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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Fatty Nitrogen Products[1]

This standard is issued under the fixed designation D 2071; 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 testing of fatty nitrogen products. They specify the use of other ASTM methods that are published separately. The test methods included are listed in Table 1 .

1.2 The test methods cover the following compounds:

1.2.1 *Fatty Amines*—Fatty primary amines, difatty secondary amines, trifatty tertiary amines.

1.2.2 *Fatty Quaternary Ammonium Chloride*—Trimethyl fatty quaternary ammonium chloride, dimethyl difatty quaternary ammonium chloride.

1.2.3 *Fatty Amidoamines*—Reaction products of fatty acids with an excess of polyamines.

1.2.4 *Fatty Diamines—N*-fatty-1,3-propylene diamines.

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.*

TABLE 1 List of Test Methods

Test Method	Sections	ASTM Designation of Test Method
Definition	4	
All Fatty Nitrogen Products:		
Sampling	5	D 1466
Color by the Gardner Method	6	D 1544
Color by the APHA Method	7	D 1209
Water by the Karl Fischer Method	8	D 2072
Fatty Amines:		
Amine values	9	D 2073
		D 2074
Iodine value	10	D 2075
Percent non-amine	11	D 2082
Primary, secondary, tertiary amines	12	D 2083
Fatty Quaternary Ammonium Chlorides:		
Acid value and amine value	13	D 2076
Ash	14	D 2077
Flash point	15	D 56
		D 88
		D 92
		D 93
		D 1310
		D 3278
Iodine value	16	D 2078
Nonvolatile matter	17	D 2079
Average molecular weight	18	D 2080
pH	19	D 2081
Fatty Amidoamines:		
Amine values	20	D 2073
Iodine value	21	D 2075
Fatty Diamines:		
Amine values	22	D 2073
Iodine values	23	D 2075
Percent non-amines	24	D 2082

2. Referenced Documents

2.1 *ASTM Standards:*

D 56 Test Method for Flash Point by Tag Closed Tester[2]

D 88 Test Method for Saybolt Viscosity[3]

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup[2]

D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester[2]

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[4]

D 1310 Test Method for Flash Point and Fire Points of Liquids by Tag Open-Cup Apparatus[5]

D 1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials[6]

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)[5]

D 2072 Test Method for Water in Fatty Nitrogen Compounds[6]

D 2073 Test Methods for Total, Primary, Secondary, and Tertiary Amine Values of Fatty Amines, Amidoamines, and Diamines by Referee Potentiometric Method[6]

D 2074 Test Methods for Total, Primary, Secondary, and Tertiary Amine Values of Fatty Amines by Alternative Indicator Method[6]

[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 05.01.

[3] *Annual Book of ASTM Standards,* Vol 04.04.

[4] *Annual Book of ASTM Standards,* Vol 06.04.

[5] *Annual Book of ASTM Standards,* Vol 06.01.

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

D 2075 Test Method for Iodine Value of Fatty Amines, Amidoamines, and Diamines[6]
D 2076 Test Methods for Acid Value and Amine Value of Fatty Quaternary Ammonium Chlorides[6]
D 2077 Test Method for Ash in Fatty Quaternary Ammonium Chlorides[6]
D 2078 Test Method for Iodine Value of Fatty Quaternary Ammonium Chlorides[6]
D 2079 Test Method for Nonvolatile Matter (Solids) in Fatty Quaternary Ammonium Chlorides[6]
D 2080 Test Method for Average Molecular Weight of Fatty Quaternary Ammonium Chlorides[6]
D 2081 Test Method for pH of Fatty Quaternary Ammonium Chlorides[6]
D 2082 Test Method for Percent of Non-amines in Fatty Nitrogen Compounds[6]
D 2083 Test Method for Calculation of Percent of Primary, Secondary, and Tertiary Amines in Fatty Amines[6]
D 3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus[5]

3. Terminology

3.1 *Definition*:

3.1.1 *fatty nitrogen products*—nitrogen containing compounds derived from vegetable and animal fatty acids.

3.1.1.1 *Discussion*—Vegetable and animal fatty acids include coconut, cotton, soya, tallow and tall oil fatty acids, fatty acid fractions thereof, and hydrogenated fatty acids.

4. Significance and Use

4.1 These test methods list the methods used to test fatty amines, fatty quaternary ammonium chlorides, fatty amidoamines and fatty diamines. The results of the tests canbe used to determine the purity of these materials and therefore can be used for establishing specifications.

ALL FATTY NITROGEN PRODUCTS

5. Sampling

5.1 The techniques outlined in Test Method D 1466, should be generally suitable with the following additions:

5.1.1 Fatty amines and diamines can sensitize and irritate. Wash from clothing and body surfaces immediately on contact. Protect from air to prevent absorption of carbon dioxide and formation of carbonates. Prolonged storage at elevated temperatures will discolor and degrade.

5.1.2 Fatty quaternary ammonium chlorides usually contain volatile alcohols. Avoid losing these components during sampling and analysis. Prolonged storage at elevated temperatures will discolor and degrade.

6. Color by the Gardner Method

6.1 Gardner color is determined by matching a standard color in an empirical series graduated from 1 (pale yellow) to 18 (dark brown).

6.2 Determine color in accordance with Test Method D 1544, but report the color as 3−, 3, 3+ , 4−, and so on. When the color is Gardner 2+ or less, determine the platinum-cobalt color also.

6.3 The precision of the method in the range of values of 1 to 18 is as follows:

6.3.1 *Repeatability*—Two single determinations performed in one laboratory should be considered suspect if they differ by more than 0.6 unit.

6.3.2 *Reproducibility*—Single determinations performed in two different laboratories should be considered suspect if they differ by more than 1.4 units.

7. Color on the Platinum-Cobalt Scale

7.1 Color on the platinum-cobalt scale (sometimes referred to as platinum-cobalt or Hazen Color) is determined by matching a standard color in an empirical series graduated from 0 (colorless) to 500 (pale yellows).

7.2 Determine color in accordance with Test Method D 1209, but report one of the following values: 0, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 150, 200, 300, 350, 400, 450, 500, DT 500.

7.3 The precision of this test method has not yet been determined for fatty nitrogen products.

8. Water by the Karl Fischer Method

8.1 Water is determined by reacting with an excess of Karl Fischer reagent, which is then back-titrated with water-methanol solution. The usual direct titration with Karl Fischer reagent results in high values for these products.

8.2 Determine the percent of water in accordance with Test Method D 2072.

FATTY AMINES

9. Amine Values

9.1 Total, primary, secondary, and tertiary amine values are defined as the milligrams of potassium hydroxide equivalent to the total, primary, secondary, and tertiary amine basicities in 1 g of sample. These empirical values may be converted to percent total, percent primary, percent secondary, and percent tertiary amines, if the average molecular weights of these components in the sample are known.

9.2 *Procedure*:

9.2.1 Determine the amine values in accordance with Test Methods D 2073.

9.2.2 Alternatively, determine the amine values in accordance with Test Methods D 2074.

10. Iodine Value

10.1 The iodine value is a measure of the unsaturation of the alkyl groups, defined as the percent by weight equivalent of iodine absorbed per gram of sample.

10.2 Determine the iodine value in accordance with Test Method D 2075. This test method is a modified Wijs method using acetic acid as the solvent and mercuric acetate as catalyst.

11. Percent of Non-amine

11.1 The non-amines are fatty amides, nitriles, alcohols and unsaponifiable matter normally present in small amounts in these products.

11.2 Determine the percent non-amine in accordance with Test Method D 2082.

12. Percent of Primary, Secondary, and Tertiary Amines

12.1 The percent of primary, secondary, and tertiary amines present in the sample can be calculated from the primary, secondary, and tertiary amine values and the percent in accordance with Test Method D 2083.

FATTY QUATERNARY AMMONIUM CHLORIDES

13. Acid Value and Amine Value

13.1 *Acid Value* is the number of milligrams of potassium hydroxide needed to neutralize 1 g of sample, and is usually due to amine hydrochloride.

NOTE 1—Most industrial quaternaries contain a small amount of methyl chloride, slow hydrolysis of which causes an increase in acid value in a sample and a corresponding decrease in its amine value.

13.2 *Amine Value* is the number of milligrams of potassium hydroxide equivalent to the fatty amine basicity in 1 g of sample.

13.3 Determine the acid and amine values in accordance with Test Methods D 2076. The test method covers conversion of acid and amine values to percent of amine hydrochloride and percent of amine for standard industrial quaternaries.

14. Ash

14.1 Ash in quaternaries is essentially sodium chloride, unless free caustic material is found as described in Test Method D 2076.

14.2 Determine the percent ash in accordance with Test Method D 2077.

15. Flash Points

15.1 Flash points are run on quaternaries to determine whether they must be classified as flammable under government regulations. The flash point of a liquid is defined as the lowest temperature, corrected to a pressure of 760 mm Hg (101.3 kPa) 1013 m bar of the sample at which application of an ignition source causes the vapor of the specimen to ignite under specified conditions of test.

15.2 Determine flash point of quaternaries having a viscosity less than 45 SUS at 100°F (9.5 cSt at 77°F) (Test Method D 88) by Test Method D 56 and flash point of quaternaries having a viscosity of 45 SUS at 100°F or more by Test Methods D 93. In addition, test suspensions of solids or liquids that tend to skin by Test Methods D 93.

15.3 *Regulatory*:

15.3.1 Determine the flash point by Test Method D 56 or by Test Method D 93 for liquid storage regulations of Occupational Safety and Health Administration of U.S. Dept. of Labor and for classification of hazardous liquids for shipments by all common carriers under the regulations of U.S. Dept. of Transportation.

15.3.2 Determine flash point of liquids regulated by the U.S. Consumer Product Safety Commission by Test Method D 1310.

15.4 Test Method D 3278, which gives comparable results to Test Method D 56 and Test Method D 93 but requires less sample and less time to run than the latter methods, may be used to check flash point of quaternaries. The U.S. Department of Transportation specifies its use as an alternative method to the methods specified in 14.3.1.

16. Iodine Value

16.1 Iodine value is a measure of the unsaturation of alkyl groups.

16.2 Determine the iodine value in accordance with Test Method D 2078. This test method is a modified Wijs method, using chloroform as the solvent and sodium lauryl sulfate to keep the free iodine in the nonaqueous phase.

17. Nonvolatile Matter

17.1 Industrial quaternary products normally contain quaternary, water, alcohol, and minor amounts of salt, amine, and amine hydrochloride. The simplest method of determining the quaternary content of a sample is to determine the percent nonvolatile which for most purposes is equivalent to the percent of quaternary.

17.2 Determine the percent of nonvolatile matter in accordance with Test Method D 2079.

18. Average Molecular Weight

18.1 The average molecular weight of an industrial quaternary varies from lot to lot and from producer to producer because of small variations in the alkyl groups. Where this variation is important the average molecular weight of the quaternary can be calculated by titrating the quaternary with perchloric acid, determining the percent nonvolatile, and correcting both for amine, amine hydrochloride, and salt (percent ash).

18.2 Determine the average molecular weight in accordance with Test Method D 2080.

19. pH

19.1 The pH of quaternaries is dependent upon the fatty amines and fatty amine hydrochlorides present in the material. Most industrial quaternaries exhibit a slow drop in pH with time because of hydrolysis of methyl chloride dissolved in the product.

19.2 Determine the pH in accordance with Test Method D 2081.

FATTY AMIDOAMINES

20. Amine Value

20.1 Amine value is defined as the milligrams of potassium hydroxide equivalent to the amine basicity of 1 g of sample.

20.2 Determine the total amine value in accordance with Test Methods D 2073. The procedures described therein for primary, secondary and tertiary amine values are not applicable to fatty amidoamines.

21. Iodine Value

21.1 The iodine value is a measure of the unsaturation of the alkyl groups, defined as the percent by weight equivalent of iodine absorbed by 1 g of sample.

21.2 Determine the iodine value in accordance with Test Methods D 2075.

FATTY DIAMINES

22. Amine Values

22.1 Total, primary, secondary and tertiary amine values are defined as the milligrams of potassium hydroxide equivalent to the total, primary, secondary and tertiary amine basicities in 1 g of sample.

22.2 Determine the amine values in accordance with Test Methods D 2073. Test Methods D 2074 are not applicable to the fatty diamines.

23. Iodine Value

23.1 Determine the iodine value in accordance with Test Methods D 2074.

24. Percent of Non-amine

24.1 The non-amines are fatty amides, nitriles, alcohols, and unsaponifiable matter normally present in small amounts in these products.

24.2 Determine the percent non-amine in accordance with Test Methods D 2082.

25. Keywords

25.1 fatty amidoamines; fatty amines; fatty diamines