



Designation: D8290 – 22

Standard Test Method for Determination of Fatty Acid Methyl Esters (FAME) in Aviation Turbine Fuel using Mid-Infrared Laser Spectroscopy¹

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1. Scope*

1.1 This test method covers the quantification of the fatty acid methyl esters (FAME) content in aviation turbine fuel in the range of 10 mg/kg to 400 mg/kg by measuring infrared (IR) transmission before, during, and after FAME is converted to molecules that absorb in a different spectral region than FAME using a selective chemical reaction facilitated by a suitable catalyst.

NOTE 1—This test method detects all FAME components with peak IR absorbance at approximately 1749 cm^{-1} and C_8 to C_{22} carbon chain length. The accuracy of this test method is based on the molecular weight of C_{16} to C_{18} FAME species. The presence of other FAME species with different molecular weights could affect the accuracy.

NOTE 2—Additives such as antistatic agents, antioxidants, and corrosion inhibitors are measured with the FAME by mid IR absorption. However, these additives do not contribute to the differential absorption spectrum used to quantify FAME, as they do not take part in the selective reaction.

1.2 This test method has interim repeatability precision only, see Section 15 for more information.

1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in Section 8.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.J0.05 on Fuel Cleanliness.

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2. Referenced Documents

2.1 ASTM Standards:²

- D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
- D1655 Specification for Aviation Turbine Fuels
- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
- D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

2.2 CEN Standard:³

- EN 14214 Liquid petroleum products – Fatty acid methyl esters (FAME) for use in diesel engines and heating applications – Requirements and test methods

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *co-hydroprocessed esters and fatty acids, n*—synthetic hydrocarbons derived from the hydroprocessing of bio-derived mono-, di-, and triglycerides, free fatty acids, and fatty acid esters with conventional hydrocarbons in accordance with the requirements of Annex A1.2.2.1 in Specification D1655.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.

*A Summary of Changes section appears at the end of this standard

3.1.3 *fatty acid methyl esters (FAME), n*—a biodiesel composed of long chain fatty acid methyl esters derived from vegetable or animal fats.

3.1.3.1 *Discussion*—Used as a component in diesel fuel and fuel oils, it is a potential source of contamination in aviation turbine fuel because of multi-fuel tankers and pipelines.

3.1.4 *identified incidental materials, n*—chemicals and compositions that have defined upper content limits in an aviation fuel specification but are not approved additives.

3.1.5 *triglycerides, TAG, n*—a naturally occurring ester formed from glycerol and three fatty acid groups, which are the main constituents of natural fats and oils, biodiesel feedstocks, fats and/or oils, that have not been transesterified into biodiesel.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *column, n*—multi-use stainless steel tube containing a suitable catalyst that facilitates the selective conversion of FAME to molecules that absorb in a different spectral region than FAME.

3.2.2 *reactant, n*—specific chemical that is added to the test specimen to convert FAME selectively in the presence of a suitable catalyst to molecules that absorb in a different spectral region away from 1749 cm⁻¹.

4. Summary of Test Method

4.1 A test specimen of aviation turbine fuel is automatically analyzed by mid infrared (IR) laser absorption before, during, and after FAME is converted to molecules that absorb in a different spectral region than FAME. The FAME content is calculated from the differential absorption spectrum from before and after the FAME conversion. This conversion is a chemical reaction between FAME and a reactant facilitated by a suitable catalyst. Test time is typically 23 min. IR absorption of the bulk of the fuel does not contribute to the differential absorption spectrum used to quantify FAME, as bulk components do not take part in the selective reaction or absorb in different spectral regions than FAME.

5. Significance and Use

5.1 The present and growing international governmental requirements to add FAME (biodiesel, as specified in standards such as Specification **D6751** and **EN 14214**) to diesel fuel has had the side effect of leading to potential FAME contamination of jet turbine fuel in multifuel transport facilities such as cargo tankers and pipelines. FAME has been added as an identified incident material to Table 3 of Specification **D1655** in which a permitted level of contamination is specified.

5.2 This test method has been developed for use in the supply chain by nonspecialized personnel to detect all kinds of FAME covering the range of 10 mg/kg to 400 mg/kg.

NOTE 3—This test method can be used to screen for unconverted esters from lipid co-hydroprocessed hydrocarbon synthetic kerosene in aviation turbine fuel. This application is detailed in **X1.2**.

6. Interferences

6.1 Chemicals, which can arise during production, storage, distribution or sampling, containing carbonyl groups, whose

spectral absorbances appear in the IR spectrum close to 1749 cm⁻¹ and react with the reactant, can affect the results of this test method. Plasticizers: dibutyl-sebacate and drilling fluid component: 2-ethyl hexyl acetate are known to increase measurement readings obtained by this test method.

NOTE 4—In a limited study, dibutyl-sebacate at a concentration of 112.6 mg/kg in aviation turbine fuel gave an increased reading of 20.5 mg/kg.

NOTE 5—In a limited study, 2-ethyl hexyl acetate at a concentration of 99.9 mg/kg in aviation turbine fuel gave an increased reading of 67.2 mg/kg.

6.2 *Triglycerides*—It is not possible to distinguish between triglycerides (that is, vegetable oils or animal fats) or FAME by this test method.

NOTE 6—See **X1.1** for further information on the measurement of triglycerides.

7. Apparatus

7.1 An automatically controlled, closely integrated, instrument comprising mid IR laser source, flow-through cell, detector, peristaltic pump, syringe pump, column with column holder, temperature stabilization, low-pressure injector, autosampler (optional), control and interface electronics, test specimen and waste containers, and solenoid valves.⁴

7.1.1 The processing computer may be integrated into the instrument.

7.1.2 This apparatus and the required column are described in more detail in **A1.1**.

7.2 *Inlet Filters*, polytetrafluoroethylene (PTFE), hydrophobic with 3.0 μm pore size and a nominal housing diameter of 30 mm.

7.2.1 Filters containing plasticizers are not suitable as esters can be released.

7.3 *Inlet Tubing*, inert tubing with 1 mm inner diameter.

7.3.1 PTFE tubing containing no plasticizers has been found to be suitable.

7.4 *Density Measuring Device*—According to Test Methods **D1298**, **D4052**, or equivalent national standards to determine the density of the aviation fuel test specimen if required.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on

⁴ The sole source of supply of the apparatus, FameSpec, column QRC, QRR02 Reactant, QRHCS01 Verification Sample, and QRHCF01 Calibration Fluids known to the committee at this time is QuantaRed Technologies GmbH, Columbusgasse 1-3/54, A-1100 Vienna, Austria. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

Analytical Reagents of the American Chemical Society where such specifications are available.⁵

8.2 *Cleaning Solvent*, heptane, reagent grade. (**Warning**—Highly flammable liquid and vapor. May be fatal if swallowed and enters airways. Causes skin irritation. May cause drowsiness or dizziness.)

8.3 *Reactant*⁴. (**Warning**—Flammable liquid and vapor. Causes severe skin burns and eye damage.)

8.4 *Verification Fluids*⁶:

8.4.1 *Verification Sample*⁴, heptane, containing between 45 mg/kg and 55 mg/kg FAME, gravimetrically prepared with a maximum expanded uncertainty of 1 mg/kg. (**Warning**—Highly flammable liquid and vapor. May be fatal if swallowed and enters airways. Causes skin irritation. May cause drowsiness or dizziness.)

8.5 *Calibration Fluids*^{4,6}, a set of ten fluids of heptane containing FAME meeting the requirements in **Annex A2**. (**Warning**—Highly flammable liquid and vapor. May be fatal if swallowed and enters airways. Causes skin irritation. May cause drowsiness or dizziness.)

8.6 *Lint-Free Cloth*, for cleaning and drying the sample inlet tubing.

9. Sampling, Test Specimens, and Test Units

9.1 Unless otherwise specified, take a sample of at least 60 mL in accordance with Practices **D4057** or **D4177** or the requirements of national standards or regulations for the sampling of liquid fuels or both.

9.2 Use new, opaque glass or epoxy lined metal containers with inert closures.

10. Preparation of Apparatus

10.1 Follow the manufacturer's instructions and on-screen instructions for the correct setup and shutdown of the apparatus.

10.2 Ensure that the verification and calibration of the instrument are in accordance with Section **11**.

10.3 Ensure that the column (**A1.1.3**) is installed according to the manufacturer's instructions and its specified use cycle and expiry date are not exceeded.

10.4 Ensure that the column compartment has a temperature between 25 °C and 50 °C.

10.5 Gently swirl the sample for homogeneity before drawing the test specimen.

10.5.1 Determine the density of the test specimen using the density measuring device (**7.4**) if the density is not known.

⁵ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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 following reagents and materials were used to develop the precision statements: Verification Sample and Calibration Fluids for FameSpec, QuantaRed Technologies GmbH, Vienna, Austria. This is not an endorsement or certification by ASTM International.

10.6 Connect inlet filter(s) (**7.2**) and inlet tubing (**7.3**) to the inlet port of the apparatus or the ports of the optional autosampler if in use.

NOTE 7—Inlet filters and inlet tubing are made of chemically inert PTFE and can be re-used. It is recommended to change filters if they show accumulation of jet fuel particulate.

10.7 Run a flushing sequence on the inlet port(s) to be used with air according to the manufacturer's instructions.

11. Calibration and Standardization

11.1 *Calibration*:

11.1.1 Calibrate the instrument according to the manufacturer's instructions and **Annex A2**.

11.2 *Verification*:

11.2.1 Follow the apparatus and test specimen preparation instructions (Section **10**) and check the validity of the verification fluids to be used.

11.2.2 Verify the correct operation of the instrument using the verification fluid (**8.4**) in accordance with the manufacturer's instructions after calibration and at least every six months or immediately after any maintenance on the measurement system. More frequent performance checks shall be carried out according to local quality control requirements.

11.2.3 If the result is not within ± 2.5 mg/kg of the verification fluid's certified value, recheck the validity date of the verification fluid and run a flushing sequence (**10.7**) and repeat the verification.

NOTE 8—The value ± 2.5 mg/kg will be re-evaluated once the reproducibility has been established in an interlaboratory study.

11.2.4 If it is not possible to meet **11.2.3** on two subsequent attempts, the measurement system is out-of-control and cannot be used to measure FAME in aviation turbine fuel until the cause of the out-of-control behavior is identified and corrected.

12. Procedure

12.1 Allow the test specimen to reach a temperature between 10 °C and 30 °C before analysis.

12.2 Commence the test measurement sequence and input the sample density in kilograms per cubic metre and sample identification in accordance with the manufacturer's instructions and the on-screen instructions.

12.3 Wipe dry the inlet tubing (**7.3**) using the lint-free cloth (**8.6**) before commencing a test.

12.4 Insert the unconnected end of the inlet tubing (**7.3**) into the test specimen container that contains at least 50 mL of test specimen.

12.5 Ensure that the outlet tube is connected to an empty waste container.

12.6 Start the test to commence the following automatic sequences as the test specimen is drawn through the instrument by the peristaltic pump (see **Fig. 1**, **Fig. A1.1**, and **Fig. A1.2**).

12.6.1 The tubing, column, and flow-through measurement cell is primed and flushed with about 40 mL of test specimen.

NOTE 9—Flushing with approximately 40 mL corresponds to about ten rinses of the tubing, column, and flow-through measurement cell.