Designation: D8144 – 22

Standard Test Method for Separation and Determination of Aromatics, Nonaromatics, and FAME Fractions in Middle Distillates by Solid-Phase Extraction and Gas Chromatography[1]

This standard is issued under the fixed designation D8144; 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 separation and determination of representative aromatics, nonaromatics, and fatty acid methyl ester (FAME) fractions in middle distillates that boil between 170 °C and 400 °C, including biodiesel blends with up to 20 % by volume of FAME, by solid phase extraction and gas chromatography.

1.2 This test method provides two procedures, A and B. Procedure A is applicable to the petroleum-based middle distillates fuel, and Procedure B is applicable to the biodiesel blends with up to 20 % by volume of FAME.

1.3 This test method is applicable to middle distillates samples with aromatics content ranging from 5 % to 50 % by mass and biodiesel blends with FAME content in the range of 0.5 % to 20 % by volume. This test method may apply to concentrations outside these ranges, but the precision has not been determined.

1.4 For Procedure B, biodiesels in the form of fatty acid ethyl ester (FAEE) can also fully elute into the FAME fraction, and they have the similar FID (flame ionization detector) relative response factors with that of FAME. The determined content of FAME fractions are the sum of concentrations of FAME and FAEE by this test method (see 3.2.5).

1.5 From the investigation results obtained for FAME determination, the low concentrations of monoglycerides (usually less than 0.5 % by mass in biodiesel blends) are not detectable under the gas chromatographic (GC) condition of this test method and will not interfere with the determination of FAME by Procedure B. As a result, biodiesel blends, conforming to the requirements of Specification D7467, containing up to 20 % by volume of biodiesel blendstock meeting the requirements in Specification D6751, typically contain concentrations of monoglycerides of less than 0.1 % by mass. The diglycerides and triglycerides, if present, are not detected under the GC condition of this test method due to their higher boiling points.

NOTE 1—If a sample is suspected of containing an abnormal FAME biodiesel feedstock than specified in Specification D6751, for example, a sample contaminated with vegetable oil with a high level of total triglycerides, the content of mono-, di-, or tri-glycerides in the isolated FAME fraction may be determined using Test Method D6584. Samples containing biodiesels with a high amount of glycerides than specified in Specification D6751 may contaminate the GC column and not recommended for this test method.

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

1.7 *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.*

1.8 *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]

D2425 Test Method for Hydrocarbon Types in Middle Distillates by Mass Spectrometry

D2549 Test Method for Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography

D2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

[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 D02.04.0L on Gas Chromatography 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

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
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
D6584 Test Method for Determination of Total Monoglycerides, Total Diglycerides, Total Triglycerides, and Free and Total Glycerin in B-100 Biodiesel Methyl Esters by Gas Chromatography
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 *Other Standards:*[3]

EN 14103 Fat and oil derivatives—Fatty Acid Methyl Esters (FAME)—Determination of ester and linolenic acid methyl ester contents
EN 14214 Automotive fuels—Fatty acid methyl esters (FAME) for diesel engines—Requirements and test methods

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aromatics fraction, n*—the portion of the sample desorbed with the dichloromethane-ethyl alcohol mixture eluants (Procedure A) and dichloromethane-n-hexane eluants (Procedure B); the aromatics fraction may contain aromatics, condensed naphthenic-aromatics, aromatic olefins, and compounds containing sulfur, nitrogen, and oxygen atoms.

3.2.2 *biodiesel, n*—a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

3.2.3 *biodiesel blend, n*—a blend of biodiesel fuel with petroleum-based diesel fuel.

3.2.4 *diesel fuel, n*—petroleum-based middle distillate fuel.

3.2.5 *fatty acid methyl ester fraction, n*—the portion of the diesel fuels blends with fatty acid methyl ester (FAME) eluted with dichloromethane-ethyl alcohol; the FAME fraction may contain FAEE and compounds containing nitrogen and oxygen atoms.

3.2.6 *nonaromatics fraction, n*—the portion of the sample eluted with n-hexane.

3.2.6.1 *Discussion*—The nonaromatics fraction is a mixture of paraffinic and naphthenic hydrocarbons if the sample is a straight-run material. If the sample is a cracked stock, the nonaromatics fraction will also contain aliphatic and cyclic olefins.

3.2.7 *solid phase extraction separating system, n*—a solid-phase extraction cartridge packed with stationary phase material to effectively separate the aromatics, nonaromatics, and other compounds (such as FAME) fractions in middle distillates based on the mechanism of solid phase extraction (SPE).

4. Summary of Test Method

4.1 *Procedure A*—The sample is charged to the top of a SPE column and separated into aromatics and nonaromatics fractions by eluants with different polarities. Two aliquots of internal standards are added to these two fractions and both fractions are analyzed by the gas chromatograph equipped with hydrogen flame ionization detector (GC-FID). The content of the aromatics and nonaromatics are calculated based on the peak areas of the aromatics, nonaromatics, and internal standards.

4.2 *Procedure B*—The sample is charged to the top of a SPE column and separated into aromatics, nonaromatics, and FAME fractions by eluants with different polarities. Three aliquots of internal standards are added to these three fractions. All of these fractions are analyzed by the gas chromatograph equipped with hydrogen flame ionization detector (GC-FID). The content of the aromatics, nonaromatics, and FAME fractions are calculated based on the peak areas of the aromatics, nonaromatics, FAME, and internal standards. The volume percent of FAME is calculated based on the density of sample and mass percent of FAME.

5. Significance and Use

5.1 For the middle distillates whose boiling range is between 170 °C and 400 °C by such distillation methods like Test Method D2887, Procedure A can separate and determine the content of total aromatics and total nonaromatics by SPE and GC analysis of the resulting fractions. The determination of the total content of saturates and aromatics in petroleum middle distillates is useful to investigate the effects of petroleum processes on production of various finished fuels.

5.2 The total aromatics content and polycyclic aromatics content are important to characterize the quality of diesel fuels. This test method is demonstrated to be time-saving and eco-friendly by reducing the amount of reagent consumption and avoiding the necessity of solvent evaporation step as required, for example, in such Test Method D2549.

5.3 The determination of detailed hydrocarbon composition by mass spectrometry requires a preliminary separation of the sample into representative aromatics and nonaromatics, as in Test Method D2425, where Test Method D2549 is used to separate the distillate fuel. The SPE fractionation procedure described herein may provide a suitable fractionation alternative approach for these mass spectrometric types of methods.

5.4 Biodiesel is a blendstock commodity primarily used as a value-added blending component with diesel fuel. Procedure B can provide a separation and determination technique to monitor the FAME content for FAME biodiesel blends.

[3] Available from British Standards Institution (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., http://www.bsigroup.com.