Designation: D8290 – 20

Standard Test Method for Determination of Fatty Acid Methyl Esters (FAME) in Aviation Turbine Fuel using Mid-Infrared Laser Spectroscopy[1]

This standard is issued under the fixed designation D8290; 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.

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.*

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

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

3.1.1.1 *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.

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

[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.

Current edition approved Nov. 1, 2020. Published December 2020. DOI: 10.1520/D8290-20.

[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.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *column, n*—multi-use column through which the test specimen flows 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.

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 3—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 4—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.

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 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.)

[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.

[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.

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.

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 5—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 6—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 Insert the unconnected end of the inlet tubing (7.3) into the test specimen container that contains at least 50 mL of test specimen.

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

12.5 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).

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

NOTE 7—Flushing with approximately 40 mL corresponds to about ten rinses of the tubing, column, and flow-through measurement cell.

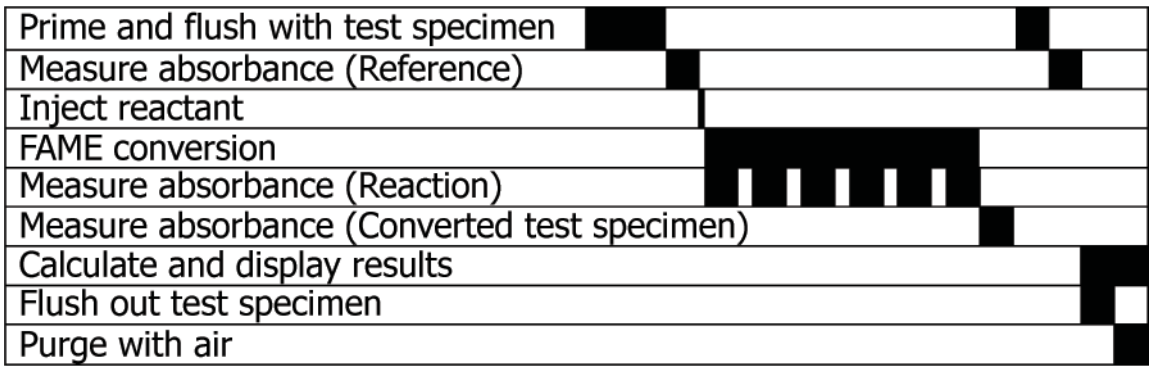

FIG. 1 Test Sequence

12.5.2 The IR transmittance of the test specimen is measured at two wavelengths to obtain a reference value for FAME absorbance.

12.5.3 Approximately 10 µL of reactant (8.3) are injected into the test specimen.

12.5.4 The test specimen containing reactant is circulated through the loop containing the column and flow-through cell via the peristaltic pump for approximately 15 min.

12.5.5 The IR transmittance of the fully converted test specimen is measured at two wavelengths.

12.5.6 The tubing and the flow-through measurement cell is flushed with about 10 mL of fresh, unconverted test specimen.

NOTE 8—Flushing with approximately 10 mL corresponds to about three rinses of the tubing and flow-through measurement cell.

12.5.7 The IR transmittance of the fresh, unconverted test specimen is measured at two wavelengths to obtain a second reference value for FAME absorbance.

NOTE 9—The measurement of a second reference according to 12.5.6 and 12.5.7 helps to minimize the delay between the measurement of converted test specimen and reference, thus increasing of the accuracy of the measurement by minimizing the influence of thermal drift and other time-dependent factors.

12.5.8 The absorbance and the FAME concentration are calculated according to Section 13.

12.5.9 The tubing, column, and flow-through measurement cell are purged with air.

12.5.10 The result is displayed numerically and graphically.

12.6 Record the test result.

13. Calculation or Interpretation of Results

13.1 The absorbance of the test specimen is calculated from IR transmittance data following:

$$Abs = -\log\left(\frac{I_{1744}/I_{1740}}{I^0_{1744}/I^0_{1740}}\right) \quad (1)$$

where:

I_{1744} = measured intensity at 1744 cm^{-1} ± 1 cm^{-1} of the converted test specimen as measured in 12.5.5,

I_{1740} = measured intensity at 1740 cm^{-1} ± 1 cm^{-1} of the converted test specimen as measured in 12.5.5,

I^0_{1744} = measured intensity at 1744 cm^{-1} ± 1 cm^{-1} of the second reference as measured in 12.5.7, and

I^0_{1740} = measured intensity at 1740 cm^{-1} ± 1 cm^{-1} of the second reference as measured in 12.5.7.

NOTE 10—Measurements are not conducted at the FAME peak maximum of 1749 cm^{-1}, but on the peak shoulder.

13.2 FAME concentrations in milligrams per kilogram are calculated from the absorbance from Eq 1 using the calibration curve, the stored value of the calibrant's density, and the test specimen's density following:

$$\text{FAME (mg/kg)} = (Abs \times k + d) \times \frac{\rho_C}{\rho_S} \quad (2)$$

where:

k = slope of the calibration curve,

d = offset of the calibration curve,

ρ_C = density of the calibration material in kg/m^3, and

ρ_S = density of the test specimen in kg/m^3.

14. Report

14.1 The test report should contain at least the following information:

14.1.1 A reference to this test method;

14.1.2 All details necessary for complete identification of the product tested;

14.1.3 The density of the test specimen and a reference to the test method used to measure it;

14.1.4 The results of the test (see Section 13) and report the amount of FAME in the test specimen to the nearest 0.1 mg/kg;

14.1.5 Any deviations, by agreement or otherwise, from the procedures specified; and

14.1.6 The time and date of the test.

15. Precision and Bias[7]

15.1 This test method has interim repeatability precision only. An interlaboratory study of this test method will be conducted and a complete precision statement is expected to be available on or before 2025.

15.2 *Precision*—The precision of this test method was determined by the statistical evaluation of an interim repeatability study which met the requirements of Practice D6300. A single laboratory measured six aviation turbine fuel samples twelve times under repeatability conditions.

NOTE 11—The FAME used in the interim repeatability study was a mixture of Soy- (SME), Palm- (PME), Rape- (RME), Tallow- (TME), and Used Cooking Oil Methyl Ester (UCOME) (20 % each) that were initially blended into conventional diesel fuel at a level of 7 % to produce B7 diesel. This B7 was then blended with the different jet fuels to produce different FAME concentrations.

15.2.1 *Repeatability*—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would exceed the following value with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method:

$$\text{Interim Repeatability} = 0.5913 \times x^{0.2003} \text{ mg/kg} \quad (3)$$

where:

x = average of the two results.

15.2.1.1 See Table 1 for a tabular illustration of precision.

TABLE 1 Interim Repeatability

Level of Results, mg/kg	Interim Repeatability, mg/kg
10	0.9
50	1.3
100	1.5
400	2.0

15.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-2012. Contact ASTM Customer Service at service@astm.org.