Designation: D7963 – 14

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]

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—Precision for middle distillate and residual fuel is provided in preliminary form at this time, details are given in Appendix X1.

NOTE 5—The scope of this test method does not include aviation turbine fuel which is addressed by Test Method D7797.

1.2 All stated values are in SI units.

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:*[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

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

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

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[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 and Cleanliness 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.

[4] Available from the Energy Institute, 61 New Cavendish Street, London, W1G7AR, U.K. www.energyinst.org.uk.

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 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 200 000 mg/kg (0.002 % to 20 %).

5.3 A similar procedure, Test Method D7797, is available for AVTUR in the range 20 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:*[5]

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.[5]

7.3 *Calibration Fluids:*[5]

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.

7.5 *Diluent*—for diluting the test sample when the FAME content is >1000 mg/kg (See Annex A2).

7.6 *Sorbent Cartridge*[6]—see Fig. A1.2, individually packed in a sealed envelope, one per test.

8. Sampling

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

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

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[5] The following reagents and materials were used to develop the preliminary 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.

[6] The sole source of supply of the apparatus known to the committee at this time is Seta FIJI and cartridge (including filter) available from 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.

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.

8.2.1.1 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. Apparatus and Sample Preparation

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 1000 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 middle distillate sample for homogeneity before drawing the test specimen. For residual fuels, warm the sample up to 50 °C and agitate the sample to provide mixing, such as orbiting the container horizontally.

9.6 If the expected concentration of FAME is >1000 mg/kg or the sample is residual fuel, follow Annex A2 to dilute the sample.

9.7 Determine the density of the middle distillate sample using the density measuring device (6.4) if the density is not known. For samples previously diluted according to Annex A2, determine the density of the diluted sample using the density measuring device (6.4) if the density is not known.

9.8 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 (see Section 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 all verification fluids (7.2.1 – 7.2.4) 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.

NOTE 7—In 10.1.4, R is the reproducibility of the test method at the certified FAME concentration.

10.1.5 Follow the manufacturer's instructions regarding fault finding and calibration if it is not possible meet the criteria in 10.1.4 to verify the correct operation of the instrument.

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 to verify the correct operation of the instrument.

10.2.1.1 Calibration uses nine 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), input the test specimen density in kilograms per metre cubed and sample identification in accordance with the manufacturer's instructions and the on-screen instructions.

11.2 Insert a new sorbent cartridge (A1.1.2) and attach a new filter (A1.1.8) 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, which has been prepared as described in Section 9, into the test specimen container (A1.1.3), locate in position, and attach the container lid and sample input tube.

11.4 Ensure that an empty waste container, lid, and output tube (A1.1.4) 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):

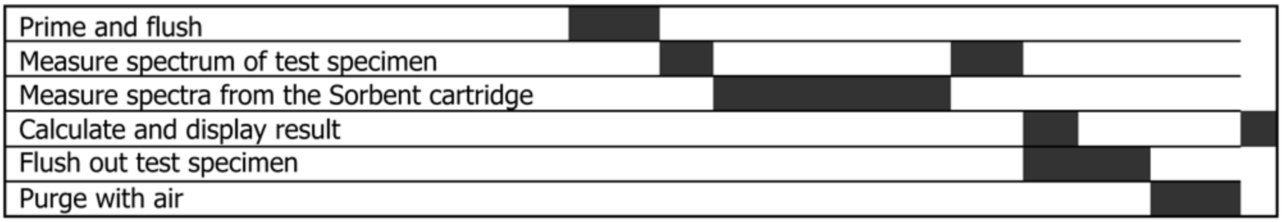


FIG. 1 Test Sequence

11.5.1 Primes and flushes the tubing and the flow-through measurement cell with the test specimen,

11.5.2 Measures the spectrum of the test specimen to check for contamination and to obtain a reference spectrum,

11.5.3 Measures 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-measures the spectrum of the test specimen to obtain a second reference spectrum,

11.5.5 Analyzes and compares the flow analysis spectra (see 11.5.3) with the reference spectrum and determines the FAME peak amplitude using a PLS-1 model (see A1.1.9) over the nominal 1660 cm^{-1} to 1800 cm^{-1} range,

11.5.6 Calculates the FAME concentration in mg/kg using the apparatus calibration, the determined peak, the stored value of the calibrant material's density, and the sample's density (see 9.7).

11.5.7 Flushes the system with the remainder of the test specimen and finally purges with air, and

11.5.8 Displays the result numerically and graphically.

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 For samples measured directly, report the amount of FAME in the sample to the nearest 0.01 mg/kg. For samples prepared following Annex A2, report the amount of FAME (percent by mass) in the sample to the nearest 0.01 %.

12.2 For samples measured directly, calculate the FAME concentration as follows:

$$\text{FAME mg/kg} = (C_m) \times \left(\frac{P_c}{P_s}\right) \tag{1}$$

where:

C_m = value directly from the integral apparatus calibration in mg/kg,
P_s = density of the sample in kg/m^3, and
P_c = density of the calibrant material in kg/m^3,

12.3 For samples prepared following Annex A2, calculate the FAME concentration as follows:

$$\text{FAME percent} = (\text{FAME mg/kg}) \times \frac{DF}{10\ 000} \tag{2}$$

where:

FAME percent = FAME, by mass percent,
FAME mg/kg = value obtained from Eq 1 for the diluted sample, and
DF = dilution factor obtained from Eq A2.1 in Annex A2.

13. Report

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

13.1.1 A reference to this test method,

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

14.1 The preliminary precision was obtained from a mini-study held at a single location. Details are given in Appendix X1. A full ILS and precision statement will be determined within 5 years.

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:

$$r = \text{to be determined} \tag{3}$$

where:

X = the average of two results being compared, in miligrams per kilogram.

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:

$$R = \text{to be determined} \tag{4}$$

where:

X = the average of two results being compared, in milligrams per kilogram.

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.

15. Keywords

15.1 biodiesel contamination; FAME; FA-FTIR; FTIR; methyl esters

ANNEXES

(Mandatory Information)

A1. APPARATUS DETAILS

A1.1 Apparatus[5] (see Figs. A1.1 and A1.2)

A1.1.1 *Fourier Transform Infrared (FTIR) Spectrometer,* meeting the requirements of Table A1.1.

TABLE A1.1 Specification of the FTIR Spectrometer

Spectral range (at least)	4000 cm^{-1} to 650 cm^{-1}
Wave-number precision	0.1 cm^{-1}
Resolution	>4 cm^{-1}
Cell path length (optical)	2.0 mm
Through flow sample cell	Single beam reflective
Sealed	Yes

A1.1.1.1 *Flow-through Cell Assembly,* including a calcium fluoride transparent window, with an effective optical path length of 2.0 mm ± 0.2 mm.

A1.1.2 *Sorbent Cartridge,*[6] see Fig. A1.2, individually packed in a sealed envelope, one per test.

A1.1.3 *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.

A1.1.4 *Waste Container,* 50 mL with a lid and provision for the waste outlet tube.

A1.1.5 *Solenoid Valves,* computer controlled, small dead volume and orifice size.

A1.1.6 *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.

A1.1.7 *Pump,* computer controlled, flow rate nominally 5 mL/min.

A1.1.8 *Filter,*[6] 5-µm nylon filter 30 mm nominal diameter, fitted to the exit of the sorbent cartridge, with luer fittings, one per test.

A1.1.9 *PLS-1 Model,* the model is built into the apparatus software, is unaffected by calibration and is not user adjustable. The model meets the requirements of Practices E1655.

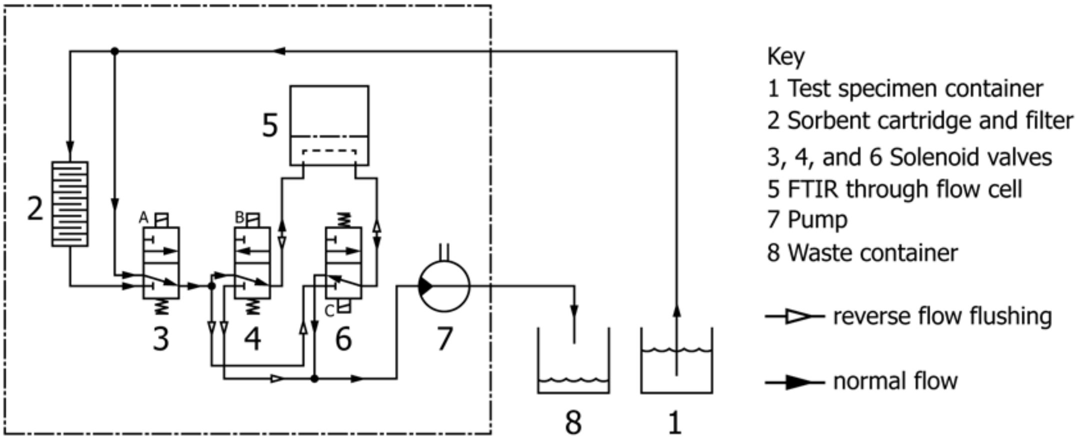
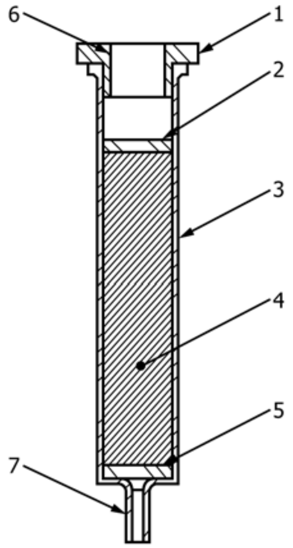


FIG. A1.1 Apparatus Schematic



Key
1 Cartridge cap
2 20 µm upper frit
3 6 mL polypropylene cartridge housing
4 2.85 g ± 5 % sorbent to specification SA5001
5 20 µm lower frit
6 Input seal housing
7 Exit Luer fitting

FIG. A1.2 Sorbent Cartridge

A2. DILUTION OF SAMPLES

A2.1 General

A2.1.1 This procedure is required when the test sample is expected to have a FAME content >1000 mg/kg or when the sample is a residual fuel.

A2.2 Reagents and Materials

A2.2.1 *Diluent*[7]—For diluting the test sample when the FAME content is >1000 mg/kg.

A2.2.1.1 *Discussion*—The diluent used may influence the test result if it contains compounds with spectral absorbances close to the FAME peak at 1749 cm^{-1}. A result of <10 mg/kg by IP 583 shall be obtained.

A2.2.1.2 *Typical Properties*—FAME-free, transparent light distillate with typical properties as follows:

(1) IBP and distillation range: 192 °C to 256 °C, 101.3 kPa (Test Method D86),

(2) Density 0.8 g/cm^3 (Test Method D4052),

(3) Kinematic viscosity in the range 1 mm^2/s to 2.5 mm^2/s at 40 °C (Test Method D445), and

(4) FAME content <10 mg/kg (Test Method IP 583).

A2.3 Apparatus

A2.3.1 *Sample Container,* 100 mL to 500 mL, glass with a screw cap.

NOTE A2.1—Graduations at 50 mL and 100 mL are recommended.

A2.3.2 *Analytical Balance,* capable of weighing to the nearest 0.001 g.

A2.3.3 *Pipette,* positive displacement, capable of dispensing approximately 0.1 mL.

A2.3.4 *Syringe,* plastic, 5 mL or 10 mL, lubricant-free, rubber-free.

A2.4 Procedure

A2.4.1 Refer to Table A2.1 or Table A2.2 for diluent and sample volumes.

A2.4.2 Refer to Section 9 for sample preparation.

A2.4.3 Place a clean sample container (without the cap) on the balance (A2.3.2), tare the reading, and add the required volume of diluent (A2.2.1). Note the resulting mass of the diluent (Md) in grams.

A2.4.4 Tare the balance, add the required amount of fuel using a clean pipette or syringe. Note the resulting mass of the added fuel (Mf) in grams.

A2.4.5 Fit the cap to the container. For middle distillates invert five times to mix the fuel and diluent. For residual oils, agitate for 15 s to 20 s to ensure thorough mixing.

TABLE A2.1 Dilution Requirements—Middle Distillates

FAME contamination (% by mass)	FAME contamination (mg/kg)	Fuel to be added (mL)	Diluent to be added (mL)	Expected result from apparatus (mg/kg)
5 to 20	50 000 to 200 000	0.1	100	50 to 200
0.5 to 5	5 000 to 50 000	1	100	100 to 1 000
0.1 to 0.5	1 000 to 5 000	5	100	50 to 250
0.001 to 0.1	10 to 1 000	No dilution required	Not applicable	10 to 1 000

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[7] The sole source of supply of the diluent known to the committee at this time is SA5028-0 available from 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.

TABLE A2.2 Dilution Requirements—Residual Oils

FAME contamination (% by mass)	FAME contamination (mg/kg)	Fuel to be added (mL)	Diluent to be added (mL)	Expected result from apparatus (mg/kg)
0.1 to 10	1 000 to 100 000	1	100	10 to 1 000

A2.4.6 Calculate the dilution factor according to Eq A2.1:

$$\text{dilution factor} = \frac{(Md + Mf)}{Mf} \qquad (A2.1)$$

where:

Md = mass of diluent in grams,
Mf = mass of fuel in grams, and
dilution factor = total mass of solution divided by mass of fuel.

A2.4.7 Determine the density of the diluted sample using the density measuring device (6.4) if the density is not known and follow the procedure in Section 11.

A2.4.8 Calculate the FAME content using Eq 2 in Section 12.

APPENDIX

(Nonmandatory Information)

X1. DETAILS OF REPEATABILITY STUDY AND PRELIMINARY PRECISION ESTIMATE

X1.1 This temporary precision is based on a mini-study[8] comprising 3 analyzers/3 operators across 13 samples, containing a known concentration of FAME in the range 0 % to 20 %, at a single location. Precision estimates based on this limited data set were calculated using the D2PP computer program, and are shown in Table X1.1 and Table X1.2. A full precision exercise will be held within five years.

X1.1.1 Since this study is conducted at one location, the resulting *R* will likely under-estimate *R* in commercial usage of this method at different locations. Hence, the *R* obtained from this study is multiplied by a factor of 1.5 to account for the additional variability anticipated due to different locations.

$$r = 0.2933 \times (\text{FAME})^{0.8302} \qquad (X1.1)$$

$$R = 1.5 \times \left(0.3508 \times (\text{FAME})^{0.8302}\right) \qquad (X1.2)$$

where:

r = repeatability in mg/kg,
R = reproducibility in mg/kg, and
FAME = value obtained from Eq 1 for the undiluted sample in mg/kg.

$$r = 0.06139 \times (\text{FAME percent})^{0.8302} \qquad (X1.3)$$

$$R = 1.5 \times \left(0.07342 \times (\text{FAME percent})^{0.8302}\right) \qquad (X1.4)$$

where:

r = repeatability in percent by mass,
R = reproducibility in percent by mass, and
FAME percent = value obtained from Eq 2 for the diluted sample, percent by mass.

TABLE X1.1 Precision Estimate for Trace FAME Contamination (mg/kg)

NOTE 1—See Eq X1.1 and Eq X1.2.

FAME Contamination (mg/kg)	Calculated Precision (mg/kg)	
	Repeatability (*r*)	Reproducibility (*R*)
25	4.2	7.62
100	13.4	24.07
500	51.0	91.59
1000	90.8	162.84

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[8] A research report titled *Determination of the Level of Trace FAME Contamination in Distillate and Residual Fuels* is available from Stanhope Seta, Chertsey, Surrey, KT16 8AP, UK

TABLE X1.2 Precision Estimate for FAME Contamination of Residual and Diesel Fuel (Mass Percent)

NOTE 1—See Eq X1.3 and Eq X1.4.

FAME Contamination (percent by mass)	Calculated Precision (FAME Percent by Mass) Repeatability	Reproducibility
0.1	0.009	0.02
0.5	0.035	0.06
1	0.061	0.11
5	0.23	0.42
10	0.42	0.74
20	0.74	1.32

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