Designation: D5354 – 95 (Reapproved 2006)

Standard Test Method for Determination of Total Active Ingredients in Sulfonated and Sulfated Oils[1]

This standard is issued under the fixed designation D5354; 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 active ingredients in a sample of sulfonated or sulfated oil, or both, as it exists in the original sample by extracting the undecomposed sulfonated or sulfated fat and other fatty matter over an acidified concentrated salt solution. Free alkali or alkali bound as soap is not included. This test method was derived from Test Methods D500.

NOTE 1—In the case of sulfated oils only, this determination may also be estimated by calculation (see 6.3), as it is equivalent to the sum of the desulfated fatty matter and neutralized organically combined sulfuric anhydride.

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:*[2]

D500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils

3. Significance and Use

3.1 This test method is intended for the determination of the total active ingredients contained in sulfonated and sulfated oils. Free alkali and alkaline soaps are excluded.

4. Reagents

4.1 *Ethyl Ether*.

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

4.3 *Sodium Chloride* (NaCl), solid.

4.4 *Sodium Sulfate* (Na_2SO_4), anhydrous.

4.5 *Potassium Hydroxide, Alcoholic Standard Solution (0.5 N)*—Accurately prepare and standardize a 0.5 *N* alcoholic potassium hydroxide (KOH) solution, the strength of which shall be expressed as milligrams of KOH per millilitre; 1 mL of 0.5 *N* alcoholic KOH solution is equivalent to 28.05 mg of KOH.

5. Procedure

5.1 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 weighing the purified extract. Proceed as described in the determination of organically combined sulfuric anhydride, Test Method B, in the absence of ammonia (6.1.1), retaining the separated active ingredients in the 150-mL beaker instead of transferring it to a crucible. Evaporate the filtrate to a total volume of about 20 mL, add exactly 2 mL of alcoholic KOH solution, mix the contents by swirling, and finally evaporate until practically free from ether.

NOTE 2—With highly sulfonated or sulfated oil, it may be necessary to add 5 to 10 mL of the alcoholic KOH solution in order to stabilize the residue.

5.2 Dry the residue at 108 to 112°C for 1½ h, cool in a desiccator, and weigh. Repeat the heating for 30-min periods until constant weight is obtained.

6. Calculation

6.1 *Correction for the Alkali Added*—Calculate the correction for the alkali added as follows:

$$\text{Correction for alkali added, g} = 0.0006791\ AS \quad (1)$$

where:
A = millilitres of KOH solution added,

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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 46-1957).

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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.