

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

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

This standard is issued under the fixed designation D 3457; 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 August 1991.

1. Scope

1.1 This test method covers a procedure for conversion of animal and vegetable fatty acids into methyl esters of the fatty acids suitable for analysis by gas-liquid chromatography.

1.2 This test method is applicable to animal and vegetable fatty acids having 8 to 24 carbon atoms.

1.3 This standard does not purport to address all of the safety problems, 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 6.4 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

3. Summary of Test Method

3.1 This test method is based upon esterification of fatty acids with methanol, using boron trifluoride as catalyst. The methyl esters are extracted with ethyl ether and recovered by evaporation of the ether.

3.2 An internal standard may be added if certain constituents are considered likely to be present (see Section 7).

4. Significance and Use

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

² Annual Book of ASTM Standards, Vol 11.01.

5. Apparatus

5.1 Separatory Funnels, 125 and 60-mL with TFE-fluorocarbon stopcocks.

5.2 Powder Funnel, wide stem.

5.3 Erlenmeyer Flasks, 2-L and 25-mL capacity.

5.4 *Glass-Stoppered Storage Bottles* for BF₃-methanol, 500-mL capacity or as required.

5.5 *Platform Balance*, 5-kg capacity; smallest scale divisions required are 0.50 g.

5.6 Analytical Balance, accurate to 1 mg.

5.7 Steam Bath, in fume hood.

5.8 *Ice Bath*, suitable to accommodate a 2-L Erlenmeyer flask.

5.9 *Glass Tubing*, borosilicate glass, 5-mm inside diameter for delivery tube.

6. Reagents and Materials

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.⁴ 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 to Type IV of Specification D 1193.

6.3 Boron Trifluoride, cylinder.⁵

6.4 *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

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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³ Annual Book of ASTM Standards, Vol 06.03.

⁴ 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.

⁵ Available from the Matheson Co., Box 966, Joliet, IL.