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An American National Standard

583

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[1,2]

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 (ε) 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 cm^{-1} and C_8 to 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 C_{16} to 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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:*[4]

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

[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] 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.

[3] 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.

[4] 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:*[5]
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:*[6]
Defence Standard 91-91 Issue 7 (DERD 2494) Turbine Fuel, Aviation Kerosine Type, Jet A1
2.5 *ASTM Adjuncts:*[7]
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.

[5] Available from the Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.
[6] Available from Procurement Executive DF5 (air), Ministry of Defence, www.dstan.mod.uk.
[7] ADJD6300 is no longer available from ASTM International Headquarters.

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^{-1}. 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*[8]:

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*[8]:

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.

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.

[8] 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.