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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Acid Value of Fatty Acids and Polymerized Fatty Acids[1]

This standard is issued under the fixed designation D 1980; 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 the determination of acid value (a measure of the acidity or amount of free fatty acids) and is applicable to all fatty acids and polymerized fatty acids.

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 required to neutralize the fatty acids in 1 g of sample.

4. Significance and Use

4.1 Drying oils are composed primarily of triglycerides of fatty acids, and normally contain low amounts of free fatty acids. However they can be saponified to produce essentially only fatty acids. This test method is used to determine the acidity (acid value) of the fatty acids and is therefore indicative of the amount of free fatty acids in a sample.

4.2 This test method is not to be used as a quality requirement since it measures all acidic components and does not distinguish between fatty acids of different composition.

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 water conforming to Type II of Specification D 1193.

5.3 *Ethyl Alcohol, Neutral* (95 %)—Use 95 % ethyl alcohol or neutral denatured alcohol conforming to Formula No. 30 or No. 3A of the U.S. Bureau of Alcohol, Tobacco, and Firearms. Boil and neutralize to the phenolphthalein end point with alkali just before using.

5.4 *Phenolphthalein Indicator Solution* (10 g/L) (Note 1)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %), methanol, or isopropanol.

NOTE 1—A "masked phenolphthalein indicator" may be used with off-color materials. Prepare by dissolving 1.6 g of phenolphthalein and 2.7 g of methylene blue in 500 mL of alcohol conforming to 5.3. Adjust the pH with sodium hydroxide (NaOH) or potassium hydroxide (KOH) solution so that the greenish-blue color is faintly tinted with purple. The color change is from green to purple when going from acid to alkaline.

5.5 *Potassium Hydroxide or Sodium Hydroxide, Standard Solution* (0.5 *N*): Prepare a stock concentrated solution by dissolving 560 g of potassium hydroxide (KOH) or 425 g of sodium hydroxide (NaOH) in 1 L of water.

5.5.1 Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the carbonate precipitate into another clean bottle. Add clear barium hydroxide ($Ba(OH)_2$) solution until no further precipitate forms. Again allow to settle until clear. Draw off about 875 mL and dilute to 10 L with freshly boiled reagent water. Preserve in a stock bottle provided with a large drying tube filled with soda-lime.

5.5.2 Standardize by titrating against potassium acid phthalate (National Bureau of Standards Acid Potassium Phthalate No. 84), using phenolphthalein as indicator. This solution is approximately 0.5 *N* but instead of adjusting it to a specific value, determine the exact normality and use in the calculations.

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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] *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.

6. Procedure

6.1 Transfer about 5 g of the sample, weighed to 1 mg to a 500-mL Erlenmeyer flask and add 75 to 100 mL of hot, ethyl alcohol. Agitation and further heating may be necessary to bring the fatty acids into complete solution.

6.2 Add 0.5 mL of the phenolphthalein indicator solution and titrate immediately, while shaking, with 0.5 *N* KOH or NaOH solution to the first pink color that persists for 30 s.

7. Calculation

7.1 Calculate the acid value as follows:

$$\text{Acid value} = (VN \times 56.1)/S \quad (1)$$

where:

V = KOH or NaOH solution required for the titration, mL,
N = normality of the KOH or NaOH solution, and
S = specimen weight, g.

8. Precision

8.1 On the basis of an interlaboratory test of this test method in which two operators in four laboratories tested three samples of fatty acids the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results by a single operator should be considered suspect if they differ by more than 2.3 in acid value.

8.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 2.4.

9. Keywords

9.1 acid value; fatty acids