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Designation: D5351 – 93 (Reapproved 2021)

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 D5351; 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 D500, 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils

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.

[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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Leather Chemists Assn., Texas Tech University, P.O. Box 45300, Lubbock, TX 79409.

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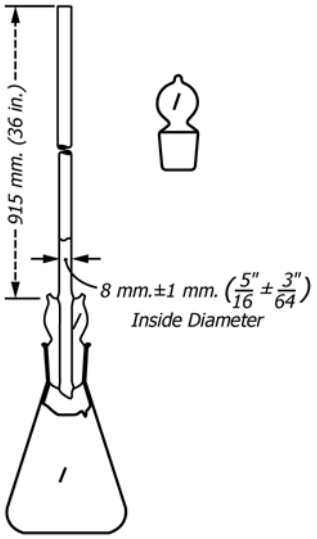

FIG. 1 Apparatus for Determination of Organically Combined Sulfuric Anhydride, Method A

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 brine, and determining the increase in acidity upon boiling the isolated product with sulfuric acid. This increase in acidity is designated as *F*.

6.1.1 *Separation of Purified Oil*—Weigh 5 g to 10 g of the sample, depending upon the concentration of the fatty matter, into a 250-mL separatory funnel containing 50 mL of concentrated NaCl solution, some solid NaCl, five drops of methyl orange indicator solution, and 50 mL of ether. Shake the mixture and neutralize with H_2SO_4 (1 + 19) until the lower layer is distinctly pink (about 0.2 mL in excess). Highly sulfated oils at this stage may form three layers instead of two. In such cases, use a fat solvent consisting of a mixture of two parts of ether and one part of alcohol. Allow the mixture in the separatory funnel to settle for at least 5 min, draw off the lower layer into another separatory funnel, and wash the ether layer with 25-mL portions of NaCl solution until practically neutral to methyl orange, that is, until one drop of 0.5 *N* NaOH solution turns the wash water strongly alkaline. Allow all separations to settle for at least 5 min. Combine the water layers, and extract with two 25-mL portions of ether. Combine the last two ether extractions and wash with NaCl solution until free from acid, as in the case of the ether layer in the first funnel. Combine all the ether layers in the decomposition flask and evaporate the ether.

6.1.2 *Increase in Acidity upon Boiling, F*—Determine the increase in acidity upon boiling in accordance with the procedure described for Test Method A in 6.1.2. Reserve the titrated solution for the subsequent determination of total desulfated fatty matter (Sections 20 to 24 of Test Methods D500). Make a blank determination as described for Test Method A in 6.1.1. Calculate the increase in acidity *F* in accordance with 6.1.2.

7. Calculation

7.1 Calculate the percentage of organically combined sulfuric anhydride as follows:

$$\text{Organically combined sulfuric anhydride, \%} = 0.1426 \times F$$

where:

0.1426 = one tenth the molecular ratio of SO_3: KOH, and
F = increase in acidity upon boiling.

8. Precision and Bias

8.1 Although this test method is widely used, precision and bias information is not available at this time.

9. Keywords

9.1 extraction; leather; sulfated oils; sulfuric anhydride; titration

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