Designation: D 5351 – 93 (Reapproved 1997)

Standard Test Method for Determination of Organically Combined Sulfuric Anhydride by Extraction Titration, Test Method B[1]

This standard is issued under the fixed designation D 5351; 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 the organically combined sulfuric anhydride existing in a sample of sulfated oil by extracting the undecomposed sulfated fat and other fatty matter over an acidulated, concentrated salt solution, boiling the residue with sulfuric acid after evaporating the solvent, and titrating the products of reaction. This test method is applicable only to sulfated oils that split off their combined SO_3 upon boiling with mineral acids, including samples containing sodium acetate or other compounds that cannot be accurately titrated in water solution with methyl orange as the indicator. This test method was derived from Test Methods D 500, Sections 20 through 24, and ALCA Method H-43.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

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 of Chemical Analysis of Sulfonated and Sulfated Oils[2]

2.2 *Other Document:*

ALCA Method H-43 Organically Combined Sulfuric Anhydride Extraction Titration Test (for Sulfated Oils)[3]

3. Significance and Use

3.1 This test method is intended for the determination of organically combined sulfuric anhydride in sulfated oils.

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 borosilicate glass, having a capacity of approximately 300 mL, and provided with a glass stopper.

4.1.2 *Condenser*—The condenser required consists 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 (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 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 milligrams of KOH per millilitre; 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 milligrams of KOH per millilitre; 1 mL of 0.5 *N* NaOH solution is equivalent to 28.05 mg of KOH.

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

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

6. Procedure

6.1 The procedure consists of isolating and purifying the fatty matter as it exists in the original oil by dissolving the sample in a solvent, acidifying and washing with saturated

[1] This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils.
Current edition approved Jan. 15, 1993. Published March 1993.

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

[3] Available from American Leather Chemists Assn., Univ. of Cincinnati-Loc. 14, Cincinnati, OH 45221.