



Standard Test Method for Unsaponifiable Matter in Drying Oils, Fatty Acids, and Polymerized Fatty Acids¹

This standard is issued under the fixed designation D 1965; 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 standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the unsaponifiable matter present in all natural and synthetic drying oils and their fatty acids and any other fatty acids or polymerized fatty acids used in protective coatings.

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 460 Test Methods for Sampling and Chemical Analysis of Soaps and Soap Products²

D 1193 Specification for Reagent Water³

D 1466 Test Method for Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials⁴

2.2 AOSC Standard:

Specification H 2-41⁵

3. Terminology

3.1 Definitions:

3.1.1 *unsaponifiable matter*—substances frequently found dissolved in fatty acids that cannot be saponified by sodium hydroxide or potassium hydroxide under the conditions prescribed but are soluble in the ordinary fat solvents. Included are the higher aliphatic alcohols, sterols, pigments, and hydrocarbons.

4. Significance and Use

4.1 The major components of drying oils and fatty acids can be saponified by reaction with alkali and converted to water-

soluble soaps. This test method determines the amount of material present that is soluble in the oil but is not saponifiable. Under conditions of this test method, this material is not converted to a water-soluble soap and is extracted from the saponification mixture with petroleum ether.

4.2 A small amount of unsaponifiable material is characteristic of drying oils and fatty acids however a large amount indicates contamination with hydrocarbon materials such as mineral oils or hydrocarbon resins.

4.3 This test method is the referee method for unsaponifiable content. The exact amount of unsaponifiable matter obtained is governed by the partition coefficient of the matter between the soap solution and the solvent. Different results will be obtained if some other solvent, such as ethyl ether, is used.

NOTE 1—See relevant specifications for appropriate numeric ranges of unsaponifiable matter in specific drying oils or fatty acids.

5. Apparatus

5.1 *Glass-Stoppered Extraction Cylinder*, graduated at 40, 80, and 130 mL, about 300 mm in height and 35 mm in diameter.

5.2 *Erlenmeyer or Soxhlet Flasks*, 100 to 200-mL capacity.

5.3 *Separatory Funnels*, 500-mL capacity.

5.4 *Glass Siphon*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. 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.⁶ 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming

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² *Annual Book of ASTM Standards*, Vol 15.04.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.03.

⁵ Available from American Oil Chemists' Society, 508 S. Sixth St., Champaign, IL 61820.

⁶ *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 *Anal. 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.