



Standard Test Method for Preparation of Methyl Esters From Oils for Determination of Fatty Acid Composition by Gas-Liquid Chromatography¹

This standard is issued under the fixed designation D 2800; 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 a rapid procedure for conversion of animal and vegetable fatty oils into methyl esters of the fatty acids suitable for analysis by gas-liquid chromatography.

1.2 This test method is believed to be applicable to most drying oils used in the paint industry including linseed, soya, safflower, and cottonseed oils. Unsaturated oils with a tendency to undergo alkaline isomerization or to polymerize in the presence of boron trifluoride (BF_3) may give erroneous results. Unsaponifiables are not removed.

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.* For specific hazard statements, see 5.4 and Note 1.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters³

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁴

D 3457 Test Method for Preparation of Methyl Esters from Fatty Acids for Determination of Fatty Acid Composition by Gas-Liquid Chromatography³

3. Summary of Test Method

3.1 This test method is based upon a rapid saponification of the oil with methanolic sodium hydroxide followed by boiling the soaps with BF_3 -methanol in the same vessel to convert quantitatively the fatty acids to methyl esters. The methyl esters are floated out of the mixture upon addition of a saturated salt solution.

3.2 Methyl margarate may be added quantitatively to the oil prior to saponification and methylation to serve as an internal standard and check on the recovery of monomeric methyl esters. For a discussion on the use of an Internal Standard see Test Method D 3457.

4. Significance and Use

4.1 This test method provides a means by which animal or vegetable fats and oils are converted into their methyl esters so that the fatty acids can then be analyzed by the use of Test Method D 1983.

5. Reagents and Materials

5.1 *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.⁵ 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 IV of Specification D 1193.

5.3 *Boron Trifluoride*, cylinder.⁶

5.4 *Boron Trifluoride Reagent* (125 g/L of Methanol)—Add 1 L of methanol to a 2-L Erlenmeyer flask and weigh on a balance. Place in an ice bath and slowly bubble boron trifluoride (BF_3) gas from a tank through a glass tube until 125 g are taken up. This operation should be performed in a good fume hood, and the gas should not flow so fast that white fumes emerge from the flask. The BF_3 must be flowing through the

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

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *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 The sole source of supply of the cylinder for boron trifluoride known to the committee at this time is Matheson Co., Box 966, Joliet, IL. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible committee,¹ which you may attend.