



## Standard Test Method for Disinfectant Quaternary Ammonium Salts by Potentiometric Titration<sup>1</sup>

This standard is issued under the fixed designation D 5806; 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 ( $\epsilon$ ) 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:

D 459 Terminology Relating to Soaps and Other Detergents<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

D 1681 Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure<sup>2</sup>

D 3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration<sup>2</sup>

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>4</sup>

### 3. Terminology

3.1 *Definitions*—See Terminology D 459.

### 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 D 1681). 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.

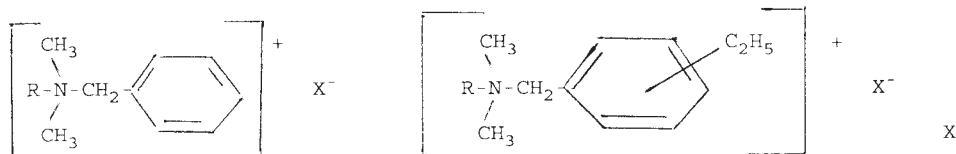
<sup>4</sup> Annual Book of ASTM Standards, Vol 15.05.

<sup>1</sup> 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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<sup>2</sup> Annual Book of ASTM Standards, Vol 15.04.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.01.

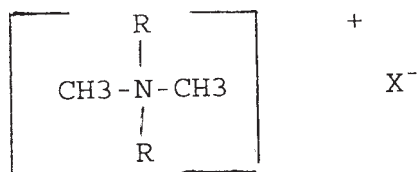


where:

$X^-$  = 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. Apparatus

6.1 *Autotitration System*,<sup>5</sup> with 10-mL buret capacity, or 20-mL buret capacity, magnetic stirrer,<sup>6</sup> evaluating ruler,<sup>7</sup> titroprocessor<sup>8</sup> with 10-mL buret capacity or equivalent autotitration system.

6.2 *Nitrate Specific Ion Electrode*,<sup>9</sup> or surfactant electrode,<sup>10</sup> or equivalent. Silver/silver chloride reference electrode.<sup>11</sup>

6.3 *Metrohm Coaxial Adaptor*, required for indicator electrode.<sup>12</sup> 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<sup>13</sup> is suggested.

## 7. Reagents

7.1 *Sodium Lauryl Sulfate*,<sup>14</sup> 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 D 3049 before using as a primary standard.

7.3 *Water, Type III*, reagent water conforming to Specification D 1193.

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

7.5 *Sodium Borate Decahydrate*, ( $Na_2B_4O_7 \cdot 10H_2O$ ), 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  $Na_2B_4O_7 \cdot 10H_2O$  and 1.0 g  $H_3BO_3$  in approximately 200 mL deionized water, with stirring; adjust pH to 9.5 with 2N NaOH, transfer to a 1000-mL volumetric flask, mix and dilute to volume with deionized water.

7.10 *Octoxynol-9 Nonionic Surfactant*.<sup>15</sup>

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,  $8 \times 10^{-3}$  N*—Weigh accurately  $2.42 \pm 0.01$  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:

<sup>13</sup> The Metrohm WA-440, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

<sup>14</sup> 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.

<sup>15</sup> Triton-X-100 has been found satisfactory. Available from Fisher Scientific Cat. #BP151-100.

<sup>5</sup> Metrohm-Brinkmann E-536, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc., Catiague Rd., Westbury, NY 11590.

<sup>6</sup> Potentiograph/E-535 and Dosimat/E-459, or equivalent, have been found satisfactory. Available from Brinkmann Instruments Inc.

<sup>7</sup> Evaluating Ruler EA-893, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

<sup>8</sup> Metrohm-Brinkmann Titroprocessor 670 has been found satisfactory. Available from Brinkmann Instruments Inc.

<sup>9</sup> Orion Model 93.07, or equivalent, has been found satisfactory. Available from Orion Research Inc., 529 Main St., Boston, MA 02129.

<sup>10</sup> Orion Model 93.42, or equivalent, has been found satisfactory. Available from Orion Research Inc.

<sup>11</sup> Metrohm Model EA-440, or equivalent, has been found satisfactory. Available from Brinkmann Instruments Inc.

<sup>12</sup> The Metrohm coaxial adaptor, or equivalent, has been found satisfactory for this purpose. Available from Brinkmann Instruments Inc.



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

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).

## 9. Procedure for Determination of Disinfectant Quaternary Ammonium Salt or Disinfectant Type Product

9.1 Weigh accurately a quantity of sample to contain approximately 0.056 meq/10 mL of disinfectant quaternary ammonium compound active material into a 200-mL volumetric flask (Note 4). Dilute to volume with deionized water. Mix well. Pipet the corresponding aliquot, then add approximately 150 mL of water and stir. While stirring, pipet in 10 mL of the borate buffer solution, 2 mL of isopropanol, and 2 mL of 1 % octoxynol-9 nonionic surfactant. Titrate potentiometrically with standard sodium lauryl sulfate solution. As the inflection point is approached, reduce the addition rate and continue titrating well past the inflection in the titration curve. (Automatic titrators can be preset to automatically slow down the addition rate as the inflection point is approached.)

NOTE 4—To determine the amount of sample needed for an approximately 5 to 7 mL titration of 0.0056 meq use the following equation:

$$W = \frac{0.0056 \times M}{D} \quad (2)$$

where:

$W$  = weight of sample to be taken for analysis, g,

$M$  = average molecular weight of the anionic active matter present, and

$D$  = approximate concentration of anionic active matter expected, weight %.

9.2 To obtain accurate sample weights of sample, it is convenient to dissolve the disinfectant quaternary ammonium compound in deionized water and take an aliquot corresponding to a known meq of active matter. For example, for a disinfectant quaternary ammonium compound containing 28.0 % actives molecular weight 335, weigh 1.3 of sample into a 200-mL volumetric flask, dilute with water, and take a 10-mL aliquot. Add 150 mL of water, 10 mL of borate buffer solution, 2 mL of 1 % Triton solution, and 2 mL of isopropanol. Titrate with the standard 0.008 N solution of sodium lauryl sulfate.

9.3 Record the titration volume at the end point. The endpoint is marked by the point of inflection on "S" shaped curve and it is determined by the use of metrohm evaluating

ruler. A typical titration curve of a disinfectant quaternary ammonium compound is shown in Fig. 3.

## 10. Calculation

10.1 Calculate the percent actives in the sample as follows:

Disinfectant quaternary ammonium compound weight % = (3)

$$\frac{A \times N \times M \times D \times 100}{S \times 1000 \times A_1}$$

where:

$A$  = standard sodium lauryl sulphate solution consumed during titration, mL,

$N$  = normality of standard sodium lauryl sulfate solution,

$M$  = average equivalent weight of the disinfectant quaternary ammonium compound,

$S$  = weight of sample, g,

$D$  = initial sample dilution, mL (that is, 200 mL), and

$A_1$  = aliquot taken for titration (that is, 10 mL).

## 11. Precision and Bias

11.1 *Repeatability (Single Analyst)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on differed days, has been estimated to be 0.25 % absolute at 23 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.80 % absolute. (See Practice E 180.)

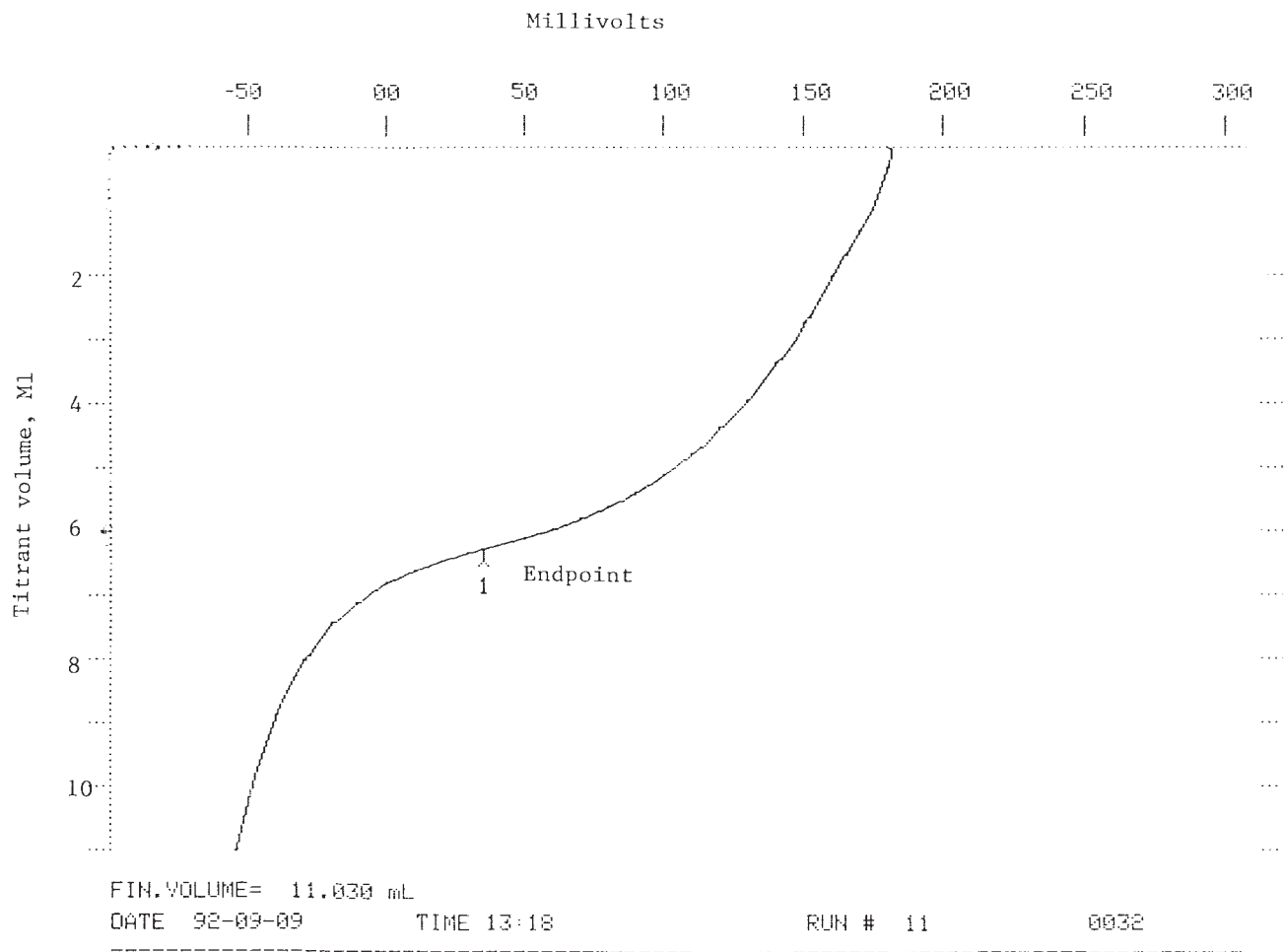
11.2 *Reproducibility (Multilaboratory)*:

11.2.1 *n-Alkyldimethylbenzylammonium Chloride*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.24 % absolute at 23 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.8 % absolute.

11.2.2 *Cetyltrimethylammonium Chloride*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.38 % absolute at 23 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 1.4 % absolute.

11.2.3 *Blend of n-octyldecyldimethylammonium Chloride, Di-n-decyldimethyl Ammonium Chloride and Di-n-decyldimethylammonium Chloride*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 1.9 % absolute at 23 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 4.4 % absolute.

NOTE 5—The precision data were derived from results of the cooperative tests by six laboratories on the following disinfectant quaternary ammonium salts: (equivalent weights in parentheses are based on commercial disinfectant quaternary ammonium salts) *n*-octyldecyldimethylammonium chloride, *di-n*-octyldimethylammonium chloride, and *di-n*-decyldimethylammonium chloride (335), *n*-alkyldimethylbenzylammonium chloride (358), cetyltrimethylammonium chloride (320).



**FIG. 3 Sample: *n*-alkyldimethylbenzylammonium chloride**  
**Titration: 0.008N Sodium lauryl sulfate. Using nitrate ion selective electrode.**

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