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Standard Test Method for Determination of the Total Alkalinity of Sulfonated or Sulfated Oils¹

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1. Scope

1.1 This test method covers determination of the total alkalinity of 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 Standard:

D 500 Test Methods for Chemical Analysis of Sulfonated and Sulfated Oils²

2.2 ALCA Method:

H 49 Method for Total Alkalinity and Ammonia³

3. Significance and Use

3.1 This test method is intended to cover determination of the total alkalinity existing in a sample of sulfonated or sulfated oil, or both, by titrating a water solution of the sample with mineral acid in the presence of methyl orange as the indicator. This test method covers determination of the total alkalinity of the fixed alkali, ammonia, and triethanolamine bound as soap, free alkali, and the alkalinity of titratable alkaline salts, but not that of nontitratable alkaline salts.

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.

4.1.3 *Glass Beads*—Perforated glass beads, made of chemically resistant glass, approximately 4 mm (3/32 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 (H₂SO₄) solution. Express the strength of the solution as mg of KOH per mL; 1 mL of 0.5 N H₂SO₄ is equivalent to 28.05 mg of KOH.

5.7 *Sulfuric Acid* (1+19)—Carefully mix 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) into 19 volumes of water while stirring.

6. Procedure

6.1 The procedure consists of two determinations: (1) alkalinity of the sample, designated as *A*; and (2) increase in acidity after boiling the sample with sulfuric acid, designated as *F*.

6.1.1 *Alkalinity, A*—Dissolve 10 g of the sample in 100 mL of water in the 300-mL glass-stoppered Erlenmeyer flask, warming to obtain solution if necessary. After cooling, add 30 g of NaCl, 25 mL of ether, and five drops of methyl orange indicator solution; then add 0.5 N H₂SO₄, with frequent but gentle shaking, until the mixture is slightly acid. Shake the contents of the flask vigorously, and complete the titration by adding first 0.5 N NaOH solution, several drops at a time until the solution is alkaline, and then the acid, one or two drops at a time until the end point is reached. Shake the solution vigorously after each addition of reagent. Drain the burets for 3 min before taking readings. Calculate the alkalinity, *A*, as follows:

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

where:

A = total alkalinity, mg of KOH/g,

B = mL of H₂SO₄ required for titration of the sample,

C = mL of NaOH solution required for titration of the sample,

¹ This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.06 on Chemical Analysis—General.

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² Annual Book of ASTM Standards, Vol 15.04.

³ Available from American Leather Chemists Association, University of Cincinnati—M.L. 14, Cincinnati, OH 45221-0014.