Designation: D 5564 – 95 (Reapproved 2001)

Standard Test Method for Determination of the Total Ammonia Contained in Sulfonated or Sulfated Oils[1]

This standard is issued under the fixed designation D 5564; 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 determination of the total ammonia contained in sulfonated or sulfated oils.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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 500 Test Methods for Chemical Analysis of Sulfonated and Sulfated Oils[2]

D 5350 Test Method for Determination of Organically Combined Sulfuric Anhydride by Titration, Test Method A[2]

3. Significance and Use

3.1 This test method of analysis is intended to determine the total ammonia in a sample of sulfonated or sulfated oil, or both, by boiling a water solution of the sample with excess alkali and determining by titration the loss in alkali after the boiling.

4. Apparatus

4.1 The apparatus required consists of a glass flask provided with a glass stopper and an air condenser. The connection between the flask and the condenser shall be a ground joint. Perforated glass beads shall be used to prevent bumping.

4.1.1 *Flask*—An Erlenmeyer flask (Fig. 1) made of a borosilicate glass, having a capacity of approximately 300 mL and provided with a glass stopper.

4.1.2 *Condenser*, consisting of a glass tube, 915 mm (36 in.) in length and 8 mm (5⁄16 in.) in outside diameter. The lower end of the tube shall be flared and ground to fit the mouth of the Erlenmeyer flask.

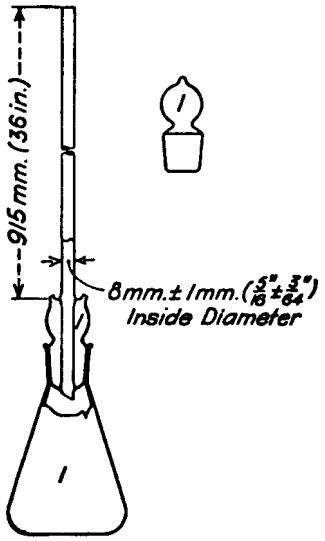

FIG. 1 Apparatus for Determination of Organically Combined Sulfuric Anhydride, Method A

4.1.3 *Glass Beads*—Perforated glass beads, made of chemically resistant glass, approximately 4 mm (5⁄32 in.) in diameter. Before using, the glass beads shall be boiled thoroughly in several portions of water or until the wash water reacts neutral to a methyl orange indicator.

5. Reagents

5.1 *Ethyl Ether*.

5.2 *Methyl Orange Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl orange in 100 mL of water.

5.3 *Sodium Chloride* (NaCl), solid.

5.4 *Sodium Hydroxide, Standard Solution* (1 *N*)—Accurately prepare and standardize a 1 *N* sodium hydroxide (NaOH) solution. Express the strength or concentration of the solution as mg of KOH per mL; 1 mL of 1 *N* NaOH solution is equivalent to 56.1 mg of KOH.

5.5 *Sodium Hydroxide, Standard Solution* (0.5 *N*)— Accurately prepare and standardize a 0.5 *N* NaOH solution. Express the strength of the solution as mg of KOH per mL; 1 mL of 0.5 *N* NaOH solution is equivalent to 28.05 mg of KOH.

5.6 *Sulfuric Acid, Standard* (0.5 *N*)—Accurately prepare and standardize a 0.5 *N* sulfuric acid (H_2SO_4) solution. Express the strength of the solution as mg of KOH per mL; 1 mL of 0.5 *N* H_2SO_4 is equivalent to 28.05 mg of KOH.

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[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 49-1957).

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[2] *Annual Book of ASTM Standards*, Vol 15.04.

5.7 *Sulfuric Acid* (1+19)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into 19 volumes of water while stirring.

6. Procedure

6.1 The procedure consists of two determinations: namely, (*1*) the total alkalinity, and (*2*) the loss in alkalinity after boiling with excess NaOH.

6.1.1 *Total Alkalinity*—Determine the total alkalinity as described in the determination of organically combined sulfuric anhydride, Test Method D 5350; Procedure.

6.1.2 *Alkalinity After Boiling*—Dissolve 10 g of the sample in 100 mL of water in a 500-mL beaker, add 25 mL of 0.5 *N* NaOH solution, and boil the mixture for 30 min or until all the ammonia is expelled as indicated by moistened red litmus paper. Cool the contents of the beaker, add methyl orange indicator solution (0.1 %), and titrate to an approximate end point. Transfer the mixture to a 250-mL glass-stoppered flask and complete the titration (with the addition of salt and ether) as described in the determination of organically combined sulfuric anhydride, Test Method D 5350; Procedure.

7. Calculation

7.1 Calculate the total ammonia, %, as follows:

$$T = A + \{[(B \times D) - (C \times E)]/W\} \quad (1)$$

or

$$\text{total ammonia} = 0.0303\ T \quad (2)$$

where:

T = total ammonia, mg of KOH/g,
A = total alkalinity, mg of KOH/g,
B = mL of NaOH solution added,
C = mL of H_2SO_4 required for titration of the sample,
D = strength of NaOH solution, mg of KOH/mL,
E = strength of H_2SO_4, mg of KOH/mL, and
W = weight of sample, g.

8. Precision and Bias

8.1 This test method is adopted from the procedures of the American Leather Chemists Association, where it has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. The original interlaboratory test data are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

9. Keywords

9.1 leather; sulfated; sulfonated; total ammonia