



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

This standard is issued under the fixed designation D 4711; 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.

^{ε1} NOTE—Keywords were added editorially in February 1995.

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 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:

D 459 Terminology Relating to Soaps and Other Detergents²

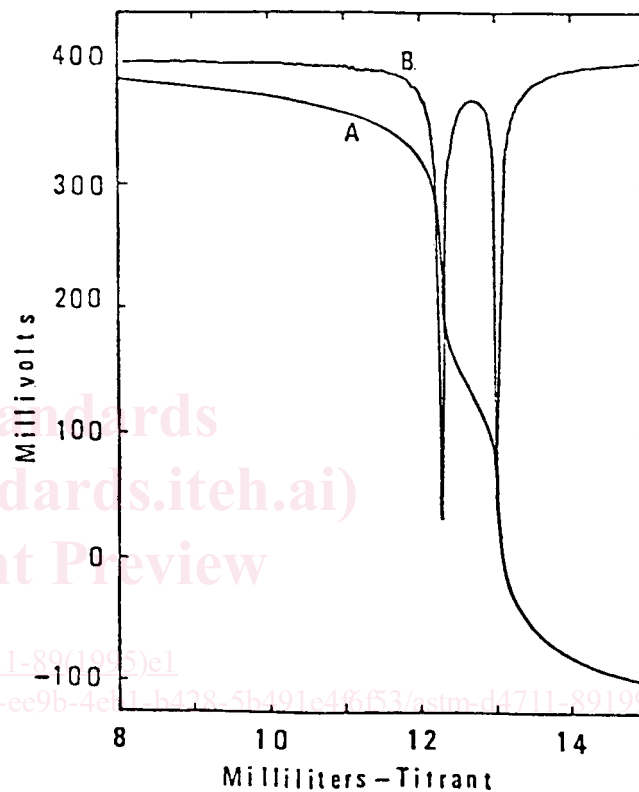
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

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 calculated 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



Solvent. Methanol
Titrant. 0.1 N Cyclohexylamine
A. Differentiating titration curve
B. First derivative curve of the curve A

FIG. 1 Titration of a detergent intermediate containing alkyl sulfonic acid and sulfuric acids.

benzene sulfonate” in Terminology D 459. 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.

¹ This test method is under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis of Soaps and Synthetic Detergents.

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² Annual Book of ASTM Standards, Vol 15.04.

³ Annual Book of ASTM Standards, Vol 15.05.