Designation: D 5353 – 95 (Reapproved 2001)

Standard Test Method for Determination of Total Desulfated Fatty Matter[1]

This standard is issued under the fixed designation D 5353; 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 total desulfated fatty matter in a sample of sulfated oils by decomposition with diluted mineral acids and extraction of the decomposed fat. This test method is not applicable to samples that are not completely decomposed upon boiling with mineral acids. This test method was derived from Test Methods D 500, Sections 29 through 32.

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 of Chemical Analysis of Sulfonated and Sulfated Oils[2]

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

D 5351 Test Method for the Determination of Organically Combined Sulfuric Anhydride by Extraction Titration, Test Method B[2]

3. Significance and Use

3.1 This test method is intended for the determination of the total desulfated matter contained in sulfated oils following their total decomposition with dilute mineral acid.

4. Reagents

4.1 *Ethyl Ether*.

4.2 *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. Procedure

5.1 The procedure consists of decomposing the sample with H_2SO_4, extracting the fatty matter with ether, evaporating the solvent, and weighing the residue. After cooling, transfer the titrated solution obtained after determining organically combined sulfuric anhydride in accordance with Test Method D 5350 or Test Method D 5351 (6.1.2) into a 250-mL separatory funnel and shake with 50 mL of ether. Draw off the water layer into another separatory funnel and extract twice with 25-mL portions of ether. Wash the combined ether layers with 15-mL portions of water until the wash water is neutral to methyl orange. Transfer the ether layer to a tared 150-mL beaker, evaporate on the water bath until practically free from solvent, dry in a hot-air oven at 105 to 110°C for 30 min, cool in a desiccator, and weigh. Repeat the heating for 30-min periods until constant weight is obtained.

NOTE 1—Reserve the extracted fatty matter for the subsequent determination of unsaponifiable matter (Sections 37 through 41 of Test Methods D 500).

6. Calculation

6.1 Calculate the total desulfated fatty matter as follows:

$$\text{Total desulfated fatty matter, \%} = (A/B) \times 100$$

where:

A = weight of residue, g, and

B = weight of sample, g.

7. Precision and Bias

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

8. Keywords

8.1 desulfated matter; leather; sulfated fats and oils

[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 45-1957).

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