



Designation: D8284 – 19

# Standard Test Method for Determination of the Total Alkalinity in Sulfated, Sulfonated Oils and Fatliquors<sup>1</sup>

This standard is issued under the fixed designation D8284; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method determines the total alkalinity of the fixed alkali, ammonia and triethanolamine which are bound as soap; free alkali, and the alkalinity of titratable alkaline salts, but not that of non-titratable alkaline salts.

1.2 Applicable to sulfated, sulfonated oils and fatliquors used in the softening and stuffing of leather.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

## 3. Apparatus

3.1 The apparatus required consists of an Erlenmeyer flask, 250 or 300 mL, provided with a glass stopper.

## 4. Reagents

4.1 *Sodium Hydroxide, Standard Solution (0.5 N)*—Accurately prepare and standardize a 0.5 N NaOH solution. Express the concentration of the solution as milligrams of

KOH per milliliter; 1 mL of 0.5 N NaOH solution is equivalent to 28.05 mg of KOH.

4.2 *Sulfuric Acid, Standard (0.5 N)*—Accurately prepare and standardize a 0.5 N sulfuric acid ( $\text{H}_2\text{SO}_4$ ) solution. Express the concentration of the solution as milligrams of KOH per milliliter; 1 mL of 0.5 N  $\text{H}_2\text{SO}_4$  is equivalent to 28.05 mg of KOH.

4.3 *Diethyl Ether*—A.C.S. grade.

4.4 *Hexane*—A.C.S. grade.

4.5 *Sodium Chloride (NaCl)*—A.C.S. grade.

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

## 5. Procedure

5.1 Dissolve 10 g of the sample in 100 mL of water in a 300-mL glass-stoppered Erlenmeyer flask, warming to obtain a solution, if necessary. After cooling, add 30 g of NaCl, 25 mL of Diethyl Ether or Hexane, and five drops of methyl orange indicator solution. Slowly add 0.5 N  $\text{H}_2\text{SO}_4$ , a few drops at a time, swirling the flask frequently to combine until the mixture is slightly acidic and the color shifts, from yellow to amber-red. Stopper and shake the contents of the flask vigorously, repeating until vigorous shaking does not cause the color shift to revert. Complete the titration by adding 0.5 N NaOH solution several drops at a time, until the solution is alkaline. Back-titrate with acid, one or two drops at a time until the end point is reached. Shake the solution vigorously after each addition of reagent. Wait 3 min before taking readings of the burette.

## 6. Calculation

6.1 The total alkalinity is expressed as mg KOH/g; calculate it as follows:

$$A = [(B \times D) - (C \times E)] \times 56.1/W \quad (1)$$

where:

A = total alkalinity, mg of KOH/g,

B = millilitres of  $\text{H}_2\text{SO}_4$  required for titration of the sample,

C = millilitres of NaOH solution required for titration of the sample,

D = titer of  $\text{H}_2\text{SO}_4$ , Standard solution (0.5 N)

E = titer of NaOH solution, Standard solution (0.5 N)

<sup>1</sup> 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.

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<sup>2</sup> 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.