sulfide or Thiosulfate Solution*—Dissolve 40 g of K_2S or Na_2S, or 80 g of $Na_2S_2O_3 \cdot 5H_2O$, in water and dilute to 1 L.

5.11 *Sulfuric Acid* (sp gr 1.84).

6. Procedure

6.1 Transfer a portion of the sample, weighed to 1 mg, to a Kjeldahl flask, using a weighing tube if the material is a liquid. The quantity of sample taken should be an amount that will contain from 150 to 250 mg of nitrogen. Add from 0.5 to 0.75 g of metallic mercury or the equivalent weight of HgO, 10 g of K_2SO_4, and 25 to 35 mL of H_2SO_4 (sp gr 1.84). If the material under test is a urethane resin or polymer the amount of H_2SO_4 (sp gr 1.84) should be increased to 60 mL. If preferred, H_2SO_4 (1 + 1) may be used for digestion in place of H_2SO_4 (sp gr 1.84) in which case the number of millilitres used should be doubled.

6.2 Mix the contents of the flask thoroughly, place on the digestion rack, and heat slowly at first until frothing subsides. Increase the heat until the acid boils briskly then continue the digestion for 2 h after the solution becomes colorless or nearly so.

6.3 After allowing the flask to cool, add about 500 mL of water and a little granular zinc or a few boiling aids to prevent bumping. Add an excess (25 to 30 mL) of K_2S, Na_2S, or $Na_2S_2O_3$ solution. If $Na_2S_2O_3$ solution is used, it should be mixed with the NaOH solution (760 g/L) so that both are added together. Add an excess (80 to 90 mL) of NaOH solution (760 g/L), pouring it slowly down the side of the flask so that it does not mix at once with the acid solution. Immediately connect the flask to the connecting bulb and condenser, and mix the contents of the flask thoroughly.

6.4 Distill the solution into 50 mL of 0.5 *N* HCl or H_2SO_4, making certain that the connecting tube from the condenser extends below the surface of the acid in the receiver. Continue the distillation until the ammonia has been collected in the receiver (about 300 mL of distillate).

6.5 Add 5 to 7 drops of methyl red or methyl purple indicator solution and titrate the excess acid with 0.5 *N* NaOH solution.

6.6 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

7. Calculation

7.1 Calculate the percent nitrogen *A* as follows:

$$A = [((B - V)N \times 0.014)/S] \times 100 \quad (1)$$

where:

B = NaOH solution required for titration of the blank, mL,
V = NaOH solution required for titration of the specimen, mL,
N = normality of the NaOH solution, and
S = specimen weight used, g.

8. Precision and Bias

8.1 On the basis of an interlaboratory test of this test method in which operators in five laboratories analyzed six materials, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same analyst should be considered suspect if they differ by more than 0.25 % absolute.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by analysts in different laboratories, should be considered suspect if they differ by more than 0.5% absolute.

8.2 *Bias*—No bias can be determined since no standard determining nitrogen containing plastics or resin is available.

9. Keywords

9.1 methyl purple; plastic; resins; thiosulfate; total nitrogen