



Standard Test Method for Active Matter in Anionic Surfactants by Potentiometric Titration¹

This standard is issued under the fixed designation D4251; 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 describes a potentiometric titration procedure for determining the anionic active matter in detergents. It is intended for the analysis of anionic surfactants such as detergent range alkylbenzenesulfonates, α -olefin sulfonates, alcohol sulfates, and alcohol ethosulfates. It has not been tested for surfactant formulations.

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

D1193 Specification for Reagent Water

D1681 Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure

D3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

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 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.

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *active matter*—the organic surface-active material present in the detergent and defined in Terminology D459 as active ingredient of a synthetic detergent.

4. Summary of Test Method

4.1 A detergent sample containing active matter is titrated potentiometrically in an aqueous medium with a standard solution of Hyamine 1622 using a nitrate ion-selective electrode. The titration reaction involves the formation of a complex between the cationic quaternary ammonium titrant (Hyamine 1622) and the anionic surfactant which precipitates. The nitrate electrode probably responds to the concentration of unreacted anionic surfactant.

5. Significance and Use

5.1 Anionic surfactants are the most widely used of the synthetic detergents. ASTM methods in current use for their determination involve two-phase aqueous/chloroform titrations with the organic dyes methylene blue (Test Method D1681) or disulphine blue/dimidium bromide (Test Method D3049) as indicators. One advantage of the potentiometric method is that it eliminates the use of chloroform whose use is restricted for environmental and toxicological reasons.

5.2 This test method is intended for use as described in 1.1.

6. Apparatus

6.1 *Potentiometric Titration Assembly*, consisting of an automatic titrator (Metrohm E536 or equivalent) fitted with a nitrate ion-selective electrode (Orion Model 93-07 Nitrate ISE, or equivalent) and a Ag/AgCl reference electrode (Metrohm EA 440 or equivalent) together with a buret assembly having a 5-mL buret (Metrohm E575) and 150-mL beaker. A TFE-fluorocarbon star-head stirring bar can be used to provide mixing and eliminate foaming during titration. Use of the Orion electrode with a Metrohm E536 requires an adapter (Metrohm EA-1046/2).

NOTE 1—The conditioning of the electrode is essential for obtaining a good break in the titration curve. Conditioning new electrodes in 0.01 M NaNO₃ aqueous solution for 60 min (or more) prior to use is recommended. Condition previously used electrodes by using again for the