Designation: ~~D8274 – 20~~ D8274 – 20a

Standard Test Method for Determination of Biodiesel (Fatty Acid Methyl Esters) Content in Diesel Fuel Oil by Portable Rapid Mid-Infrared Analyzer[1]

This standard is issued under the fixed designation D8274; 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 determination of the content of biodiesel (fatty acid methyl esters (FAME)) in diesel fuel oils for volume fractions of 0.1 % to 31.0 % by mid-infrared analyzer with a resolution of 0.1 %.

NOTE 1—ASTM and ISO specification fuels falling within the scope of this test method include Specifications: D975 grades No. 1D and No. 2D, D7467, distillate grades of D396, MIL-DTL-16884, and distillate grades of marine fuel specification ISO 8217.

1.2 The accuracy of this test method is based on the molecular weight of C16 and C18 FAME species.

1.2.1 *Discussion*—Biodiesel contains a variety of species with different molecular weights. Typical market FAMEs from North America and Europe, which are predominantly soy, rapeseed, and used cooking oil derived FAME were included in the pilot study. FAME derived from coconut, which predominantly contains C12, will over-read by approximately 30 %.

1.3 ~~It is not possible to~~ This method cannot distinguish between vegetable oils, animal fats, FAEE, compounds containing carbonyl groups, and FAME. For more information, see Section 6.

1.4 This test method has interim repeatability precision only, see Section 14 for more information.

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

1.6 *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.*

1.7 *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]

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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 D02.04.0F on Absorption Spectroscopic Methods.

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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.

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

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
D396 Specification for Fuel Oils
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D975 Specification for Diesel Fuel
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
D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
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
D7371 Test Method for Determination of Biodiesel (Fatty Acid Methyl Esters) Content in Diesel Fuel Oil Using Mid Infrared Spectroscopy (FTIR-ATR-PLS Method)
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)
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
D7806 Test Method for Determination of Biodiesel (Fatty Acid Methyl Ester) and Triglyceride Content in Diesel Fuel Oil Using Mid-Infrared Spectroscopy (FTIR Transmission Method)
D7963 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

2.2 *ISO Standard:*[3]

ISO 8217 Petroleum products—Fuels (class F)—Specifications of marine fuels

2.3 *Other Standard:*[4]

MIL-DTL-16884 Specification for Naval distillate fuel (NATO designation F76)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Abbreviations:*

3.2.1 *FAME, n*—fatty acid methyl esters

4. Summary of Test Method

4.1 This test method describes a rapid procedure to determine the volume concentration of biodiesel in diesel. The test specimen is introduced into a liquid cell of a mid-infrared analyzer. The absorbance of the sample is measured in two spectroscopic bands. The amount of biodiesel is determined by the absorbance in a band which includes 1749 cm^{-1}, where the carbonyl stretching frequency is located. The second band, centered around 1890 cm^{-1} is used to correct for the non-biodiesel matrix of the test specimen. The test takes less than one minute, including introducing the test specimen and removing the previous test specimen.

5. Significance and Use

5.1 Biodiesel is a fuel commodity primarily used as a blending component with diesel fuel. It is important to check the concentration of biodiesel in the diesel fuel in order to verify it is within limits or does not exceed the maximum allowable limit.

5.2 This test method is applicable for quality control in the production and distribution of diesel fuel and biodiesel blends.

5.3 This test is simple to run, completed in less than one minute, with no dilution of the test sample, no cleaning solvents are required, and the analyzer is portable and self-contained.

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

[4] Copies of these documents are available online at https://assist.dla.mil/quicksearch/ or http://assistdocs.com/ or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.

6. Interferences

6.1 *Undissolved Water and Particulates*—Samples containing undissolved water or particulates can result in erroneous results. If the sample is cloudy or water saturated after it has been equilibrated between 10 °C to 30 °C, filter the sample through a qualitative filter paper until clear prior to its measurement.

6.2 *Vegetable Oils*—~~It is not possible to~~ This method cannot distinguish between vegetable oils, animal fats, and ~~FAME by this test method.~~ FAME. Measurements of vegetable oils and animal fats will give a similar response as that of FAME.

6.3 *Fatty Acid Ethyl Esters (FAEE)*—It is not possible to distinguish between FAEE and FAME by this test method. Measurements of FAEE and animal fats will give a similar response as that of FAME.

6.4 *Carbonyl Groups*—This method cannot distinguish FAME from compounds containing carbonyl groups, such as aldehydes and ketones. The results for samples containing volume fractions of these compounds less than 0.1 % would not be affected.

7. Apparatus[5]

7.1 *Mid-Infrared Analyzer,* comprising of a liquid cell, infrared emitter, detectors, a display, microprocessor, and a sample loading mechanism.

7.2 The apparatus is further described in Annex A1.

8. Reagents and Materials

8.1 *Absorbent paper towel.*

8.2 *Verification sample,* containing a known concentration of FAME between 3 % and 7 % (volume fraction). Refer to Annex A2 on the preparation of verification samples.

8.3 *Calibration standards,* containing known concentrations of FAME between 0 % and 40 %. Refer to Annex A2 on the preparation of calibration samples.

9. Sampling

9.1 *General Requirements:*

9.1.1 Obtain samples using procedures outlined in Practice D4057 or D4177. Do not use “sampling by water displacement.” FAME is more water-soluble than the hydrocarbon base in a biodiesel blend.

9.1.2 Protect samples from excessive temperatures prior to testing.

9.1.3 Avoid using plastic materials for sampling, and do not use rubber caps or plastic bottles for storage of the sample.

10. Preparation of Apparatus

10.1 Switch on the analyzer.

10.2 Place the inlet tube over a waste container, and empty the analyzer by depressing the fill button.

NOTE 2—During this step, any remaining test specimen is ejected into the waste container.

[5] The sole source of supply of the apparatus, SetaCheck Bio (part number SA5500), 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.

10.3 Allow the fill button to come up slowly. Repeat 10.2 and 10.3 a minimum of three times.

10.4 Wipe the inlet tube with absorbent paper towel (8.1).

11. Calibration and Standardization

11.1 Follow the manufacturer's instructions to calibrate the analyzer.

11.2 The analyzer is calibrated using calibration standards blended as described in Annex A2.

11.3 Verify the correct operation of the analyzer by testing a verification sample (8.2) at least once per year, and following recalibration or repair. If a result more than 0.5 % (volume fraction) from the expected is obtained, then follow the manufacturer's instructions to recalibrate the analyzer.

11.4 Confirm the in-statistical-control status of the test method each day it is used by measuring the biodiesel concentration of at least one quality control sample that is similar in composition and matrix to samples routinely analyzed. For details on quality control sample selection, preparation, testing, and control charting, refer to Practice D6299. Follow the manufacturer's instructions if the analyzer is out of statistical control.

12. Procedure

12.1 Allow the samples and analyzer to reach a temperature between 18 °C and 24 °C before analysis.

NOTE 3—The FAME concentration of the sample can be measured with the analyzer and sample in the temperature range 5 °C to 40 °C, but the precision has not been determined.

12.2 Place the inlet tube over a waste container, and fully depress the fill button to empty the analyzer of any remaining test specimen.

12.3 Allow the fill button to return slowly to its initial position. Repeat 12.2 and 12.3 a minimum of three times.

12.4 Fully depress the fill button.

12.5 Keep the fill button depressed while wiping the outside of the inlet tube with an absorbent paper towel (8.1).

12.6 Insert the inlet tube into the test specimen.

12.7 Allow the fill button to return slowly to the initial position, and ensure that the inlet tube remains submerged in the sample. Visually inspect the inlet tube, and if air bubbles are present, then abort the test.

NOTE 4—If the inlet tube is removed from the sample while the syringe is still taking sample in, then air ingress will result and the measurement could be affected.

12.8 Once the fill button has returned to its initial position, remove the inlet tube from the sample and press the start measurement button.

12.9 The following steps occur automatically:

12.9.1 The emitter is switched on.

12.9.2 The absorbance of the sample is measured in two spectroscopic bands (see A1.1.5).

12.9.3 The microprocessor calculates the result, which is displayed within 0.1 %.

12.10 Record the biodiesel concentration, as the volume fraction, on the digital display to 0.1 %.

12.11 Repeat 12.2 and 12.3 to remove the sample from the analyzer.

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[6]

14.1 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 2024.

14.2 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 fourteen samples twelve times under repeatability conditions. The samples which included diesel meeting Specifications D975 and EN 590 contained biodiesel in the range 0.1 % to 31 % (volume fraction) from North American and European sources. A pilot program, also meeting the requirements of Practice D6300, included three operators, three analyzers, eleven samples, and two replicates at a single site covering a range from 0.1 % to 31 % volume fraction of biodiesel in a range of middle distillates yielded similar repeatability, and included fuels meeting specifications ISO 8217 and MIL-DTL-16884.

14.3 *Repeatability, r*—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:

$$\text{Repeatability} = 0.004685 \cdot x^{0.9}$$

where x is the average of the two results.

14.4 *Reproducibility*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material 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 is to be determined.

14.5 *Bias*—No known reference materials were tested as part of this study, therefore no statement on bias can be made at this time.

15. Keywords

15.1 biodiesel; biodiesel blend; biodiesel concentration; FAME; fatty acid methyl esters; infrared spectroscopy

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1906. Contact ASTM Customer Service at service@astm.org.