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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Percent of Non-Amines in Fatty Nitrogen Compounds[1]

This standard is issued under the fixed designation D 2082; 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 method was prepared jointly by the American Society for Testing and Materials and the American Oil Chemists' Society.

1. [1]Scope

1.1 This method covers the determination of the percentage of non-amine components in fatty amines and diamines.

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. Summary of Test Method

3.1 A specimen of the fatty amine compound is dissolved in alcohol and passed through an ion exchange column. The amine components of the specimen are retained on the column and the nonamine components pass through the column and are collected and weighed. A correction factor is applied to correct for any amine components that pass through the column.

4. Apparatus

4.1 *Chromatographic Columns*, made by attaching 500-mL bulbs with 24/40 joints to chromatographic tubes and with attached fritted-glass disks inside a 19/22 joint (Fig. 1).

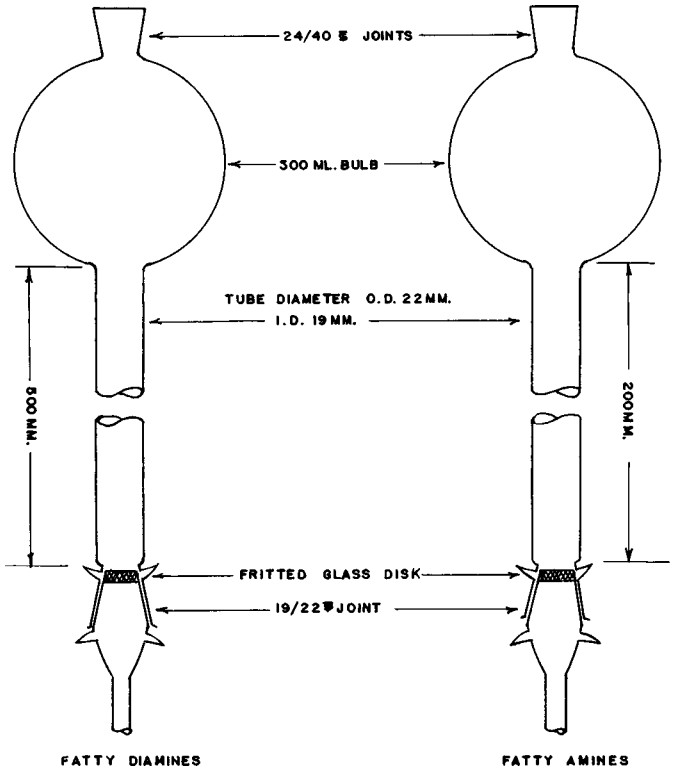

FIG. 1 Diagram of Chromatographic Columns

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[3], where such specifications are available. 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, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5.3 *Bromphenol Blue Indicator Solution*—Dissolve 0.2 g of bromphenol blue in 100 mL of methanol, ethanol, or isopropanol.

5.4 *Hydrochloric Acid, Standard Solution* (0.2*N*)—Add 34 mL of concentrated HCl (sp gr 1.19) to 1000 mL of isopropanol in a 2-L volumetric flask. Make up to volume after cooling to room temperature. Standardize against sodium carbonate using bromcresol green as the indicator.

5.5 *Methanol, Ethanol, or Isopropanol* (99 %).

5.6 *Resin, Cationic, Ion-Exchange*, 500 to 100 mesh.

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[1] This 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 D01.32 on Drying Oils.

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[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.

6. Preparation of Chromatographic Columns

6.1 Place the resin in a 250-mL beaker, pour 100 mL of methanol, ethanol, or isopropanol on the resin, and stir.

6.2 Filter with suction through a 350-mL coarse sintered-glass funnel. Repeat until there is no residue left upon evaporation of the alcohol used to wash the resin. Transfer the washed resin to a bottle and add enough isopropyl alcohol to just cover the resin.

6.3 To fill the chromatographic column first place approximately 10 mm of glass wool on top of the fritted-glass disk then pour a slurry of resin in methanol, ethanol, or isopropanol on top of the glass wool. For fatty amines use a 200-mm column and pour in enough resin to just fill the column after the resin has settled. For fatty diamines a 500-mm column should be packed with 450 mm of resin. Once the resin has been placed in the column, keep it covered with alcohol at all times.

NOTE 1—Blanks should be run on each new batch of resin. Use 80 g of resin for duplicate determinations of fatty amines using 200-mm columns and 200 g of resin for duplicate determinations using 500-mm columns for fatty diamines. Experience has shown that the total quantity of methanol, ethanol, or isopropanol required for washing the resin is 400 mL for 80-g batches and 900 mL for 200-g batches of resin.

7. Procedure

7.1 Accurately weigh into a 150-mL beaker approximately 5 g of the sample for fatty amines or 2.5 g of the sample for fatty diamines. Add 75 mL of alcohol and warm to dissolve the specimens.

NOTE 2—Since some amines are not very soluble in alcohol, it is necessary to heat the chromatographic column bulb so that the amine solution is maintained at 55 to 60°C until it has passed onto the resin. The heating can be discontinued when the 500 mL of alcohol are added. The bulb can be conveniently heated by suspending the bulb through a heating mantle top, of a size to fit a 500-mL flask, which is connected to an autotransformer.

7.2 Allow the alcohol to fall to the top of the resin column and quantitatively transfer the amine solution onto the column so as not to disturb the resin. Wash the beaker with a small quantity of hot (55 to 60°C) alcohol and add to the column.

7.3 Place a 1-L beaker under the column to collect the eluate. When the specimen solution has run down to the top of the resin, slowly add 500 mL of alcohol so as not to disturb the resin. When the alcohol level is low enough, add another 200 mL of alcohol to the bulb. Collect the eluate until the flow stops.

7.4 Evaporate the eluate on the steam bath to about 75 mL and quantitatively transfer it to a tared 250-mL beaker containing several glass beads. Evaporate the concentrated eluate to dryness on the steam bath. Dry the residue in a vacuum oven at 70°C for 2 h at a vacuum (negative gage pressure) of 27 to 29 in. (absolute pressure of 25 to 35 mm Hg).

7.5 Weigh the beaker containing the dried residue. Dissolve the residue in 50 mL of alcohol. Add 5 to 10 drops of bromphenol blue indicator solution and, while swirling, titrate to a yellow end point with 0 to 2 *N* HCl from a microburet.

NOTE 3—The titration is performed to determine the small amount of fatty amine that may be present in the residue. It is assumed that amines that pass through the column have the same mean molecular weight as those in the original sample.

8. Calculation

8.1 Calculate the percent of nonamine as follows:

$$\text{Nonamine, \%} = [1.00 - (V \times N \times 56.1)/(R \times C)] \times (R/S) \times 100 \quad (1)$$

where:

V = millilitres of HCl required for titration of the solution,
N = normality of the HCl,
R = weight of residue, g,
C = amine value of the sample, and
S = specimen weight used, g.

9. Precision and Bias

9.1 Precision and bias were not established at the time this test method was written. An effort is being made to obtain the precision and, if obtainable, it will be published in future revisions. This test method has been in use for many years, and its usefulness has been well established.

10. Keywords

10.1 fatty nitrogen compounds; nonamines