Designation: D7963 – 22

Standard Test Method for Determination of Contamination Level of Fatty Acid Methyl Esters in Middle Distillate and Residual Fuels Using Flow Analysis by Fourier Transform Infrared Spectroscopy—Rapid Screening Method[1]

This standard is issued under the fixed designation D7963; 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) processing for the quantitative determination of the fatty acid methyl ester (FAME) contamination of middle distillates, in the range of 20 mg⁄kg to 1000 mg⁄kg, and of middle distillates and residual fuels, following dilution, for levels above 0.1 %.

NOTE 1—Annex A2 describes a dilution procedure to significantly expand the measurement range above 1000 mg/kg for distillates and to enable measurement of residual oils

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 D6751 and EN 14214. The accuracy of the test method is based on the molecular mass of C_{16} to C_{18} FAME species; the presence of other FAME species with different molecular masses 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 any potential interference effects of these additives are eliminated by the flow analysis processing.

NOTE 4—The scope of this test method does not include aviation turbine fuel which is addressed by Test Method D7797.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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:*[2]

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

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

D7797 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

E1655 Practices for Infrared Multivariate Quantitative Analysis

2.2 *CEN Standard:*[3]

EN 14214 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 D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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[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 the American National Standards Institute (ANSI) 25W 43rd St, 4th Floor. New York, NY 10036.

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

2.3 *Energy Institute Standards:*[4]
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:*
MIL-DTL-16884 Specification for Fuel, Naval Distillate[5]

3. Terminology

3.1 *Definitions:*
3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.
3.1.2 *FAME, n*—fatty acid methyl esters, also known as biodiesel.

3.1.2.1 *Discussion*—Used as a component in automotive diesel fuel and the potential source of contamination in fuels 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 Infrared 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 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 The test specimen 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.

4.2 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}.

4.3 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.

4.4 Test time is typically less than 20 min.

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 fuels in multi-fuel transport facilities such as cargo tankers and pipelines, and industry wide concerns. This has led to a requirement to measure contamination levels in diesel and other fuels to assist custody transfer issues.

5.2 Analytical methods have been developed with the capability of measuring down to <5 mg/kg levels of FAME in aviation turbine fuel (AVTUR), 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 20 mg⁄kg to 500 000 mg⁄kg (0.002 % to 50 %).

5.3 A similar procedure, Test Method D7797, is available for AVTUR in the range 10 mg/kg to 150 mg⁄kg. Test Method D7797 uses the same apparatus, with a specific model developed for AVTUR.

6. Apparatus

6.1 Automatically controlled, closely integrated instrument comprising a 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 test specimen if required.

7. Reagents and Materials

7.1 *Cleaning Solvent*—heptane, reagent grade.

7.2 *Verification Fluids:*[6]
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.
7.2.3 *400 mg/kg*—containing 400 mg/kg ± 40 mg/kg of FAME, with a certified value and uncertainty.
7.2.4 *900 mg/kg*—containing 900 mg/kg ± 90 mg/kg of FAME, with a certified value and uncertainty.[6]

7.3 *Calibration Fluids:*[6]
7.3.1 *A Set of Nine Fluids*—containing amounts of FAME with certified values and uncertainty.

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

[4] Available from the Energy Institute, 61 New Cavendish Street, London, W1G7AR, U.K. www.energyinst.org.uk.
[5] Copies of these documents are available online at the ASSIST Quick Search website https://quicksearch.dla.mil or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.
[6] 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. The sole source of supply of Seta Verification and Calibration fluids for Seta FIJI 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,[1] which you may attend.