Designation: D6173 – 97(Reapproved 2005)

Standard Test Method for Determination of Various Anionic Surfactant Actives by Potentiometric Titration[1]

This standard is issued under the fixed designation D6173; 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 based on a potentiometric titration of common anionic surfactants and blends of anionic surfactant with a hydrotrope. This test method solely is intended for the analysis of active matter in the following surfactants: alcohol ether sulfate, alpha olefin sulfonate, alkylbenzene sulfonic acid, alcohol sulfate, sodium alkylbenzene sulfonate/sodium xylene sulfonate blend (5:1), sodium alkylbenzene sulfonate/sodium xylene sulfonate blend (16:1), and sodium alkylbenzene sulfonate/sodium xylene sulfonate blend (22:1). It has not been tested for surfactant formulations.

1.2 *This standard does not purport to address all 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
D1681 Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure
D3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *active matter, n*—the organic surface-active material present in the detergent. Also defined in Terminology D459 as active ingredient of detergents.

4. Summary of Test Method

4.1 This test method describes a potentiometric titration procedure for determining active matter in anionic surfactant. The anionic surfactant is first dissolved in water, and the pH of the solution is adjusted according to the type of anionic surfactant being measured. In the potentiometric titration the anionic surfactant is titrated with a standard solution of Hyamine using a surfactant electrode, and the reaction involves the formation of a complex between the anionic surfactant and the cationic titrant (Hyamine), which then precipitates. At the end point the surfactant electrode appears to respond to an excess of titrant with potential change large enough to give a well defined inflection in the titration curve.

5. Significance and Use

5.1 The most common anionic surfactants presented in this test method are used widely in synthetic detergents and other household cleaners. Current analysis of the active matter in these anionic surfactants involves the two-phase aqueous/chloroform titration with a mixed indicator, organic dyes such as disulphine blue/dimidium bromide (see Test Method D3049), and methylene blue (see Test Method D1681). This test method eliminates the use of hazardous chloroform, the use of which is restricted for environmental and toxicological reasons.

5.2 This test method also describes the titration of various ratio blends of sodium alkylbenzene sulfonate and sodium xylene sulfonate. Active matter content in these blends is attributable directly to sodium alkylbenzene sulfonate. Therefore, the presence of various amounts of sodium xylene sulfonate in these blends does not interfere with the determination of percent actives.

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[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.

6. Apparatus

6.1 *Autotitration System,* equipped with a delivery buret system, 10 or 20-mL capacity.[3,4]

6.2 *Phoenix Surfactant Combination Electrode*[3,4], nitrate specific ion electrode, or surfactant electrode[4,5] equipped with silver/silver chloride reference electrode.[5] The conditioning of this type of electrode is essential for obtaining a good break in the titration curve. Therefore, each electrode should be conditioned in sodium lauryl sulfate 0.0001M solution for 15 min. For other electrode requirements, follow the manufacturer's instruction manual.

6.3 *Analytical Balance.*

6.4 *Standard Laboratory Glassware.*

7. Reagents[6]

7.1 *Hyamine 1622,* diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride monohydrate.

7.2 *Potassium Chloride,* reagent grade.

7.3 *Triton X-100*[4,7], electrophoresis grade.

7.4 *Potassium Chloride 4.0 M Solution*—Prepare by weighing out 30.00 g to the nearest 0.01 g of potassium chloride into a 100 mL volumetric glass and dissolving in deionized water. Dilute to the mark with deionized water.Add exactly one drop of Triton X-100 to the solution and mix well. This is only a reference electrode filling solution for the Phoenix electrode.

7.5 *Triton X-100, 1 % Solution*—Prepare by weighing 1.00 g of Triton X-100 into a 100-mL volumetric flask and diluting to the mark with deionized water.

7.6 *Sodium Lauryl Sulfate*[4,8], primary standard.

NOTE 1—Sodium lauryl sulfate shall be analyzed for purity according to the reagent section of Test Method D3049 before its use as primary standard.

7.7 *Buffer Solution pH* 4.00[4,9]

7.8 *Buffer Solution pH* 7.00[4,9]—Pipet 10 mL of buffer solution pH 7.00 and transfer to a 100-mL volumetric flask. Dilute to volume and mix well.

7.9 *Sodium Lauryl Sulfate 0.0001 m Solution,* buffered at pH 4.00, with a graduated cylinder transfer 2 mL of sodium lauryl sulfate 0.004 M solution to a 150-mL beaker. Add 80 mL of deionized water and 1 mL of buffer solution pH 4.00 and mix well. This solution is use for conditioning the electrodes.

7.10 *Electrode Rinse Solution*—Transfer approximately 50 mL of 0.1 M HCl solution to 1 L volumetric flask and dilute to volume.

8. Preparation of Primary Standard Reagents

8.1 *Sodium Lauryl Sulfate Solution,* 4×10^{-3} N. Weigh accurately 1.15 + 0.01 g of sodium lauryl sulfate to the nearest 0.1 mg. Dissolve in water and dilute to a final volume of 1L. Calculate the normality of the solution according to the following equation:

$$\text{Normality of Sodium Lauryl Sulfate} = \frac{W \times P}{(288.38 \times 100)} \quad (1)$$

where:

P = purity of the sodium lauryl sulfate, weight %, and
W = weight of sodium lauryl sulfate, g.

8.2 Keep the solution no longer than one month before making a fresh solution.

8.3 *Hyamine 1622 Solution,* 4×10^{-3} N—Dissolve 1.85 + 0.5 g of Hyamine 1622 in deionized water and dilute to a final volume of 1 L.

8.4 *Hyamine 1622 Solution Standardization,* 4×10^{-3} N—This determination shall be done in triplicate. Pipet 5.00 mL of the standard lauryl sulfate solution into a 150-mL beaker. Add 50 mL of dionized water, and, while stirring, add 1 mL of buffer solution pH 4 and 1 mL of Triton-X-100, 1 % solution. To minimize noise in the titration, make sure that the buret tip is placed close to the center of the stir bar. Stir moderately. Erroneous results can occur if excessive foaming takes place. Titrate potentiometrically with the Hyamine 1622 solution and record the titration volume. The Titroprocessor will perform the titration, determine the inflection point and calculate the results according to the following equation:

$$\text{Normality of Hyamine 1622} = \frac{N \times 5}{V} \quad (2)$$

where:

N = normality of sodium lauryl sulfate standard solution,
5 = sodium lauryl sulfate aliquot taken for titration, mL, and
V = Hyamine 1622 solution required to reach the endpoint, mL.

8.5 The electrode should be cleaned between each titration. A satisfactory procedure is to first rinse with the acid rinse solution and then with deionized water. Blot dry with a soft, lint-free tissue. For other electrodes follow the manufacturer's instructions.

9. Hazards

9.1 All reagents and chemicals shall be handled with care. Before using any chemical, read and follow all safety instructions on the manufacturer's label or MSDS (Material Safety Data Sheet).

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[3] The sole source of supply of the autotitration system and Phoenix electrode known to the committee at this time is Brinkmann Instruments, Inc. Cantiague Rd., Westbury, NY 11590.

[4] If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

[5] The sole source of supply of the electrodes known to the committee at this time is Orion Research, Inc., 529 Main St., Boston, MA 02129.

[6] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc., (USPC), Rockville, MD.

[7] The sole source of supply of Triton-X-100 known to the committee at this time is Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785.

[8] The sole source of supply of sodium lauryl sulfate known to the committee at this time is Gallard Schlesinger Chemical Manufacturing Corp., 584 Mineola Ave., Carle Place, NY 11514.

[9] The sole source of supply of buffer solution known to the committee at this time is Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785.