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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Preparation of Methyl Esters From Oils for Determination of Fatty Acid Composition by Gas-Liquid Chromatography[1]

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 Water[2]

D 1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters[3]

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints[4]

D 3457 Test Method for Preparation of Methyl Esters from Fatty Acids for Determination of Fatty Acid Composition by Gas-Liquid Chromatography[3]

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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[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 D 01.32 on Drying Oils.

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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.

glass tube before it is placed in and until it is removed from the methanol, or the methanol may be drawn into the gas cylinder valve system and cause an explosion. This reagent has an excellent shelf life and may be used up to four months from preparation. Refrigerate it in a glass-stoppered bottle.

NOTE 1—**Warning:** Handling BF_3 gas is at best quite hazardous. It may be preferable to buy the reagent.

5.5 *Methanol*, anhydrous.

5.6 *Methyl Margarate*—Methyl ester of margaric acid (heptadecanoic acid).

5.7 *Petroleum Ether*, redistilled, boiling point 30 to 60°C.

5.8 *Sodium Chloride, Saturated Solution*—Prepare a saturated solution of sodium chloride (NaCl) in water.

5.9 *Sodium Hydroxide, Methanol Solution* (0.5 *N*)—Prepare a 0.5 *N* solution of sodium hydroxide (NaOH) in methanol.

6. Procedure

6.1 Weigh to 0.1 mg about 300 mg of oil into a 50-mL volumetric flask. Add to this specimen about 50 mg weighed to 0.1 mg of methyl margarate. If an internal standard or a check on the recovery of the methyl esters is not desired, the oil need not be weighed and the methyl margarate may be omitted.

6.2 Add 6 mL of 0.5 *N* methanolic NaOH solution, swirl, and heat the mixture on a steam bath until the oil globules go into solution. This step will take 5 to 10 min.

NOTE 2—With some lipid materials it may take somewhat longer; however, excessive reaction times should be avoided due to the possibility of alkali isomerization.

6.3 Add 8 mL of BF_3-methanol reagent and boil for 2 min. Cool and add 1 mL of petroleum ether to the flask.

6.4 Add enough saturated NaCl solution to float the methyl esters up with the petroleum ether into the narrow neck of the flask.

6.5 Withdraw the methyl ester layer by means of a syringe, and analyze immediately in accordance with Test Method D 1983. The oils may usually be identified from their fatty acid composition in accordance with Method D 2245.

7. Precision and Bias

7.1 Precision and bias were not established at the time this test method was written. An effort is being made to obtain the precision and, if obtainable, it will be published in future revisions. This test method has been in use for many years, and its usefulness has been well established.

8. Keywords

8.1 drying oils; fatty acid; gas-liquid chromatography; methyl esters