



Designation: D3049 – 89 (Reapproved 2024)

Standard Test Method for Synthetic Anionic Ingredient by Cationic Titration¹

This standard is issued under the fixed designation D3049; 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 amount of synthetic anionic ingredient in a surfactant by direct titration with a standardized cationic reagent. The test method is a simple and convenient means for the quantitative estimation of the anionic material. The end point is detected by the transfer of a colored complex from an organic solvent phase to an aqueous phase. The colored complex is formed by the addition of a solution of dye to a solution of the anionic surfactant. This complex is soluble in the organic-solvent phase and insoluble in the aqueous phase. When this solution is titrated with a standardized solution of a cationic reagent, the dye is displaced from the colored complex and, being water-soluble, migrates to the aqueous phase. Therefore, a cationic titrating solution that has been standardized against a characterized anionic agent can be used to analyze for other anionics of known molecular mass.

1.2 This test method is applicable to alkylaryl sulfonates, alkyl sulfonates, alkyl sulfates and hydroxy-sulfates, alkylphenol- and fatty alcohol ethoxy-sulfates and dialkylsulfosuccinates. It applies to active materials containing one hydrophilic group per molecule.

1.3 The analytical procedures appear in the following order:

	Sections
Reagents	6
Primary Standard	8
Purity	8.2
Alcohols	8.3
Free Acid or Alkali	8.4
Preparation of Mixed Indicator Solution	9
Preparation of Hyamine Solution	10
Standardization of Hyamine Solution	11
General Procedure for Anionic-Active Material	12

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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. A precautionary statement appears in Section 7. Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

1.6 *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:*
D1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 An aqueous solution of an anionic-type detergent, to which is added a small amount of mixed indicator (dimidium bromide and disulphine blue), is shaken with aqueous Hyamine solution and chloroform. The pink complex which is formed by the reaction between the anionic detergent and the cationic dye, dimidium bromide, is extracted into the chloroform. Increments of additional Hyamine solution are added with a thorough mixing after each addition. At first the reaction takes place between the Hyamine and the excess anionic-type detergent, during which there is no noticeable change in the color (pink) of the chloroform phase. As the equivalence point between the anionic and cationic materials is approached, the dimidium bromide portion of the anionic detergent active-dimidium bromide complex (pink) is gradually released and transferred to the aqueous layer. As excess Hyamine is added it reacts with the anionic dye, disulphine blue, to form a chloroform-soluble blue complex. During the transition at the end point the chloroform layer, therefore, changes from pink to gray, to blue. The gray color is taken as the end point.

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.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.