Designation: D5070 – 90 (Reapproved2005)

Standard Test Method for Synthetic Quaternary Ammonium Salts in Fabric Softeners by Potentiometric Titrations[1]

This standard is issued under the fixed designation D5070; 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 the determination of quaternary ammonium salts in fabric softeners. This test method is intended for the analysis of known quaternary ammonium salts such as the dialkyl dimethyl quaternary ammonium compound type and the diamidoamine based quaternary ammonium compound type.

1.2 The quaternary ammonium salts conform to the structures shown in Fig. 1 and Fig. 2.

1.3 The analytical procedure appears in the following order:

1.4 *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.* See 6.4, 6.6, 6.7 and Section 8 for specific warning statements.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration

3. Summary of Test Method

3.1 Quaternary ammonium compounds present in fabric softeners, as the active materials, are titrated potentiometrically in an aqueous medium with a standard solution of sodium lauryl sulfate using a nitrate ion-selective electrode. In this potentiometric titration, the reaction involves the formation of a complex between the quaternary ammonium compound and the anionic surfactant which then precipitates. At the end point, the nitrate ion electrode appears to respond to an excess of titrant with a potential change large enough to give a well defined inflection in the titration curve. Alternatively the quaternary ammonium compound can be first complexed with an excess of standard sodium lauryl sulfate; the excess sodium lauryl sulfate is titrated potentiometrically with standard Hyamine 1622.[3]

4. Significance and Use

4.1 This test method is used to determine the quaternary ammonium salts commonly found in fabric softeners. Quaternary ammonium compounds being the active ingredients in fabric softeners requires accurate determination to assess the cost and performance of such compounds.

5. Apparatus

5.1 *Autotitration System*—buret with 10 or 20 mL capacity;[4] magnetic stirrer;[5] evaluating ruler.[6]

5.2 *Electrodes*—(*1*) nitrate specific ion electrode;[7] (*2*) surfactant electrode;[8] (*3*) Ag/AgCl reference electrode.[9]

[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 sole source of supply of Hyamine 1622 known to the committee at this time is Gallard Schlesinger Manufacturing Corp., 584 Mineola Ave., Carle Place, NY 11514. 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.

[4] Metrohm-Brinkman E-536, or equivalent, has been found satisfactory. Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.

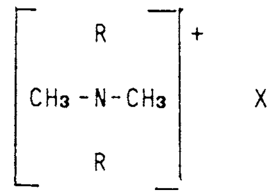
[5] Potentiograph/E-535 and Dosimat/E-459, or equivalent, have been found satisfactory. Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.

[6] Evaluating Ruler EA-893, or equivalent, has been found satisfactory. Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.

[7] Orion Model 93.07, or equivalent, has been found satisfactory. Available from Orion Research Inc., 529 Main St., Boston, MA 02129.

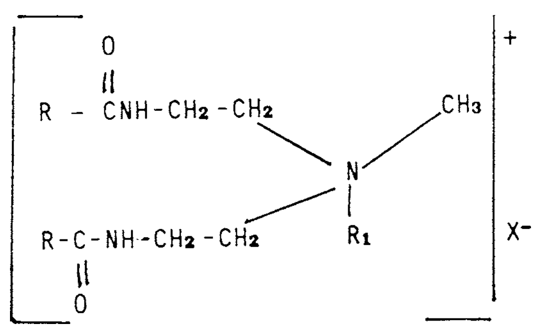
[8] Orion Model 93.42, or equivalent, has been found satisfactory. Available from Orion Research Inc., 529 Main St., Boston, MA 02129.

[9] Metrohm Model EA-440, or equivalent, has been found satisfactory. Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.



X^- = chloride or methyl sulfate
R = fatty alkyl groups saturated or unsaturated, normal or branched C_8 – C_{22}

FIG. 1 Dialkyl Dimethyl Quaternaries



X^- = usually methyl sulfate
R = fatty alkyl groups, saturated or unsaturated, normal or branched C_{12} – C_{18}
R_1 = 2-hydroxyethyl,
2-hydroxypropyl

FIG. 2 Diamidoamine Based Quaternaries

5.3 *Adaptors*—(*1*) coaxial adaptor, required for indicator electrode,[10] (*2*) banana plug adaptor, required for reference electrode.

NOTE 1—To ensure electrical continuity (after assembly) shake down electrode in the manner of a clinical thermometer. Also, the conditioning of the electrode is essential for obtaining a good break in the titration curve. Conditioning new electrodes in 0.01 M KNO_3, aqueous solution for 60 min (or more) prior to use is recommended.

NOTE 2—Other electrodes (for example, a calomel electrode) are suitable as the reference electrode provided they give a stable reference potential during the titration. Reference electrodes having a ceramic or an asbestos junction tend to clog with use. Therefore, a ground-glass sleeve electrode (such as the Metrohm EA 440 or equivalent)[11] is suggested.

6. Reagents

6.1 *Hyamine 1622,*[3]diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride monohydrate.

6.2 *Sodium Lauryl Sulfate,*[12]primary standard (Note 3).

NOTE 3—Sodium lauryl sulfate must be analyzed for purity according to the Reagent section of Test Method D3049, before its use as a primary standard.

6.3 *Water,* type III reagent water conforming to Specification D1193.

6.4 *Isopropanol,* reagent grade. (**Warning**—Highly flammable.)

6.5 *Sodium Borate Decahydrate*—($Na_2B_4O_7$ $10H_2O$), reagent grade.

6.6 *Boric Acid* (H_3BO_3), reagent grade. (**Warning—**Causes irritation.)

6.7 *Sulfuric Acid* (H_2SO_4), reagent grade. (**Warning**—Causes severe burns on contact with skin. See Section 8.)

6.8 *Five percent (V/V) Sulfuric Acid Solution*—Using a graduated cylinder, transfer 80 mL of deionized water to a 100-mL volumetric flask. Slowly, carefully, and with stirring, add 5 mL of concentrated sulfuric acid. Cool to room temperature and dilute to the mark with water.

6.9 *Borate Buffer Solution pH 6.00*—In a 500 mL beaker, dissolve 5.0 g ± 0.02 g of sodium borate decahydrate and 7.0 g ± 0.02 g of boric acid in approximately 300 mL water, with stirring; adjust pH to 6.00 with 5 % sulfuric acid solution. Transfer to a 500-mL volumetric flask, mix, and dilute to volume with water.

7. Preparation of Standard Reagents

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

$$\text{Normality of sodium lauryl sulfate} = \frac{W \times P}{(288.38)\,(100)} \quad (1)$$

where:

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

7.2 Keep the solution no longer than 1 month before making a fresh solution.

7.3 *Hyamine 1622 Solution*, 4×10^{-3} N—Dissolve 1.85 ± 0.5 g of Hyamine 1622 in deionized water. Transfer to a 1 L volumetric flask, and dilute to volume with water.

8. Hazards

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

9. Standardization of Hyamine 1622 Solution

9.1 This determination must be done in triplicate. Pipet 5.00 mL of the standard lauryl sulfate into a 150 mL beaker. Add approximately 100 mL of deionized water and while stirring add by pipet 2 mL of the borate buffer.

9.2 The electrode should be cleaned between each titration. A satisfactory procedure is to first rinse it with water, then with alcohol (ethyl alcohol) (Note 5) and again with water followed by wiping the surface with a soft tissue.

NOTE 4—The electrode can be quickly washed with ethyl alcohol when followed immediately by a water rinse. Prolonged contact of the electrode with alcohol or other organic solvent can cause failure of the electrode membrane.

[10] The Metrohm coaxial adaptor, or equivalent, has been found satisfactory for this purpose. Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.

[11] Available from Brinkman Instruments Inc., Cantiague Rd., Westbury, NY 11590.

[12] Available from British Drug House, LTD, or in the U.S. from Gallard Schlesinger Manufacturing Corp., 584 Mineola Ave., Carle Place, NY 11514.