Designation: D5350 – 95 (Reapproved 2012)

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

This standard is issued under the fixed designation D5350; 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 boiling the sample with sulfuric acid and determining the acidity of the reaction mixture. This test method is applicable only to oils that split off their combined SO_3 upon boiling with mineral acids and that do not contain 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 15 through 19.

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:*[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 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, Standard (0.5 N)*—Accurately prepare and standardize a 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.

5.7 *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.

[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 42–1957).

Current edition approved April 1, 2012. Published April 2012. Originally approved in 1993. Last previous edition approved in 2006 as D5350 – 95(2006). DOI: 10.1520/D5350-95R12.

[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.

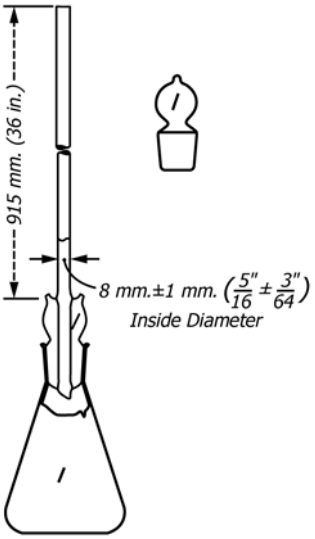

FIG. 1 Apparatus for Determination of Organically Combined Sulfuric Anhydride, Test Method A

6. Procedure

6.1 The procedure consists of two determinations: namely, (*1*) the alkalinity of the sample, designated as *A*, and (*2*) the increase in acidity after boiling the sample with sulfuric acid, designated as *F*.

6.1.1 *Alkalinity, A*—Dissolve 10 g of the sample in 100 mL of water in the 300-mL glass-stoppered Erlenmeyer flask, warming to obtain solution, if necessary. After cooling, add 30 g of NaCl, 25 mL of ether, and five drops of methyl orange indicator solution; then add 0.5 *N* H_2SO_4 with frequent but gentle shaking until the mixture is slightly acid. Shake the contents of the flask vigorously, and complete the titration by adding first 0.5 *N* NaOH solution several drops at a time until the solution is alkaline and then the acid one or two drops at a time until the end point is reached. Shake the solution vigorously after each addition of reagent. Drain burets for 3 min before taking readings. Calculate the alkalinity, *A*, as follows:

$$A = [(B \times D) - (C \times E)]/W \quad (1)$$

where:

- A = total alkalinity, mg of KOH/g,
- B = millilitres of H_2SO_4 required for titration of the sample,
- C = millilitres of NaOH solution required for titration of the sample,
- D = strength of H_2SO_4, mg of KOH/mL,
- E = strength of NaOH solution, mg of KOH/mL, and
- W = weight of sample, g.

6.1.2 *Increase in Acidity upon Boiling, F*—Weigh 10 g of the sample into the Erlenmeyer flask and boil under the air condenser with H_2SO_4 (1+19) for 1½ h or until both the oil and water layers are perfectly clear, using glass beads to prevent bumping. The volume of H_2SO_4 added shall be sufficient to neutralize the total alkalinity, *A*, of the sample plus 25 mL in excess. Regulate the heating so that the solution boils rather vigorously but with very little evaporation taking place. At the end of the heating period allow the contents to cool, wash the condenser with a spray of water from a wash bottle, and disconnect the condenser. Add 30 g of NaCl, 25 mL of ether, 50 mL of water, and five drops of indicator solution and titrate the solution with 1 *N* NaOH solution to the same end point as in the total alkalinity titration, 6.1.1. During the titration stopper the flask frequently and shake the contents of the flask thoroughly. Drain the burets for 3 min before readings are taken.

NOTE 1—Reserve the titrated solution for the subsequent determination of total desulfated fatty matter (Section 29 to 32 of Test Methods D500).

6.1.2.1 Make a blank determination simultaneously with the sample, using the same amount and strength of the H_2SO_4, approximately the same weight of glass beads, and heating and titrating under the same conditions as the sample.

6.1.2.2 Calculate the increase in acidity upon boiling, *F*, as follows:

$$F = [(S - B)N]/W \quad (2)$$

where:

- F = increase in acidity (Note 2) upon boiling, mg of KOH/g,
- S = millilitres of NaOH solution required for titration of the sample,
- B = millilitres of NaOH solution required for titration of the blank,
- N = strength of NaOH solution, mg of KOH/mL, and
- W = weight of sample, g.

NOTE 2—The increase in acidity, *F*, may be negative, in which case retain the sign obtained from the equation in Section 7.

7. Calculation

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

$$\text{Organically combined sulfuric anhydride, \%} = 0.1426(A + F) \quad (3)$$

where:

- 0.1426 = one tenth of the molecular ratio of SO_3:KOH,
- A = total alkalinity, mg of KOH/g, and
- F = increase in acidity upon boiling, mg of KOH/g.
  = Retain the sign of F as obtained in 6.1.2.

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 leather; sulfated oils; sulfuric anhydride; titration