Designation: D 2080 – 92 (Reapproved 1998)

Standard Test Method for Average Molecular Weight of Fatty Quaternary Ammonium Chlorides[1]

This standard is issued under the fixed designation D 2080; 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.

This method was prepared jointly by the American Society for Testing and Materials and the American Oil Chemists' Society.

1. Scope

1.1 This test method covers the determination of the average molecular weight of a fatty quaternary ammonium chloride by converting to the acetate, titrating potentiometrically, and correcting for the nonquaternary components.

1.2 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 2076 Test Methods for Acid Value and Amine Value of Fatty Quaternary Ammonium Chlorides[3]

D 2077 Test Method for Ash in Fatty Quaternary Ammonium Chlorides[3]

D 2079 Test Method for Nonvolatile Matter (Solids) in Fatty Quaternary Ammonium Chlorides[3]

E 70 Test Method for pH of Aqueous Solutions With the Glass Electrod[4]

3. Apparatus

3.1 *Buret*, having a capacity of 25 mL.

3.2 *Glass Electrode pH Meter*, conforming to the requirements of Test Method E 70[4] or similar potentiometric titrator, and carefully standardized in accordance with the manufacturer's instructions.

3.3 *Magnetic Stirrer*, with inert plastic-coated stirring bar.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

4.3 *Acetic Acid* (*Glacial*) (CH_3COOH).

4.4 *Acetic Anhydride* $(CH_3CO)_2O$.

4.5 *Chloroform* ($CHCl_3$).

4.6 *Mercuric Acetate Solution*—Dissolve 6 g of mercuric acetate [$Hg(C_2H_3O_2)_2$] in 100 mL of glacial acetic acid. Prepare fresh for each determination.

Caution—Mercury compounds are harmful and accumulate in the aquatic environment. Mixtures containing mercury compounds should not be flushed down a drain but disposed of as a hazardous waste.

4.7 *Perchloric Acid, Standard Solution* (0.1 *N*):

4.7.1 Add 28.4 g of 70 to 72 % perchloric acid ($HClO_4$) to 1000 mL of glacial acetic acid in a 2-L beaker while stirring. Carefully add 46.6 g of acetic anhydride while stirring. Carefully pour the solution through a glass funnel into a 2-L volumetric flask and dilute to mark with glacial acetic acid. Mix the solution and allow to stand for 24 h before standardizing.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.32 on Drying Oils.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 06.03.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *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.

4.7.2 Standardize against acid potassium phthalate ($HKC_8H_4O_4$).[6] Weigh 0.31 to 0.39 g to the nearest 0.1 mg of finely ground and dried $HKC_8H_4O_4$ into a 200-mL beaker. Add 50 mL of glacial acetic acid and warm gently to dissolve. Cool and add an additional 50 mL of glacial acetic acid so as to wash down the sides of the beaker. Insert the stirring bar into the beaker and adjust the beaker so that the lower half of each electrode of the pH meter is immersed in the solution. Start the stirrer and adjust the speed so that there is vigorous stirring without spattering. Titrate with the $HClO_4$ solution using the millivolt scale. Record the millivolt readings every millilitre, but in the vicinity of the end point, record the millivolt readings every 0.1 mL. Plot a graph showing the millivolts against the millilitres required for titration. Calculate the normality of the $HClO_4$ solution as follows:

$$\text{Normality} = (4.8967 \times A)/V \quad (1)$$

where:

A = grams of $HKC_8H_4O_4$ used, and
V = millilitres of the $HClO_4$ solution required for titration of the $KHC_8H_4O_4$.

5. Procedure

5.1 Determine the acid value, amine value, percent of free amine, and percent amine hydrochloride in accordance with Test Methods D 2076. Determine the percent of ash in accordance with Test Method D 2077. Determine the percent of nonvolatile matter in accordance with Test Method D 2079.

5.2 Melt the sample if not liquid, in a water bath, mix thoroughly, and weigh 1.0 to 1.5 g to the nearest 0.1 mg. The weight will vary somewhat with the average molecular weight.

5.3 Add 100 mL of glacial acetic acid and heat gently, if necessary to affect solution. Add 15 mL of $Hg(C_2H_3O_2)_2$ solution. Insert the stirring bar into the beaker and stir for 5 min.

5.4 Add 20 mL $CHCl_3$ and adjust the beaker so that the lower half of each electrode of the pH meter is immersed in the solution. Adjust the speed of the stirrer so that there is vigorous stirring without spattering. Titrate with 0.1 N $HClO_4$ using the millivolt scale. Record the millivolt readings every millilitre, but in the vicinity of the end point, record the millivolt readings every 0.1 mL. Plot a graph showing the millivolts against titration.

6. Calculation

6.1 Calculate the average molecular weight as follows:

$$\text{Average molecular weight} = \frac{(L - A - C - D)}{\left[\left(\frac{V \times N}{S \times 10}\right) - \frac{A}{58.5} - \frac{E}{561} - \frac{F}{561}\right]} \quad (2)$$

where:

L = percent of nonvolatiles,
A = percent of ash,
C = percent of amine hydrochloride,
D = percent of free amine,
E = acid value,
F = amine value,
N = normality of the $HClO_4$ solution,
S = specimen weight used, g, and
V = millilitres of $HClO_4$ required for titration of the solution.

7. Precision and Bias

7.1 Precision and bias were not established at the time this test method was written. An effort is being made to obtain the precision and, if obtainable, it will be published in future revisons. This test method has been in use for many years, and its usefulness has been well established.

8. Keywords

8.1 molecular weight; quaternary ammonium chlorides

[6] National Institute of Standards and Technology standard sample No. 84f of HKC_8H_4O is recommended for this purpose and should be treated as directed in the certificate of analysis accompanying the standard sample.