



Designation: D4711 – 89 (Reapproved 2017)

Standard Test Method for Sulfonic and Sulfuric Acids in Alkylbenzene Sulfonic Acids¹

This standard is issued under the fixed designation D4711; 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 is applicable to the determination of sulfonic and sulfuric acids in branched and linear alkylbenzene sulfonic acids used as intermediates in synthetic detergents.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 *ASTM Standards:*²

D459 Terminology Relating to Soaps and Other Detergents
E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

3. Summary of Test Method

3.1 A methanolic solution of the sample is titrated with cyclohexylamine in methanol to yield a potentiometric curve. (See Fig. 1.) The first inflection represents the neutralization of strong acids, such as sulfonics and alkylsulfurics, and the first hydrogen of sulfuric acid. The second inflection represents the neutralization of the second hydrogen of sulfuric acid. The amount of sulfonic acid is calculated based on the titrant volume of the first inflection minus that between the two inflections. The amount of sulfuric acid meanwhile is calcu-

¹ This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components. Current edition approved Jan. 1, 2017. Published February 2017. Originally approved in 1987. Last previous edition approved in 2009 as D4711-89(2009). DOI: 10.1520/D4711-89R17.

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

lated from the titrant volume between the two inflections, which is equivalent to the amount of base required for neutralization of the bisulfate anion.

4. Significance and Use

4.1 Alkylbenzene sulfonic acids are important intermediates in the synthetic detergent industry and are defined under "alkyl benzene sulfonate" in Terminology D459. This test method is suitable for the rapid monitoring of the sulfonic and sulfuric acid levels, both of which have a vital bearing on final product performance and appearance.

5. Interferences

5.1 Strong acids, like nitric and hydrochloric, interfere, as do weak acids, such as carboxylic acids. Small amounts of water originally present in the sample do not interfere in the determination. However, if as much as 5 % of water is present in the total solution (solvent plus sample), the end point becomes less sharp.

6. Apparatus

- 6.1 *Potentiometric Titrator*,⁴ and combination calomel reference electrode.
- 6.2 *Buret Assembly*, having a 20 mL buret.⁴
- 6.3 *Beaker*, 180 mL tall form.
- 6.4 *Volumetric Flask*, Class A, 500 mL.
- 6.5 *Magnetic Stirrer*, and stirring bar.

7. Reagents and Materials

- 7.1 *Methanol*, anhydrous,
- 7.2 *Cyclohexylamine* (0.10 N)—Dissolve 10 g of reagent cyclohexylamine in 1000 mL of anhydrous methanol. Standardize against sulfamic acid as described in Section 8.
- 7.3 *Sulfamic Acid*, acidimetric standard.⁵

⁴ Metrohm A436, or its equivalent, has been found suitable for this purpose. Available from Brinkman Instruments.

⁵ J. T. Baker No. 4898, or its equivalent, has been found suitable for this purpose. Available from Sargent-Welch Scientific Co., 7300 Linder Ave., PO Box 1026, Skokie, IL 60077.