Designation: D 5553 – 95 (Reapproved 2001)

Standard Test Method for Determination of the Unsaponifiable Nonvolatile Matter in Sulfated Oils[1]

This standard is issued under the fixed designation D 5553; 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 unsaponifiable, nonvolatile (above 80°C) matter existing in a sample of sulfated oil by saponifying the desulfated fatty matter and extracting the unsaponifiable matter, and extracting the unsaponifiable matter from the soap solution with ethyl ether.

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

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

D 5353 Test Method for Determination of Total Desulfated Fatty Matter[2]

3. Significance and Use

3.1 This test method is intended for use in the determination of the unsaponifiable, nonvolatile matter contained in sulfated oils for the purpose of quality assurance.

4. Apparatus and Reagents

4.1 *Saponification Flask*—The apparatus required for the saponification consists of a glass flask provided with an air condenser.

4.2 *Ethyl Ether*.

4.3 *Potassium Hydroxide, Alcoholic Solution* (28 g/L)—Dissolve approximately, but not less than, 28 g of KOH in ethyl alcohol and dilute to 1 L.

4.4 *Potassium Hydroxide Aqueous Solution* (28 g/L)—Dissolve 28 g of potassium hydroxide (KOH) in water and dilute to 1 L.

5. Procedure

5.1 The procedure consists of decomposing the sample with mineral acid, extracting the desulfated fatty matter, saponifying the latter, and extracting the unsaponifiable matter from the soap solution with ethyl ether.

5.1.1 *Desulfated Fatty Matter*—Determine the desulfated fatty matter as described in the determination of total desulfated fatty matter. See Test Methods D 5350, D 5351, and D 5353.

5.1.2 *Saponification*—Accurately weigh 2 to 2.5 g of the desulfated fatty matter in the flask, add 25 mL of the alcoholic KOH solution, and simmer the contents (without loss of alcohol) for 1 h with occasional swirling, over an electric hot plate or other source of heat.

5.1.3 *Extraction*—Transfer the content of the flask to a 250-mL separatory funnel and wash the flask several times with a total of 50 mL of water, pouring it into the separatory funnel. Extract the solution while still warm (about 30°C) with 50 mL of ether (rinse the saponification flask with the ether before adding it to the separatory funnel), shaking vigorously for about 1 min, and allow the layers to settle and clear. Draw off the lower layer in a second 250-mL separatory funnel and extract in a similar manner with two 50-mL portions of ether.

5.1.4 *Preliminary Water Washing*—Add 20 mL of water to the combined ether layer, turn the separatory funnel and contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer twice more with 20-mL portions of water, shaking vigorously after each addition.

5.1.5 *Alkali and Water Washing*—Add 20 mL of aqueous KOH solution to the washed ether layer, turn the separatory funnel and contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer with 20 mL of water, shaking vigorously. Continue washing with 20 mL of KOH solution followed by 20 mL of water, shaking vigorously after each addition, until the alkali layer upon strong acidification with HCl and settling for a few minutes is only faintly opalescent. Finally wash the ether

[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 47–1957).

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

layer with 20-mL portions of water until the wash water is no longer pink to phenolphthalein.

5.1.6 *Solvent Removal*—Transfer the ether layer to a 150-mL beaker (counterbalance the beaker if the amount of unsaponifiable matter is small and important) and evaporate over a hot-water bath until practically free of ether. Heat the beaker at 75 to 80°C until constant weight is obtained.

NOTE 1—A 30 % alcoholic solution by volume is the most satisfactory solution from which to extract the unsaponifiable matter.

NOTE 2—If emulsions form, add 5 mL of alcohol, pouring it down the side of the funnel.

NOTE 3—It is important to maintain the volume of ether at not less than 150 mL, otherwise small quantities of unsaponifiable matter may be lost.

NOTE 4—To check whether or not the unsaponifiable matter is free from fatty acids, dissolve the residue in 10 mL of freshly boiled neutral alcohol and titrate with 0.1 *N* alcoholic KOH solution, using phenolphthalein as the indicator. Not more than 0.1 mL should be required for neutralization. If more is required, the determination has not been carried out effectively and must be repeated.

6. Calculation

6.1 Calculate the unsaponifiable matter as follows:

$$U = (R/G) \times P \quad (1)$$

where:

U = unsaponifiable nonvolatile matter, %,
R = weight of residue, g,
G = weight of desulfated fatty matter, g, and
P = desulfated fatty matter, %.

7. Precision and Bias

7.1 This test method is adopted from the procedures of the American Leather Chemists Association where it has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. The original interlaboratory test data are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

8. Keywords

8.1 leather; nonvolatile matter; sulfated oils; unsaponifiable