

Standard Test Method for Determination of the Acidity as Free Fatty Acids/Acid Number in the Presence of Ammonium or Triethanolamine Soaps¹

This standard is issued under the fixed designation D 5562; 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 determination of the acidity as free fatty acids in the presence of ammonium or triethanolamine soaps, or both, existing in a sample of sulfonated or sulfated oil, or both, by calculation from the total alkalinity and free-and-alkali combined fatty acids.

1.2 The values 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 A²

3. Significance and Use

3.1 This test method is intended for use in the determination of the acid number of sulfonated and sulfated oils for the purpose of quality assurance.

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 a borosilicate glass, having a capacity of approximately 300 mL and provided with a glass stopper.

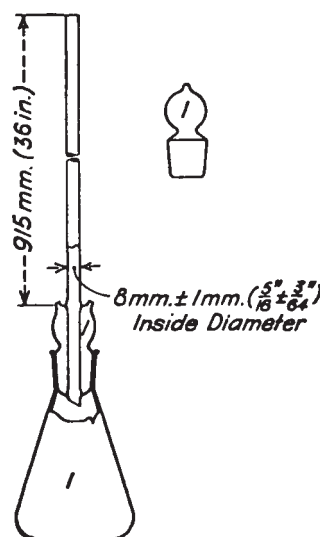


FIG. 1 Apparatus for Determination of Organically Combined Sulfuric Anhydride, Method A

4.1.2 *Condenser*, consisting of a glass tube, 915 mm (36 in.) in length and 8 mm ($\frac{5}{16}$ 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 ($\frac{5}{32}$ 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 a 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 mg of KOH per mL; 1 mL of 1 N NaOH solution is equivalent to 56.1 mg of KOH.

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 52-1956).

Current edition approved May 15, 1995. Published July 1995. Originally published as D 5562 – 94. Last previous edition D 5562 – 94.

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

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 mg of KOH per mL; 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 (H₂SO₄) solution. Express the strength of the solution as mg of KOH per mL; 1 mL of 0.5 N H₂SO₄ is equivalent to 28.05 mg of KOH.

5.7 *Sulfuric Acid (1 + 19)*—Carefully mix 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) into 19 volumes of water while stirring.

6. Procedure

6.1 The procedure consists of two determinations: (1) total alkalinity and (2) free-and-alkali-combined fatty acids.

6.1.1 *Total Alkalinity*—Determine the total alkalinity as described in the determination of organically combined sulfuric anhydride (see Test Method D 5350).

6.1.2 *Free and Alkali-Combined Fatty Acids*—Extract the total sulfated and sulfonated fatty matter as described in the determination of total active ingredients (see Test Method D 5350), but omit the dehydration of the extract (see Note 1). Add exactly 0.5 mL of 0.5 N NaOH solution to the extract, evaporate the ether to a volume of approximately 25 mL, add approximately 50 mL of neutral alcohol, and titrate the mixture with 0.5 N NaOH solution to a phenolphthalein end point.

NOTE 1—In the presence of acetates, wash the ether layer carefully with concentrated NaCl solution until one or two drops of 0.5 N NaOH solution added to the final wash water are neutral to phenolphthalein indicator.

7. Calculation

7.1 Calculate the free and alkali-combined fatty acids as described in the determination of acidity as free fatty acids, (see Test Methods D 5350). Calculate the acidity as free fatty acids in the presence of ammonium or triethanolamine soaps, or both, as follows:

$$K = J - A \quad (1)$$

or

$$\% \text{ free fatty acids} = 0.503 K \quad (2)$$

where:

K = acid number, mg of KOH/g,

J = free and alkali-combined fatty acids, and

A = total alkalinity.

8. Precision and Bias

8.1 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.

9. Keywords

9.1 acidity; free fatty acids; leather; sulfonated and sulfated oils

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