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An American National Standard

583/15

# Standard Test Method for Determination of the Fatty Acid Methyl Esters Content of Aviation Turbine Fuel Using Flow Analysis by Fourier Transform Infrared Spectroscopy—Rapid Screening Method<sup>1,2</sup>

This standard is issued under the fixed designation D7797; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method specifies a rapid screening method using flow analysis by Fourier transform infrared (FA-FTIR) spectroscopy with partial least squares (PLS-1) processing for the determination of the fatty acid methyl ester (FAME) content of aviation turbine fuel (AVTUR), in the range of 10 mg/kg to 150 mg/kg.

NOTE 1—Specifications falling within the scope of this test method are: Specification **D1655** and Defence Standard 91-91.

NOTE 2—This test method detects all FAME components, with peak IR absorbance at approximately  $1749\text{ cm}^{-1}$  and  $\text{C}_8$  to  $\text{C}_{22}$  molecules, as specified in standards such as Specification **D6751** and EN 14214. The accuracy of the method is based on the molecular weight of  $\text{C}_{16}$  to  $\text{C}_{18}$  FAME species; the presence of other FAME species with different molecular weights could affect the accuracy.

NOTE 3—Additives such as antistatic agents, antioxidants and corrosion inhibitors are measured with the FAME by the FTIR spectrometer. However the effects of these additives are removed by the flow analysis processing.

NOTE 4—FAME concentrations from 150 mg/kg to 500 mg/kg, and below 10 mg/kg can be measured but the precision could be affected.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

<sup>1</sup> 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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<sup>2</sup> This standard has been developed through the cooperative effort between ASTM International and the Energy Institute, London. The IP and ASTM logos imply that the ASTM and IP standards are technically equivalent, but their use does not imply that both standards are editorially identical.

1.4 *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:<sup>3</sup>

- 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 and Lubricants
- D6751** Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
- E1655** Practices for Infrared Multivariate Quantitative Analysis

### 2.2 CEN Standards:<sup>4</sup>

- EN 14214** Specification Automotive Fuels—Fatty Acid Methyl Esters (FAME) for Diesel Engines—Requirements and Test Methods

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

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

2.3 *Energy Institute Standards*.<sup>5</sup>

**IP 583 Test Method for Determination of the Fatty Acid Methyl Esters Content of Aviation Turbine Fuel Using Flow Analysis by Fourier Transform Infrared Spectroscopy—Rapid Screening Method**

2.4 *Other Standards*.<sup>6</sup>

**Defence Standard 91-91 Issue 7 (DERD 2494) Turbine Fuel, Aviation Kerosine Type, Jet A1**

2.5 *ASTM Adjuncts*.<sup>7</sup>

**ADJD6300 (D2PP) Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products**

### 3. Terminology

3.1 *Definitions*:

3.1.1 *FAME, n*—Fatty acid methyl esters, also known as biodiesel.

3.1.1.1 *Discussion*—Used as a component in automotive diesel fuel and the potential source of contamination in aviation turbine fuel due to multi-fuel tankers and pipelines.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *FA-FTIR, n*—flow analysis by Fourier Transform Infra red technique uses a flow-through measurement cell to make a number of measurements on a stream of test specimen.

3.2.1.1 *Discussion*—The test specimen is analyzed before and after passing through a sorbent that is designed to retard the FAME contamination to be measured. The results are compared to enable the amount of FAME present in the aviation fuel to be determined.

3.2.2 *sorbent cartridge, n*—a cartridge, through which the test specimen flows, containing a specific sorbent

3.2.2.1 *Discussion*—The sorbent cartridge is discarded after each test.

### 4. Summary of Test Method

4.1 A test specimen of aviation turbine (AVTUR) fuel is automatically analyzed, by an FTIR spectrometer, in a 2 mm effective path length flow-through cell, before and after flowing through a cartridge containing a sorbent designed to have a relatively long residence time for FAME. The spectroscopic absorbance differences of the IR spectra, between the measurements, are processed in conjunction with a PLS-1 model to determine the presence and amplitude of the carbonyl peak of FAME at approximately 1749 cm<sup>-1</sup>. Test time is typically 20 min. The flow analysis by FTIR enables the effects of potential interferences to be removed by using their relative retardance times through the sorbent in conjunction with their absorbance at specific wavelengths.

### 5. Significance and Use

5.1 The present and growing international governmental requirements to add fatty acid methyl esters (FAME) to diesel fuel has had the unintended side-effect of leading to potential

FAME contamination of jet turbine fuel in multifuel transport facilities such as cargo tankers and pipelines, and industry wide concerns.

5.2 Analytical methods have been developed with the capability of measuring down to <5 mg/kg levels of FAME, however these are complex, and require specialized personnel and laboratory facilities. This Rapid Screening method has been developed for use in the supply chain by non specialized personnel to cover the range of 10 mg/kg to 150 mg/kg.

### 6. Apparatus

6.1 Automatically controlled, closely integrated, instrument comprising FTIR spectrometer with a 2 mm effective optical path length flow-through cell, computer controlled pump, sorbent cartridge holder, control and interface electronics, test specimen and waste containers, and solenoid valves.

6.2 The processing computer can be integrated into the instrument.

6.3 This apparatus and the required sorbent cartridge are described in more detail in **Annex A1**.

6.4 *Density Measuring Device (optional)*—According to Test Methods **D1298**, or **D4052**, or equivalent national standards, to determine the density of the aviation fuel test sample if required.

### 7. Reagents and Materials

7.1 *Cleaning Solvent*, heptane, reagent grade.

7.2 *Verification Fluids*:<sup>8</sup>

7.2.1 *100 mg/kg*, containing 100 mg/kg ± 10 mg/kg of FAME, with a certified value and uncertainty.

7.2.2 *30 mg/kg*, containing 30 mg/kg ± 5 mg/kg of FAME, with a certified value and uncertainty.

7.3 *Calibration Fluids*:<sup>8</sup>

7.3.1 *Set of Five Fluids*, containing amounts of FAME with certified values and uncertainty.

7.4 *Lint-free Cloth*, for cleaning and drying the sample input tube.

### 8. Sampling

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

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

8.2.1 Used sample containers are permitted provided it can be confirmed they have not been used for unknown fluids or for fluids containing >5 % FAME.

NOTE 5—New sample containers are strongly recommended due to concerns over the difficulty in removing all traces of FAME retained from previous samples.

<sup>5</sup> Available from the Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

<sup>6</sup> Available from Procurement Executive DF5 (air), Ministry of Defence, [www.dstan.mod.uk](http://www.dstan.mod.uk).

<sup>7</sup> ADJD6300 is no longer available from ASTM International Headquarters.

<sup>8</sup> The following reagents and materials were used to develop the precision statements: Seta Verification and Calibration fluids for Seta FIJI, Stanhope-Seta, Chertsey, Surrey, KT16 8AP, UK. This is not an endorsement or certification by ASTM.

8.2.2 Rinse all sample containers with heptane (7.1) or another suitable solvent and drain. Then rinse with the product to be sampled at least three times. Each rinse shall use product with a volume of 10 % to 20 % of the container volume. Each rinse shall include closing and shaking the container for a minimum of 5 s and then draining the product.

**9. Preparation of Apparatus**

9.1 Follow the manufacturer’s instructions and on-screen instructions for the correct set up and shut down of the apparatus.

9.2 Run a flushing sequence using heptane (7.1) in accordance with the manufacturer’s instructions if the last test sample contained FAME in excess of 150 mg/kg.

9.3 Wipe dry the sample input tube with a lint free cloth (7.4) before commencing a test.

9.4 Ensure that the verification and calibration of the instrument are in accordance with Section 10.

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

9.6 Determine the density of the sample using the density measuring device (6.4) if the density is not known.

9.7 Use a new test specimen container, or if there is enough test sample available it is permissible to clean and dry the test specimen container thoroughly before each test using heptane and then partially fill with the test sample, swirl and drain, repeat three times.

NOTE 6—New specimen containers are strongly recommended due to concerns over the difficulty in removing all traces of FAME retained from previous test specimens.

**10. Calibration and Standardization**

*10.1 Verification:*

10.1.1 Follow the apparatus and test specimen preparation instructions (9) and check the validity of the verification fluids to be used.

10.1.2 Verify the correct operation of the instrument using the verification fluid (7.2.1), in accordance with the manufacturer’s instructions, at least every six months. More frequent performance checks shall be carried out according to local quality control requirements.

10.1.3 Verify the correct operation of the instrument using both verification fluids (7.2.1 and 7.2.2) in accordance with the manufacturer’s instructions at least every 12 months or immediately after any maintenance on the measurement system.

10.1.4 If the result is not within  $R/\sqrt{2}$  plus the uncertainty of the verification fluid’s certified value or within the tolerances supplied with the verification fluid, recheck the validity date of the verification fluid and run a flushing sequence (9.2) and repeat the verification.

NOTE 7—In 10.1.4, *R* is the reproducibility of the test method at 100 mg/kg or 30 mg/kg, respectively.

10.1.5 If it is not possible to meet the criteria in 10.1.4 to verify the correct operation of the instrument, follow the manufacturer’s instructions regarding fault finding and calibration.

*10.2 Calibration:*

10.2.1 Calibrate the instrument according to the manufacturer’s instructions when it is not possible to meet the criteria in 10.1.4 to verify the correct operation of the instrument.

10.2.1.1 Calibration uses five (5) calibration standards (7.3) covering the scope of the test method, containing known amounts (mg/kg) of FAME in a known fluid.

**11. Procedure (see Fig. 1)**

11.1 Commence the test measurement sequence (see Section 9), and input the sample density in kilograms per cubic metre (kg/m<sup>3</sup>) and sample identification in accordance with the manufacturer’s instructions and the on-screen instructions.

NOTE 8—If the density of the aviation fuel is not known, a nominal value of 807.5 kg/m<sup>3</sup> is assumed. This could affect the result by a maximum of 4 %.

11.2 Insert a new sorbent cartridge (A1.1.3) and attach a new filter (A1.1.9) to the exit (bottom) of the sorbent cartridge; follow the manufacturer’s instructions to fit the input tube to the cartridge.

11.3 Pour approximately 50 mL of sample into the test specimen container (A1.1.4), that has been prepared as described in 9.7, locate in position and attach the container lid and sample input tube.

11.4 Ensure that an empty waste container, lid and output tube (A1.1.5) are in position.

11.5 Start the test to commence the following automatic sequences as the test specimen is drawn through the instrument by the programmed pump: (see Fig. 1 and Fig. A1.1):

11.5.1 Prime and flush the tubing and the flow-through measurement cell with the test specimen.

11.5.2 Measure the spectrum of the test specimen to check for contamination and to obtain a reference spectrum.



FIG. 1 Test Sequence

11.5.3 Measure the spectra of the output from the sorbent cartridge until a stable value is reached and compares with the reference spectrum.

11.5.4 Re-measure the spectrum of the test specimen to obtain a second reference spectrum.

11.5.5 Analyze and compare the flow analysis spectra (see 11.5.3) with the reference spectrum and determines the FAME peak amplitude using a PLS-1 model (see A1.1.10) over the nominal 1660 cm<sup>-1</sup> to 1800 cm<sup>-1</sup> range.

11.5.6 Calculate the FAME concentration in mg/kg using the calibration curve, the determined peak, the stored value of the calibrant material's density and the sample's density (see 9.6).

11.5.7 Flush the system with the remainder of the test specimen and finally purges with air.

11.5.8 Display the result numerically and graphically (see Fig. A1.1 for a typical example).

11.6 Record the test result and follow the manufacturer's instructions to remove and dispose of the used sorbent cartridge and filter.

## 12. Calculation

### 12.1

$$\text{FAME mg/kg} = (C_m) \times (P_c / P_s) \quad (1)$$

where:

$C_m$  = value directly from the integral calibration curve mg/kg,

$P_s$  = density of the sample in kg/m<sup>3</sup>,

$P_c$  = density of the calibrant material in kg/m<sup>3</sup>.

Report the amount of FAME in the sample to the nearest 0.1 mg/kg.

## 13. Report

13.1 The test report shall contain at least the following information:

13.1.1 A reference to this standard,

13.1.2 All details necessary for complete identification of the product tested,

13.1.3 The result of the test (see Section 12),

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

13.1.5 The time and date of the test.

## 14. Precision and Bias<sup>9</sup>

14.1 *General*—The precision was obtained from a 2013 Energy Institute ILS carried out in Europe using eight

instruments/operators, located in separate laboratories, and a sample set comprising 13 aviation turbine fuel samples in duplicate blended with known amounts of FAME. Samples included hydro-treated fuel, non-hydro-treated fuel, and synthetic fuel and were sourced from the U.S., U.K., and Europe. The precision values given in 12.1 were derived from statistical analysis of these test results.

14.1.1 The precision was obtained by statistical examination of the ILS test results according to Practice D6300 using ADJD6300 D2PP.

14.2 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in 20:

$$r = 4.589 \text{ mg/kg} \quad (2)$$

See Table 1 for a tabular illustration of precision.

14.3 *Reproducibility*—The difference between two test results independently obtained by different operators using different apparatus on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in 20:

$$R = 0.04967(X + 100) \quad (3)$$

where  $X$  is the average of two results being compared, in mg/kg. See Table 1 for a tabular illustration of precision.

14.4 *Bias*—Since there is no accepted reference material for determining the bias for the procedure in this test method, a bias cannot be determined. A sample-specific bias may be present for some samples. The evaluation of 100 separate fuel samples to determine potential fuel effects is reported in "IP583 FAME in Jet Worldwide Test Programme Research Report."<sup>9</sup>

## 15. Keywords

15.1 AVTUR; biodiesel in jet fuel; contamination; FAME; FTIR; FA-FTIR; methyl esters

**TABLE 1 Tabulated Repeatability ( $r$ ) and Reproducibility ( $R$ )**

Level of Result mg/kg	$r$ mg/kg	$R$ mg/kg
10.0	4.6	5.5
20.0	4.6	6.0
30.0	4.6	6.5
40.0	4.6	7.0
50.0	4.6	7.5
60.0	4.6	8.0
70.0	4.6	8.4
80.0	4.6	8.9
90.0	4.6	9.4
100.0	4.6	9.9
150.0	4.6	12.4

<sup>9</sup> Supporting data have been filed at the Energy Institute, UK, www.energyinst.org.uk and may be obtained by requesting the IP 583 Round Robin Research Report or IP 583 FAME in Jet Worldwide Test Programme Research Report.

(Mandatory Information)

A1. APPARATUS DETAILS

A1.1 Apparatus

A1.1.1 See Fig. A1.1 and Fig. A1.2.<sup>10</sup>

A1.1.2 *Fourier Transform Infrared (FTIR) Spectrometer*, meeting the requirements of Table A1.1.

A1.1.2.1 *Flow-through Cell Assembly*, including Calcium Fluoride transparent windows with an effective optical path length of 2.0 mm ± 0.2 mm.

A1.1.3 *Sorbent Cartridge*,<sup>10</sup> see Fig. A1.2, individually packed in a sealed envelope, one per test.

A1.1.4 *Test Specimen Container*, with approximately 50 mL volume marked, with a lid and provision for holding the input tube at approximately 5 mm above the bottom of the container.

A1.1.5 *Waste Container*, 50 mL with a lid and provision for the waste outlet tube.

<sup>10</sup> The sole source of supply of the apparatus, Seta FIJI SA5000 and cartridge (including filter) SA5005, known to the committee at this time is Stanhope-Seta, Chertsey, Surrey, KT16 8AP, UK. 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,<sup>1</sup> which you may attend.

A1.1.6 *Solenoid Valves*, computer controlled, small dead volume and orifice size.

A1.1.7 *Connecting Tubing*, narrow bore nylon or other suitable material resistant to aviation turbine fuel and FAME.

NOTE A1.1—Tubing containing plasticizers is not suitable as esters can be released.

A1.1.8 *Pump*, computer controlled, flow rate nominally 5 mL/min.

A1.1.9 *Filter*,<sup>10</sup> 5 micron nylon filter 30 mm nominal diameter, fitted to the exit of the sorbent cartridge, with luer fittings, one per test.

A1.1.10 *PLS-1 Model*—This “frozen” model is built into the apparatus software, is unaffected by calibration and is not user adjustable. It has been derived in accordance with the mathematics outlined in Practices E1655. It uses five loading vectors and is based on over 900 data points from measurements of AVTUR (Mercox and hydrotreated) from over 20 locations at refineries, airports and storage facilities.

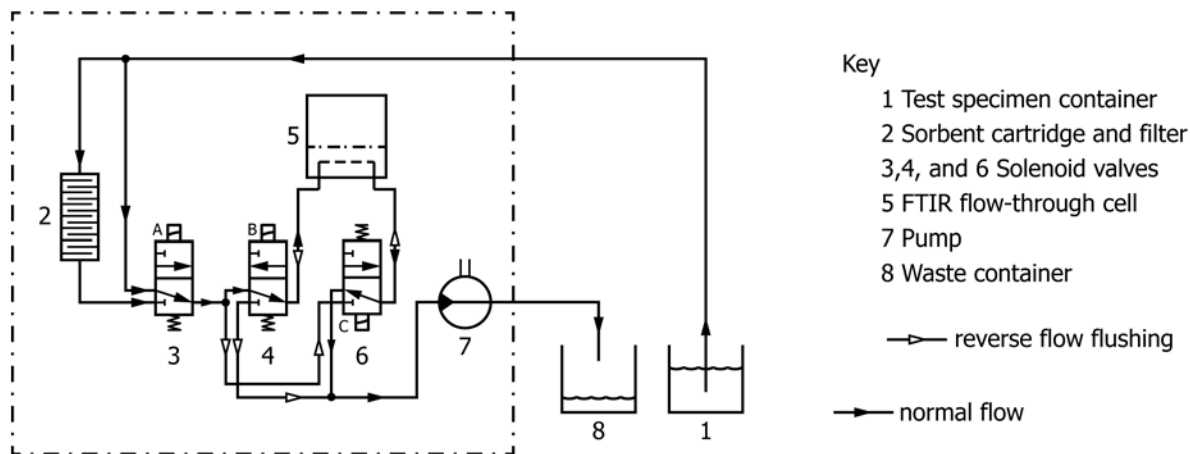


FIG. A1.1 Apparatus Schematic



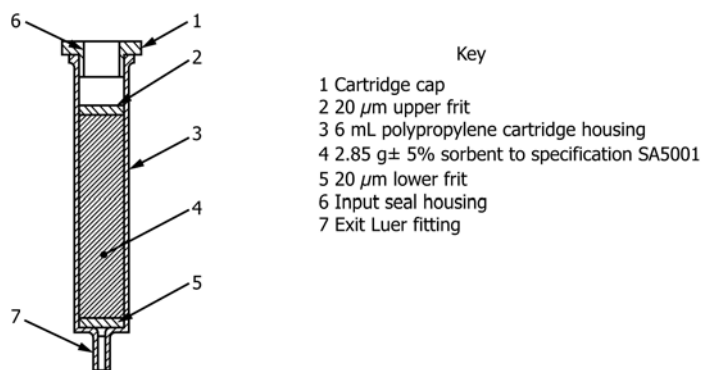


FIG. A1.2 Sorbent Cartridge

TABLE A1.1 Specification of the FTIR Spectrometer

Spectral Range (at least)	4000 $\text{cm}^{-1}$ to 650 $\text{cm}^{-1}$
Wave-number precision	0.1 $\text{cm}^{-1}$
Resolution	<4 $\text{cm}^{-1}$
Cell path length (optical)	2.0 mm
Flow-through sample cell	Single beam reflective
Sealed	Yes

## SUMMARY OF CHANGES

Subcommittee D02.J0 has identified the location of selected changes to this standard since the last issue (D7797 – 16a) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Added yeardate to IP designation.

Subcommittee D02.J0 has identified the location of selected changes to this standard since the last issue (D7797 – 16) that may impact the use of this standard. (Approved Oct. 1, 2016.)

(1) Revised subsection 14.4.

Subcommittee D02.J0 has identified the location of selected changes to this standard since the last issue (D7797 – 12) that may impact the use of this standard. (Approved May 15, 2016.)

(1) Revised subsections 1.1 and 5.2, Note 4, and Section 14. (2) Revised Table 1.

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