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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Hydroxyl Value of Fatty Oils and Acids[1]

This standard is issued under the fixed designation D 1957; 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.

ε[1] NOTE—Keywords were added editorially in May 1995.

1. Scope

1.1 This test method covers the determination of the hydroxyl content of castor oil, dehydrated castor oil, and their derivatives. This test method may also be used for other fatty products such as fatty alcohols, mono- and diglycerides, and hydroxystearic acid, but the precision will not necessarily be as indicated.

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

3. Significance and Use

3.1 Hydroxyl value is important in establishing reactivity with acids and isocyanates. It is also a measure of the degree of dehydration of castor oil.

3.2 This test method determines the total amount of residual hydroxyl groups present in oils and other fatty acid-containing materials, reported as hydroxyl value.

3.3 This test method involves the acetylation of hydroxyl-containing fatty oils and acids using pyridine as solvent. Other groups that will react with acetic anhydride such as primary and secondary amines under the conditions of the method will be reported as hydroxyl. The hydroxyl value is expressed as milligrams of potassium hydroxide equivalent to the hydroxyl content of 1 g of the oil. A correction is applied for acid groups present.

4. Apparatus

4.1 *Erlenmeyer Flask*, 250-mL, standard ground-glass-stoppered.

4.2 *Condensers*, straight-tube, Liebig type, having standard ground-glass joints.

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 I of Specification D 1193.

5.3 *Acetic Anhydride*, (**Warning**—See 6.5) fresh.

5.4 *n-Butyl Alcohol* (**Warning**—See 6.1), neutralized with 0.5 *N* alcoholic potassium hydroxide (KOH) solution to a faint pink phenolphthalein end point.

5.5 *Ethyl Alcohol or Denatured Alcohol*, conforming to Formula No. 3A or 30 of the U. S. Bureau of Alcohol, Tobacco, and Firearms. Formula No. 3A is a mixture of 100 parts by volume of ethanol to 5 parts by volume of methanol. Formula 30 is 100 parts by volume of ethanol and 10 parts by volume of methanol.

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

5.7 *Potassium Hydroxide, Alcoholic Solution* (0.5 N) (**Warning**—See 6.3)—Prepare and standardize a 0.5 *N* solution of potassium hydroxide (KOH) in ethanol. The strength should be not less than 0.5 *N* in order that the blank titrations will take less than 50 mL to avoid refilling the buret.

5.8 *Pyridine* (**Warning**—See 6.4), distilled at 114 to 115°C.

5.9 *Pyridine-Acetic Anhydride Solution* (3 + 1)—Mix 3 volumes of pyridine with 1 volume of acetic anhydride. Prepare fresh before using.

[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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.