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Standard Test Method for Disinfectant Quaternary Ammonium Salts by Potentiometric Titration[1]

This standard is issued under the fixed designation D5806; 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 a potentiometric titration procedure for determining active matter in disinfectant quaternary ammonium salts. This test method is intended for the analysis of quaternary ammonium salts used as disinfectants, and only applies to the following commonly used quaternary ammonium salts: *n*-alkyldimethylbenzylammonium chloride (see Fig. 1), cetyltrimethylammonium chloride, and a blend of *n*-octyldecyl dimethylammonium chloride, di-*n*-octyl dimethylammonium chloride, and di-*n*-decyldimethyl ammonium chloride (see Fig. 2). Also, this test method can be applied to the analysis of disinfectant type products where the formula ingredients are known and the quaternary ammonium salt is one of the above. Interferences such as amines oxides and betaines present in disinfectant formulations were not tested.

1.2 This disinfectant quaternary ammonium salt conforms to the structures in Fig. 1 and Fig. 2.

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.* For specific precautionary information, see Section 8.

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[3]

3. Terminology

3.1 *Definitions*—See Terminology D459.

4. Summary of Test Method

4.1 Disinfectant type quaternary ammonium compounds present, as the active materials in disinfectant type products are titrated potentiometrically in an aqueous medium with a standard solution of sodium lauryl sulphate using a nitrate ion-selective electrode or a surfactant electrode (see also Test Method D1681). In this potentiometric titration, the reaction involves the formation of a complex between the disinfectant quaternary ammonium compound and the anionic surfactant which then precipitates. At the end point, the nitrate ion electrode or surfactant 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.

5. Significance and Use

5.1 This test method is used to determine the percent actives in each type of the disinfectant quaternary ammonium salts, and also in the disinfectant products. Quaternary ammonium compounds being the active ingredients in disinfectant-type products require accurate determination to assess the cost and antimicrobial performance of such products.

6. Apparatus

6.1 *Autotitration System,*[4] with 10-mL buret capacity, or 20-mL buret capacity, magnetic stirrer,[5] evaluating ruler,[6] titroprocessor[7] with 10-mL buret capacity or equivalent autotitration system.

[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 of Soaps and Synthetic Detergents.

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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] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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

[5] Potentiograph/E-535 and Dosimat/E-459, or equivalent, have been found satisfactory. Available from Brinkmann Instruments Inc.

[6] Evaluating Ruler EA-893, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

[7] Metrohm-Brinkmann Titroprocessor 670 has been found satisfactory. Available from Brinkmann Instruments Inc.

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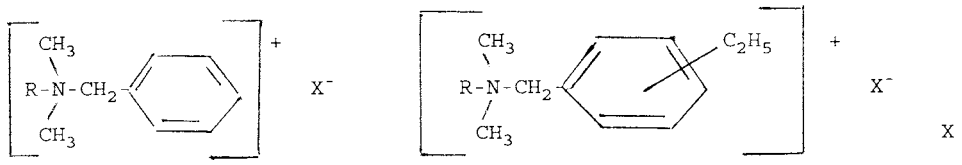
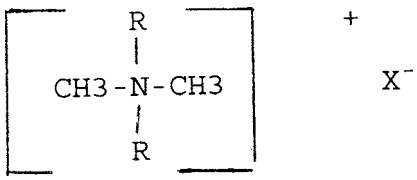


where:
X^- = chloride, and
R = aliphatic, normal $C_8 - C_{22}$.

FIG. 1 *n*-alkyldimethylbenzylammonium chloride and *n*-alkyldimethylethylbenzylammonium chloride



where:
X^- = chloride, and
R = aliphatic, normal $C_8 - C_{22}$.

FIG. 2 Dialkyldimethyl quaternaries

6.2 *Nitrate Specific Ion Electrode,*[8] or surfactant electrode,[9] or equivalent. Silver/silver chloride reference electrode.[10]

6.3 *Metrohm Coaxial Adaptor*, required for indicator electrode.[11] 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 0.004 *M* sodium lauryl sulfate, aqueous solution for 60 min (or more) prior to use is recommended. Also applies to the nitrate or surfactant electrode.

NOTE 2—Other electrodes (for example, calomel electrodes) 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[12] is suggested.

7. Reagents

7.1 *Sodium Lauryl Sulfate,*[13] primary standard (see Note 3).

7.2 Standardize with Hyamine 1622, dried previously at 105°C for 1 h.

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

7.3 *Water, Type III*, reagent water conforming to Specification D1193.

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

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

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

7.7 *Sodium Hydroxide*, (NaOH), reagent grade. (**Warning**—Causes severe burns on contact with skin.)

7.8 *Sodium Hydroxide, 2N Solution*—Dissolve 40 g of sodium hydroxide in approximately 300 mL of deionized water with stirring. Transfer to a 500-mL volumetric flask, dilute to volume with deionized water, and mix well.

7.9 *Borate Buffer Solution*—Dissolve 1.5 g $Na_2B_4O_7 \cdot 10$ H_2O and 1.0 g H_3BO_3 in approximately 200 mL deionized water, with stirring; adjust pH to 9.5 with 2N HaOH, transfer to a 1000-mL volumetric flask, mix and dilute to volume with deionized water.

7.10 *Octoxynol-9 Nonionic Surfactant.* [14]

7.11 *Triton Solution, 1 %*—Pipet 1 mL of the octoxynol-9 nonionic surfactant and transfer to a 100-mL volumetric flask diluted to volume with deionized water.

7.12 *Sodium Lauryl Sulfate Solution, 8×10^{-3} N*—Weigh accurately 2.42 ± 0.01 g of sodium lauryl sulfate to nearest 0.1 mg; dissolve in water and dilute to a final volume of 1 L. Determine 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 %, and
W = weight of sodium lauryl sulfate, g.

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

8. Hazards

8.1 Handle all reagents and chemicals with care. Before using any chemical, read and follow all safety precautions and instructions of the manufacturer label or MSDS (Material Safety Data Sheet).

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

[9] Orion Model 93.42, or equivalent, has been found satisfactory. Available from Orion Research Inc.

[10] Metrohm Model EA-440, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

[11] The Metrohm coaxial adaptor, or equivalent, has been found satisfactory for this purpose. Available from Brinkmann Instruments Inc.

[12] The Metrohm WA-440, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

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

[14] Triton-X-100 has been found satisfactory. Available from Fisher Scientific Cat. #BP151-100.

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