

Designation: D 6751 - 07b

An American National Standard

## Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels<sup>1</sup>

This standard is issued under the fixed designation D 6751; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

#### 1. Scope\*

1.1 This specification covers biodiesel (B100) Grades S15 and S500 for use as a blend component with middle distillate fuels.

1.2 This specification prescribes the required properties of diesel fuels at the time and place of delivery. The specification requirements may be applied at other points in the production and distribution system when provided by agreement between the purchaser and the supplier.

1.3 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 1—The generation and dissipation of static electricity can create problems in the handling of distillate fuel oils with which biodiesel may be blended. For more information on the subject, see Guide D 4865.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

#### 2. Referenced Documents

2.1 ASTM Standards: <sup>2</sup>

- ASTM D67
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test
- D 189 Test Method for Conradson Carbon Residue of Petroleum Products
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

- D 613 Test Method for Cetane Number of Diesel Fuel Oil
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- D 874 Test Method for Sulfated Ash from Lubricating Oils and Additives
- D 974 Test Method for Acid and Base Number by Color-Indicator Titration
- D 975 Specification for Diesel Fuel Oils
- D 976 Test Method for Calculated Cetane Index of Distillate Fuels
- D 1160 Test Method for Distillation of Petroleum Products at Reduced Pressure
- D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)
- D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
- D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
- D 2500 Test Method for Cloud Point of Petroleum Products
- D 2622 Test Method for Sulfur in Petroleum Products by
- Wavelength Dispersive X-ray Fluorescence Spectrometry
- D 2709 Test Method for Water and Sediment in Middle
- Distillate Fuels by Centrifuge D 2880 Specification for Gas Turbine Fuel Oils
- D 3117 Test Method for Wax Appearance Point of Distillate Fuels
- D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
- D 3242 Test Method for Acidity in Aviation Turbine Fuel
- D 3828 Test Methods for Flash Point by Small Scale Closed Cup Tester
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy-Dispersive X-ray Fluorescence Spectrometry

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

<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

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<sup>&</sup>lt;sup>2</sup> 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.

- D 4530 Test Method for Determination of Carbon Residue (Micro Method)
- D 4737 Test Method for Calculated Cetane Index by Four Variable Equation
- D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
- D 4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
- D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
- D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
- D 6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
- D 6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester
- D 6469 Guide for Microbial Contamination in Fuels and Fuel Systems
- D 6584 Test Method for Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters By Gas Chromatography
- D 6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
- D 7039 Test Method for Sulfur in Gasoline and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry
- 2.2 Government Standard:
- 40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act<sup>3</sup>
- 2.3 Other Documents:<sup>4</sup>
- UOP 389 Trace Metals in Oils by Wet Ashing and ICP-OES
- UOP 391–91 Trace Metals in Petroleum Products or Organ-09a ics by AAS
- EN 14112 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of oxidation stability (Accelerated oxidation test)<sup>5</sup>
- EN 14110 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of methanol content<sup>5</sup>
- EN 14538 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of Ca, K, Mg and Na content by optical emission spectral analysis with inductively coupled plasma (ICP OES)<sup>5</sup>

#### 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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

3.1.1.1 *Discussion—Biodiesel*, as defined above, is registered with the U.S. EPA as a fuel and a fuel additive under Section 211(b) of the Clean Air Act. There is, however, other usage of the term biodiesel in the marketplace. Due to its EPA registration and the widespread commercial use of the term biodiesel in the U.S. marketplace, the term biodiesel will be maintained for this specification.

3.1.1.2 *Discussion*—Biodiesel is typically produced by a reaction of a vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-alkyl esters and glycerin, which is removed. The finished biodiesel derives approximately 10% of its mass from the reacted alcohol. The alcohol used in the reaction may or may not come from renewable resources.

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

3.1.2.1 *Discussion*—In the abbreviation BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.3 *biodiesel fuel*, *n*—synonym for *biodiesel*.

3.1.4 *diesel fuel*, *n*—middle petroleum distillate fuel.

3.1.5 *free glycerin*, *n*—a measure of the amount of glycerin remaining in the fuel.

3.1.6 *Grade S15 B100*, *n*—a grade of biodiesel meeting ASTM Specification D 6751 and having a sulfur specification of 15 ppm maximum.

3.1.7 *Grade S500 B100*, *n*—a grade of biodiesel meeting ASTM Specification D 6751 and having a sulfur specification of 500 ppm maximum.

3.1.8 *middle distillate fuel*, *n*—kerosines and gas oils boiling between approximately 150°C and 400°C at normal atmospheric pressure and having a closed-cup flash point above 38°C.

3.1.9 *total glycerin*, *n*—the sum of the free glycerin and the glycerin portion of any unreacted or partially reacted oil or fat.
4. Requirements

4.1 The biodiesel specified shall be mono-alkyl esters of long chain fatty acids derived from vegetable oils and animal fats.

4.2 Unless otherwise specified, samples for analysis shall be taken by the procedure described in Practices D 4057 or D 4177.

4.3 The biodiesel specified shall conform to the detailed requirements shown in Table 1.

NOTE 2—A considerable amount of experience exists in the U.S. with a 20 % blend of biodiesel, primarily produced from soybean oil, with 80 % diesel fuel (B20). Experience with biodiesel produced from animal fat and other oils is similar. Experience with B20 and lower blends in other applications is not as prevalent. Although biodiesel (B100) can be used, blends of over 20 % biodiesel with diesel fuel (B20) should be evaluated on a case by case basis until further experience is available.

NOTE 3—The user should consult the equipment manufacturer or owner's manual regarding the suitability of using biodiesel or biodiesel blends in a particular engine or application.

#### 5. Test Methods

5.1 The requirements enumerated in this specification shall be determined in accordance with the following methods.

5.1.1 *Flash Point*—Test Methods D 93, except where other methods are prescribed by law. Test Methods D 3828 or

<sup>&</sup>lt;sup>3</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

<sup>&</sup>lt;sup>4</sup> Available from ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA. Visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org.

<sup>&</sup>lt;sup>5</sup> Available from the National CEN Members listed on the CEN website (www.cenorm.be) or via the liaised CEN/TC19 secretariat (energy@nen.nl).

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| Property                            | Test Method <sup>A</sup> | Grade S15<br>Limits  | Grade S500<br>Limits        | Units        |
|-------------------------------------|--------------------------|----------------------|-----------------------------|--------------|
| Calcium and Magnesium, combined     | EN 14538                 | 5 max                | 5 max                       | ppm (µg/g)   |
| Flash point (closed cup)            | D 93                     | 93 min               | 93 min                      | °C           |
| Alcohol Control                     |                          |                      |                             |              |
| One of the following must be met:   |                          |                      |                             |              |
| 1. Methanol content                 | EN 14110                 | 0.2 max              | 0.2 max                     | % volume     |
| 2. Flash point                      | D 93                     | 130 min              | 130 min                     | °C           |
| Water and sediment                  | D 2709                   | 0.050 max            | 0.050 max                   | % volume     |
| Kinematic viscosity, 40°C           | D 445                    | 1.9-6.0 <sup>B</sup> | 1.9–6.0 <sup><i>B</i></sup> | mm²/s        |
| Sulfated ash                        | D 874                    | 0.020 max            | 0.020 max                   | % mass       |
| Sulfur <sup>C</sup>                 | D 5453                   | 0.0015 max (15)      | 0.05 max (500)              | % mass (ppm) |
| Copper strip corrosion              | D 130                    | No. 3 max            | No. 3 max                   |              |
| Cetane number                       | D 613                    | 47 min               | 47 min                      |              |
| Cloud point                         | D 2500                   | Report <sup>D</sup>  | Report <sup>D</sup>         | °C           |
| Carbon residue <sup>E</sup>         | D 4530                   | 0.050 max            | 0.050 max                   | % mass       |
| Acid number                         | D 664                    | 0.50 max             | 0.50 max                    | mg KOH/g     |
| Free glycerin                       | D 6584                   | 0.020                | 0.020                       | % mass       |
| Total glycerin                      | D 6584                   | 0.240                | 0.240                       | % mass       |
| Phosphorus content                  | D 4951                   | 0.001 max            | 0.001 max                   | % mass       |
| Distillation temperature,           | D 1160                   | 360 max              | 360 max                     | °C           |
| Atmospheric equivalent temperature, |                          |                      |                             |              |
| 90 % recovered                      |                          |                      |                             |              |
| Sodium and Potassium, combined      | EN 14538                 | 5 max                | 5 max                       | ppm (µg/g)   |
| Oxidation Stability                 | EN 14112                 | 3 minimum            | 3 minimum                   | hours        |

<sup>A</sup> The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.

<sup>B</sup> See X1.3.1. The 6.0 mm<sup>2</sup>/s upper viscosity limit is higher than petroleum based diesel fuel and should be taken into consideration when blending.

<sup>C</sup> Other sulfur limits can apply in selected areas in the United States and in other countries.

<sup>D</sup> The cloud point of biodiesel is generally higher than petroleum based diesel fuel and should be taken into consideration when blending.

<sup>E</sup> Carbon residue shall be run on the 100 % sample (see 5.1.11).

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D 6450 can also be used. The precision and bias of Test Methods D 3828 and D 6450 with biodiesel is not known and is currently under investigation. Test Methods D 93 shall be the referee method.

5.1.2 *Water and Sediment*—Test Method D 2709. Test Method D 1796 may also be used. Test Method D 2709 shall be the referee method. The precision and bias of these test methods with biodiesel is not known and is currently under investigation.

5.1.3 Viscosity—Test Method D 445.

- 5.1.4 Sulfated Ash—Test Method D 874.
- 5.1.5 Oxidation Stability—Test Method EN 14112.

5.1.6 *Sulfur*—Test Method D 5453. Test Method D 7039 may also be used. Other test methods may also be suitable for determining up to 0.05 % (500 ppm) sulfur in biodiesel fuels such as Test Methods D 1266, D 2622, D 3120 and D 4294 but may provide falsely high results (see X1.5) although their precision and bias with biodiesel is unknown. Test Method D 5453 shall be the referee test method.

5.1.7 Corrosion—Test Method D 130, 3 h test at 50°C.

5.1.8 *Cetane Number*—Test Method D 613. Test Method D 6890 may also be used. Test Method D 613 shall be the referee method.

5.1.9 *Cloud Point*—Test Method D 2500. Test Method D 5773 may also be used. Test Method D 3117 may also be used because it is closely related. Test Method D 2500 shall be the referee test method. The precision and bias of Test Method D 3117 for biodiesel is not known and is currently under investigation.

5.1.10 *Acid Number*—Test Method D 664. Test Methods D 3242 or D 974 may also be used. Test Method D 664 shall be the referee test method.

5.1.11 *Carbon Residue*—Test Method D 4530. A 100 % sample shall replace the 10 % residual, with percent residue in the original sample reported using the 10 % residual calculation (see X1.9.1). Test Methods D 189 or D 524 may also be used. Test Method D 4530 shall be the referee method.

5.1.12 Total Glycerin—Test Method D 6584.

5.1.13 Free Glycerin—Test Method D 6584.

5.1.14 Phosphorus Content—Test Method D 4951.

5.1.15 *Distillation Temperature, Reduced Pressure*—Test Method D 1160.

5.1.16 *Calcium and Magnesium, combined*—Test Method EN 14538. Test Method UOP 389 may also be used. Test Method EN 14538 shall be the referee test method.

5.1.17 Sodium and Potassium, combined—Test Method EN 14538. Test Method UOP 391 may also be used. Test Method EN 14538 shall be the referee test method.

#### 6. Workmanship

6.1 The biodiesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

#### 7. Keywords

7.1 alternative fuel; biodiesel fuel; diesel fuel oil; fuel oil; renewable resource

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#### APPENDIXES

#### (Nonmandatory Information)

#### **X1. SIGNIFICANCE OF PROPERTIES SPECIFIED FOR BIODIESEL FUEL**

#### **X1.1 Introduction**

X1.1.1 The properties of commercial biodiesel fuel depends upon the refining practices employed and the nature of the renewable lipids from which it is produced. Biodiesel, for example, can be produced from a variety of vegetable oils or animal fats which produce similar volatility characteristics and combustion emissions with varying cold flow properties.

X1.1.2 The significance of the properties in this appendix are based primarily on the commercial use of biodiesel in on-road and off-road diesel engine applications. Some of the properties may take on other significance if biodiesel is used as a fuel or blending component in other applications. See the respective finished product specifications for additional information on significance of properties of those applications.

#### X1.2 Flash Point

X1.2.1 The flash point, as specified, is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage that are normally specified to meet insurance and fire regulations.

X1.2.2 The flash point for biodiesel has been set at 93°C (200°F) minimum, so biodiesel falls under the non-hazardous category under National Fire Protection Association codes.

#### X1.3 Viscosity

X1.3.1 For some engines it may be advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum allowable viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system. The upper limit for the viscosity of biodiesel (6.0 mm<sup>2</sup>/s at 40°C) is higher than the maximum allowable viscosity in Specification D 975 Grade 2-D and 2-D low sulfur (4.1 mm/s at 40°C). Blending biodiesel with diesel fuel close to its upper limit could result in a biodiesel blend with viscosity above the upper limits contained in Specification D 975.

#### X1.4 Sulfated Ash

X1.4.1 Ash-forming materials may be present in biodiesel in three forms: (1) abrasive solids, (2) soluble metallic soaps, and (3) unremoved catalysts. Abrasive solids and unremoved catalysts can contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to filter plugging and engine deposits.

#### X1.5 Sulfur

X1.5.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can also affect emissions control systems performance and various

limits on sulfur have been imposed for environmental reasons. B100 is essentially sulfur-free.

NOTE X1.1—Test Method D 5453 should be used with biodiesel. Use of other test methods may provide falsely high results when analyzing B100 with extremely low sulfur levels (less than 5 ppm). Biodiesel sulfur analysis from RR: D02-1480<sup>6</sup>, *Biodiesel Fuel Cetane Number Testing Program, January-April, 1999*, using Test Method D 2622 yielded falsely high results due to the presence of the oxygen in the biodiesel. Sulfur results using Test Method D 2622 were more accurate with B20 than with B100 due to the lower oxygen content of B20. Potential improvements to Test Method D 2622 may provide more accurate values in the future.

#### X1.6 Copper Strip Corrosion

X1.6.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system. The presence of acids or sulfur-containing compounds can tarnish the copper strip, thus indicating the possibility for corrosion.

#### X1.7 Cetane Number

X1.7.1 Cetane number is a measure of the ignition quality of the fuel and influences white smoke and combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions.

X1.7.2 The calculated cetane index, Test Methods D 976 or D 4737, may not be used to approximate the cetane number with biodiesel or its blends. There is no substantiating data to support the calculation of cetane index with biodiesel or biodiesel blends.

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#### X1.8 Cloud Point

X1.8.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of crystals appears in the fuel under prescribed test conditions which generally relates to the temperature at which crystals begin to precipitate from the fuel in use. Biodiesel generally has a higher cloud point than petroleum based diesel fuel. The cloud point of biodiesel and its impact on the cold flow properties of the resulting blend should be monitored by the user to ensure trouble-free operation in cold climates. For further information, consult Appendix X4 of Specification D 975.

#### X1.9 Carbon Residue

X1.9.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil. While not directly correlating with engine deposits, this property is considered an approximation. Although biodiesel is in the distillate boiling range, most biodiesels boil at approximately the same temperature and it is difficult to leave a 10 % residual upon distillation. Thus, a 100 % sample is used to replace the 10 % residual

<sup>&</sup>lt;sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1480.