Designation: D 2076 – 92 (Reapproved 1998)

Standard Test Methods for Acid Value and Amine Value of Fatty Quaternary Ammonium Chlorides[1]

This standard is issued under the fixed designation D 2076; 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.

These test methods were prepared jointly by ASTM and the American Oil Chemists' Society.

1. Scope

1.1 These test methods cover the determination of acid value and amine value in fatty quaternary ammonium chlorides.

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]

3. Terminology

3.1 *Definitions:*

3.1.1 *acid value*—the number of milligrams of potassium hydroxide needed to neutralize 1 g of sample, and is usually due to amine hydrochloride.

3.1.2 *amine value*—the number of milligrams of potassium hydroxide equivalent to the fatty amine basicity in 1 g of sample.

4. Apparatus

4.1 *Erlenmeyer Flasks*, wide-mouth, alkali-resistant, borosilicate glass, 250-mL capacity.

4.2 *Micro Buret*, 10-mL capacity graduated to 0.02 mL.

5. Reagents

5.1 *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.[3] 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.

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

5.3 *Bromphenol Blue Indicator Solution*—Dissolve 0.2 g of bromphenol blue in 100 mL of methanol, ethanol, or isopropanol.

5.4 *Hydrochloric Acid, Standard Solution* (0.1 *N*)—Add 17 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1000 mL of isopropyl alcohol in a 2-L volumetric flask. Make up to volume after cooling to room temperature. Standardize with sodium carbonate using 0.1 % bromcresol green as the indicator.

5.5 *Hydrochloric Acid, Standard Solution* (0.2 *N*)—Add 34 mL of concentrated HCl (sp gr 1.19) to 1000 mL of isopropyl alcohol in a 2-L volumetric flask. Make up to volume after cooling to room temperature. Standardize with sodium carbonate using 0.1 % bromcresol green as the indicator.

5.6 *Isopropyl Alcohol* (99 %)—Neutralize to the phenolphthalein end point with 0.1 *N* methanolic sodium hydroxide (NaOH) just before running the acid value test.

5.7 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

5.8 *Sodium Hydroxide, Standard Solution* (0.1 *N*)—Dissolve 4.0 g of sodium hydroxide (NaOH) in 1000 mL of cold methyl alcohol. Allow to stand overnight in a cold room. Siphon the supernatant liquid into a clean bottle. After coming

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

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

[3] *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.

to room temperature, standardize the material with acid potassium phthalate using 1 % phenolphthalein as the indicator.

6. Procedure for Acid Value

6.1 Melt the specimen, if it is not already liquid, in a water bath, mix thoroughly, and weigh 5 to 20 g to 1 mg into a 250-mL flask. Add 100 mL of neutralized alcohol and swirl to dissolve. Heat if necessary.

6.2 Add 1 mL of phenolphthalein indicator. If the solution remains colorless, titrate with 0.1 *N* NaOH solution to the appearance of the first persistent pink color of the same intensity as that of the neutralized alcohol before addition. If the solution turns pink, the presence of free caustic material is indicated. If free caustic material is present proceed in accordance with Section 8.

7. Procedure for Amine Value

7.1 Weigh 5 to 20 g of sample to 1 mg into a 250-mL flask. Add 100 mL of alcohol and swirl to dissolve. Heat if necessary.

7.2 Add 1 mL of bromphenol blue indicator and titrate with 0.2 *N* HCl to a yellow end point.

NOTE 1—If the amine value is very low, 0.1 *N* HCl should be used for better accuracy.

8. Procedure for Free Caustic Material

8.1 Titrate the specimen with 0.2 *N* HCl until the solution turns colorless.

8.2 Add 1 mL of bromphenol blue indicator and continue the titration to a yellow end point.

9. Calculation

9.1 Calculate the acid value and amine value as follows:

$$\text{Acid value or amine value} = (V \times N \times 56.1)/S \quad (1)$$

where:
- V = titrant used, mL,
- N = normality of titrant used, and
- S = specimen weight used, g.

9.2 If free caustic material is present, calculate the percent of free sodium hydroxide and the amine value as follows:

$$\text{Free sodium hydroxide, \%} = (A \times N \times 4)/S \quad (2)$$

$$\text{Amine value} = (V \times N \times 56.1)/S \quad (3)$$

where:
- A = HCl required for titration of the solution to the phenolphthalein end point, mL,
- V = HCl required for titration of the solution from the phenolphthalein end point to the bromphenol blue end point, mL,
- N = normality of the HCl used, and
- S = specimen weight used, g.

9.3 Calculate the percent of amine hydrochloride and free amine as follows (Note 2):

$$\text{Amine hydrochloride or free amine, \%} = (A \times V)/561 \quad (4)$$

where:
- A = acid value or amine value, and
- V = molecular weight of the amine hydrochloride or free amine (Table 1).

NOTE 2—Molecular weights listed in Table 1 are averages of products from several processors. They are suitable for use with this test method only, since normal variations between different products do not seriously affect the calculated percent. They are not suitable in other tests, such as the determination of the percent of quaternary ammonium chlorides by titration.

10. Precision and Bias

10.1 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. These test methods have been in use for many years, and its usefulness has been well established.

11. Keywords

11.1 acid value; amine value; quaternary ammonium chlorides

TABLE 1 Molecular Weights

Fatty Quaternary Ammonium Chloride (QAC)	Amine Molecular Weight	Amine Hydrochloride Molecular Weight
Lauryl trimethyl QAC	184	220
Myristyl trimethyl QAC	215	252
Palmityl trimethyl QAC	248	284
Stearyl trimethyl QAC	274	310
Oleyl trimethyl QAC	268	304
Tall oil fatty trimethyl QAC	282	318
Coco trimethyl QAC	210	246
Tallow trimethyl QAC	260	296
Cotton trimethyl QAC	268	304
Dilauryl dimethyl QAC	350	386
Dimyristyl dimethyl QAC	401	438
Dipalmityl dimethyl QAC	454	490
Distearyl dimethyl QAC	539	576
Ditall oil fatty dimethyl QAC	504	540
Dicoco dimethyl QAC	387	424
Dihydrogenated tallow dimethyl QAC	505	542
Furfuryl hydrogenated tallow dimethyl QAC	354	390