Designation: D6751 – 15c$^{\varepsilon 1}$

Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels[1]

This standard is issued under the fixed designation D6751; 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.

ε[1] NOTE—Subsection X1.3.1 was corrected editorially in February 2016.

1. Scope*

1.1 This specification covers four grades of biodiesel (B100) for use as a blend component with middle distillate fuels. These grades are described as follows:

1.1.1 *Grade No. 1-B S15*—A special purpose biodiesel blendstock intended for use in middle distillate fuel applications which can be sensitive to the presence of partially reacted glycerides, including those applications requiring good low temperature operability, and also requiring a fuel blend component with 15 ppm sulfur (maximum).

1.1.2 *Grade No. 1-B S500*—A special purpose biodiesel blendstock intended for use in middle distillate fuel applications which can be sensitive to the presence of partially reacted glycerides, including those applications requiring good low temperature operability, and also requiring a fuel blend component with 500 ppm sulfur (maximum).

1.1.3 *Grade No. 2-B S15*—A general purpose biodiesel blendstock intended for use in middle distillate fuel applications that require a fuel blend component with 15 ppm sulfur (maximum).

1.1.4 *Grade No. 2-B S500*—A general purpose biodiesel blendstock intended for use in middle distillate fuel applications that require a fuel blend component with 500 ppm sulfur (maximum).

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 D4865.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D189 Test Method for Conradson Carbon Residue of Petroleum Products

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

D613 Test Method for Cetane Number of Diesel Fuel Oil

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D874 Test Method for Sulfated Ash from Lubricating Oils and Additives

D974 Test Method for Acid and Base Number by Color-Indicator Titration

D975 Specification for Diesel Fuel Oils

D976 Test Method for Calculated Cetane Index of Distillate Fuels

D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure

D1266 Test Method for Sulfur in Petroleum Products (Lamp Method)

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, 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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[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**

D2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D2500 Test Method for Cloud Point of Petroleum Products
D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
D2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge
D2880 Specification for Gas Turbine Fuel Oils
D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)[3]
D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
D3242 Test Method for Acidity in Aviation Turbine Fuel
D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
D4530 Test Method for Determination of Carbon Residue (Micro Method)
D4737 Test Method for Calculated Cetane Index by Four Variable Equation
D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
D5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
D5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)
D5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
D5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
D6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
D6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester
D6469 Guide for Microbial Contamination in Fuels and Fuel Systems
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
D6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
D7039 Test Method for Sulfur in Gasoline, Diesel Fuel, Jet Fuel, Kerosine, Biodiesel, Biodiesel Blends, and Gasoline-Ethanol Blends by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry

[3] The last approved version of this historical standard is referenced on www.astm.org.

D7344 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Mini Method)
D7345 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Micro Distillation Method)
D7397 Test Method for Cloud Point of Petroleum Products (Miniaturized Optical Method)
D7501 Test Method for Determination of Fuel Filter Blocking Potential of Biodiesel (B100) Blend Stock by Cold Soak Filtration Test (CSFT)
D7668 Test Method for Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils—Ignition Delay and Combustion Delay Using a Constant Volume Combustion Chamber Method
D7689 Test Method for Cloud Point of Petroleum Products (Mini Method)

2.2 *Government Standard:*
40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act[4]

2.3 *Other Documents:*
AOCS Standard Procedure Ck 2-09[5] Determination of Various Properties of Biodiesel by the QTA System Method[6]
UOP 389 Trace Metals in Oils by Wet Ashing and ICP-OES[7]
UOP 391–91 Trace Metals in Petroleum Products or Organics by AAS[7]
EN 14105 Fat and Oil Derivatives—Fatty Acid Methyl Esters (FAME)—Determination of Free and Total Glycerol and Mono-, Di-, Triglyceride Contents (Reference Method)[8]
EN 14110 Fat and Oil Derivatives—Fatty Acid Methyl Esters (FAME)—Determination of Methanol Content[8]
EN 14112 Fat and Oil Derivatives—Fatty Acid Methyl Esters (FAME)—Determination of Oxidation Stability (Accelerated Oxidation Test)[8]
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)[8]
EN 15751 Automotive Fuels—Fatty Acid Methyl Ester (FAME) Fuel and Blends with Diesel Fuel—Determination of Oxidation Stability by Accelerated Oxidation Method[8]

3. Terminology

3.1 *Definitions:*

3.1.1 *biodiesel, n*—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 (40 CFR Part 79). 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*—blend of biodiesel fuel with diesel fuel oils.

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 D6751 and having a sulfur specification of 15 ppm maximum.

3.1.7 *Grade S500 B100, n*—a grade of biodiesel meeting ASTM Specification D6751 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 *monoglyceride, n*—a partially reacted fat or oil molecule with one long chain alkyl ester group on a glycerin backbone.

3.1.10 *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 D4057 or D4177.

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.

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.
[5] Available from AOCS Headquarters, 2710 S. Boulder, Urbana, IL 61802–6996. Download Product Code: MC-CK209 from www.aocs.org.
[6] QTA is a registered trademark of the Cognis Corporation, 5051 Estecreek Drive, Cincinnati, OH 45232-1446.
[7] 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.
[8] Available from the National CEN Members listed on the CEN website (www.cenorm.be) or from the CEN/TC19 secretariat (astm@nen.nl).

TABLE 1 Detailed Requirements for Biodiesel (B100) Blend Stocks

Property	Test Method[A]	Grade No. 1-B S15	Grade No. 1-B S500	Grade No. 2-B S15	Grade No. 2-B S500
Sulfur,[B] % mass (ppm), max	D5453	0.0015 (15)	0.05 (500)	0.0015 (15)	0.05 (500)
Cold soak filterability, seconds, max	D7501	200	200	360[C]	360[C]
Monoglyceride content, % mass, max	D6584	0.40	0.40	...	...
			Requirements for All Grades		
Calcium and Magnesium, combined, ppm (µg/g), max	EN 14538	5	5	5	5
Flash point (closed cup), °C, min	D93	93	93	93	93
Alcohol control					
One of the following shall be met:					
1. Methanol content, mass %, max	EN 14110	0.2	0.2	0.2	0.2
2. Flash point, °C, min	D93	130	130	130	130
Water and sediment, % volume, max	D2709	0.050	0.050	0.050	0.050
Kinematic viscosity,[D] mm^2/s, 40 °C	D445	1.9-6.0	1.9-6.0	1.9-6.0	1.9-6.0
Sulfated ash, % mass, max	D874	0.020	0.020	0.020	0.020
Copper strip corrosion, max	D130	No. 3	No. 3	No. 3	No. 3
Cetane number, min	D613	47	47	47	47
Cloud point,[E] °C	D2500	Report	Report	Report	Report
Carbon residue,[F] % mass, max	D4530	0.050	0.050	0.050	0.050
Acid number, mg KOH/g, max	D664	0.50	0.50	0.50	0.50
Free glycerin, % mass, max	D6584	0.020	0.020	0.020	0.020
Total glycerin, % mass, max	D6584	0.240	0.240	0.240	0.240
Phosphorus content, % mass, max	D4951	0.001	0.001	0.001	0.001
Distillation temperature, Atmospheric equivalent temperature, 90 % recovered, °C, max	D1160	360	360	360	360
Sodium and Potassium, combined, ppm (µg/g), max	EN 14538	5	5	5	5
Oxidation stability, hours, min	EN 15751	3	3	3	3

[A] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.
[B] Other sulfur limits may apply in selected areas in the United States and in other countries.
[C] For additional cold weather considerations, see Appendix X3.
[D] See X1.3.1. The 6.0 mm^2/s upper viscosity limit is higher than petroleum based diesel fuel and should be taken into consideration when blending.
[E] The cloud point of biodiesel is generally higher than petroleum based diesel fuel and should be taken into consideration when blending.
[F] Carbon residue shall be run on the 100 % sample (see 5.1.12).

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 D93, except where other methods are prescribed by law. Test Methods D3828 or D6450 can also be used. The precision and bias of Test Methods D3828 and D6450 with biodiesel is not known and is currently under investigation. Test Methods D93 shall be the referee method.

5.1.2 *Water and Sediment*—Test Method D2709. Test Method D1796 may also be used. Test Method D2709 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 D445.

5.1.4 *Monoglycerides*—Test Method D6584, Test Method EN 14105, and AOCS Standard Procedure Ck 2-09 may be used. Test Method D6584 shall be the referee test method.

5.1.5 *Sulfated Ash*—Test Method D874.

5.1.6 *Oxidation Stability*—Test Method EN 15751. Test Method EN 14112 may also be used. See X1.19.1 for further information. Test Method EN 15751 shall be the referee test method.

5.1.7 *Sulfur*—Test Method D5453. Test Method D7039 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 D1266, D2622, D3120 and D4294 but may provide falsely high results (see X1.5) although their precision and bias with biodiesel is unknown. Test Method D5453 shall be the referee test method.

5.1.8 *Corrosion*—Test Method D130, 3 h test at 50°C.

5.1.9 *Cetane Number*—Test Method D613. Test Method D6890 or D7668 (see Note 4) may also be used. In case of dispute, Test Method D613 shall be the referee method.

NOTE 4—Precision from Test Method D7668 were obtained from results produced by laboratories using externally obtained pre-blended calibration reference material.

5.1.10 *Cloud Point*—Test Method D2500. Test Method D5771, D5772, D5773, D7397, D7689, or AOCS Standard Procedure Ck 2-09 may also be used. Test Method D3117 may also be used because it is closely related. Test Method D2500 shall be the referee test method. The precision and bias of Test Method D3117 for biodiesel is not known and is currently under investigation.

5.1.11 *Acid Number*—Test Method D664. Test Methods D3242 or D974 may also be used. Test Method D664 shall be the referee test method.

5.1.12 *Carbon Residue*—Test Method D4530. 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 D189 or D524 may also be used. Test Method D4530 shall be the referee method.

5.1.13 *Total Glycerin*—Test Method D6584. AOCS Standard Procedure Ck 2-09 may also be used. Test Method D6584 is the referee method.

5.1.14 *Free Glycerin*—Test Method D6584. AOCS Standard Procedure Ck 2-09 may also be used. Test Method D6584 is the referee method.

5.1.15 *Phosphorus Content*—Test Method D4951.

5.1.16 *Distillation Temperature*—Test Method(s) D1160 (reduced pressure method), or D7344, or D7345 may be used. When using Test Method D7344, correct for observed bias by adding 3 °C to the temperature result before comparing the result to the Table 1 requirement and report the Test Method D7344 value as "bias-corrected." Test Method D1160 shall be the referee test method.

5.1.17 *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.

5.1.18 *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.

5.1.19 *Cold Soak Filterability*—Test Method D7501. B100 intended for blending into diesel fuels that is expected to give satisfactory vehicle performance at fuel temperatures at or below –12°C shall comply with a cold soak filtration limit of 200 s maximum.

5.1.20 *Methanol Content*—Test Method EN 14110. AOCS Standard Procedure Ck 2-09 may also be used. Test Method EN 14110 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

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^2/s at 40 °C) is higher than the maximum allowable viscosity in Specification D975 Grade 2-D and 2-D low sulfur (4.1 mm^2/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 D975.

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 D5453 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[9], *Biodiesel Fuel Cetane Number Testing Program, January-April, 1999*, using Test Method D2622 yielded falsely high results due to the presence of the oxygen in the biodiesel. Sulfur results using Test Method D2622 were more accurate with B20 than with B100 due to the lower oxygen content of B20. Potential improvements to Test Method D2622 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 D976 or D4737, 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.

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 D975.

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.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1480.

Thus, a 100 % sample is used to replace the 10 % residual sample, with the calculation executed as if it were the 10 % residual. Parameter E (final weight flask charge/original weight flask charge) in 8.1.2 of Test Method D4530-93 is a constant 20/200.

X1.10 Acid Number

X1.10.1 The acid number is used to determine the level of free fatty acids or processing acids that may be present in biodiesel. Biodiesel with a high acid number has been shown to increase fueling system deposits and may increase the likelihood for corrosion.

NOTE X1.2—Acid number measures a different phenomenon for biodiesel than petroleum based diesel fuel. The acid number for biodiesel measures free fatty acids or degradation by-products not found in petroleum based diesel fuel. Increased recycle temperatures in new fuel system designs may accelerate fuel degradation which could result in high acid values and increased filter plugging potential.

X1.11 Free Glycerin

X1.11.1 The free glycerin method is used to determine the level of glycerin in the fuel. High levels of free glycerin can cause injector deposits, as well as clogged fueling systems, and result in a buildup of free glycerin in the bottom of storage and fueling systems.

X1.12 Total Glycerin

X1.12.1 The total glycerin method is used to determine the level of glycerin in the fuel and includes the free glycerin and the glycerine portion of any unreacted or partially reacted oil or fat. Low levels of total glycerin ensure that high conversion of the oil or fat into its mono-alkyl esters has taken place. High levels of mono-, di-, and triglycerides can cause injector deposits and may adversely affect cold weather operation and filter plugging.

X1.13 Monoglycerides

X1.13.1 See information provided in Appendix X3.

X1.14 Phosphorus Content

X1.14.1 Phosphorus can damage catalytic converters used in emissions control systems and its level must be kept low. Catalytic converters are becoming more common on diesel-powered equipment as emissions standards are tightened, so low phosphorus levels will be of increasing importance. Biodiesel produced from U.S. sources has been shown to have low phosphorus content (below 1 ppm) and the specification value of 10 ppm maximum is not problematic. Biodiesel from other sources may or may not contain higher levels of phosphorus and this specification was added to ensure that all biodiesel, regardless of the source, has low phosphorus content.

X1.15 Reduced Pressure Distillation

X1.15.1 Biodiesel exhibits a boiling point rather than a distillation curve. The fatty acids chains in the raw oils and fats from which biodiesel is produced are mainly comprised of straight chain hydrocarbons with 16 to 18 carbons that have similar boiling temperatures. The atmospheric boiling point of biodiesel generally ranges from 330 to 357°C, thus the specification value of 360°C is not problematic. This specification was incorporated as an added precaution to ensure the fuel has not been adulterated with high boiling contaminants.

NOTE X1.3—The density of biodiesel meeting the specifications in Table 1 falls between 0.86 and 0.90, with typical values falling between 0.88 and 0.89. Since biodiesel density falls between 0.86 and 0.90, a separate specification is not needed. The density of raw oils and fats is similar to biodiesel, therefore use of density as an expedient check of fuel quality may not be as useful for biodiesel as it is for petroleum based diesel fuel. This section has been added to provide users and engine interests with this information.

NOTE X1.4—In certain items of fuel injection equipment in compression ignition engines, such as rotary/distributor fuel pumps and injectors, the fuel functions as a lubricant as well as a source for combustion. Blending biodiesel fuel with petroleum based compression-ignition fuel typically improves fuel lubricity.

X1.16 Alcohol Control

X1.16.1 Alcohol control is to limit the level of unreacted alcohol remaining in the finished fuel. This can be measured directly by the volume percent alcohol or indirectly through a high flash point value.

X1.16.2 The flash point specification, when used for alcohol control for biodiesel, is intended to be 100°C minimum, which has been correlated to 0.2 vol % alcohol. Typical values are over 160 °C. Due to high variability with Test Method D93 as the flash point approaches 100°C, the flash point specification has been set at 130 °C minimum to ensure an actual value of 100 °C minimum. Improvements and alternatives to Test Method D93 are being investigated. Once complete, the specification of 100 °C minimum may be reevaluated for alcohol control.

X1.17 Calcium and Magnesium

X1.17.1 Calcium and magnesium may be present in biodiesel as abrasive solids or soluble metallic soaps. Abrasive solids can contribute to injector, fuel pump, piston, and ring wear, as well as to engine deposits. Soluble metallic soaps have little effect on wear, but they may contribute to filter plugging and engine deposits. High levels of calcium and magnesium compounds may also be collected in exhaust particulate removal devices, are not typically removed during passive or active regeneration, and can create increased back pressure and reduced time to service maintenance.

X1.18 Sodium and Potassium

X1.18.1 Sodium and potassium may be present in biodiesel as abrasive solids or soluble metallic soaps. Abrasive solids can contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear, but they may contribute to filter plugging and engine deposits. High levels of sodium or potassium compounds may also be collected in exhaust particulate removal devices, are not typically removed during passive or active regeneration, and they can create increased back pressure and reduced period to service maintenance.

X1.19 Oxidation Stability

X1.19.1 Products of oxidation in biodiesel can take the form of various acids or polymers, which, if in high enough

concentration, can cause fuel system deposits and lead to filter clogging and fuel system malfunctions. Additives designed to retard the formation of acids and polymers can significantly improve the oxidation stability performance of biodiesel. See Appendix X2 for additional information on long-term storage.

It is recommended that EN 15751 be utilized for measurement of biodiesel oxidation stability, because EN 14112 may be withdrawn in the future as an option for testing biodiesel and biodiesel blends.

X2. LONG-TERM STORAGE OF BIODIESEL

X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of biodiesel (B100) who may wish to store quantities of fuels for extended periods. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage. This appendix is directed toward biodiesel (B100) and may be more or less applicable to blends of biodiesel with petroleum based diesel fuel.

X2.1.2 Normally produced biodiesel has adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products, although data suggests some biodiesel may degrade faster than petroleum based diesel fuel. Biodiesel that is to be stored for prolonged periods should be selected to avoid formation of sediments, high acid numbers, and high viscosities that can clog filters, affect fuel pump operation or plug combustor nozzles or injectors. The selection of biodiesel should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirement imposed by the warranty of the distillate fuel equipment manufacturers or by federal, state, or local government regulations. Although they cannot replace knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the biodiesel fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored.

X2.2 Terminology

X2.2.1 *bulk fuel*—fuel in the storage facility in quantities over 50 gallons.

X2.2.2 *combustor fuel*—fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

X2.2.3 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.4 *fuel-degradation products* —those materials formed in fuel after it is produced. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (acids and gums) may be more or less volatile than the fuel and may cause an increase in injector and nozzle deposits. The formation of degradation products may be catalyzed by contact with metals, especially those containing copper and, to a lesser extent, iron.

X2.2.5 *long-term storage*—storage of fuel for longer than 6 months after it is received by the user.

X2.3 Fuel Selection

X2.3.1 The stability properties of biodiesel are not fully understood and appear to depend on the vegetable oil and animal fat sources, severity of processing, and whether additional production plant treatment has been carried out or stability additives are present.

X2.3.2 The composition and stability properties of biodiesel produced at specific production plants may be different. Any special requirements of the user, such as long-term storage, should be discussed with the supplier.

X2.4 Fuel Additives

X2.4.1 Available fuel additives appear to improve the long term storage of biodiesel. Most additives should be added as close to the production site as possible to obtain maximum benefits.

X2.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in the fuel phase or the water phase, or both. Refer to Guide D6469 for a more complete discussion.

X2.5 Tests for Fuel Quality

X2.5.1 Test methods for estimating the storage stability of biodiesel (B100) are being developed. Modifications of Test Method D2274 to use glass fiber filters, varying times and temperatures, and the measurement of pre-test and post-test acid number and viscosity appear promising. However, correlation of this test with actual storage stability is unknown, and may depend upon field conditions and fuel composition.

X2.5.2 Performance criteria for accelerated stability tests that ensure satisfactory long-term storage of biodiesel (B100) have not been established.

X2.6 Fuel Monitoring

X2.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful monitoring program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X2.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D4057 provides guidance for sampling. Fuel contaminants and degradation products may settle

to the bottom of a quiescent tank although detrimental changes to biodiesel can occur (rising acid value) without causing sediment formation. A *Bottom* or *Clearance* sample, as defined in Practice D4057, should be included in the evaluation along with an *All Level* sample.

X2.6.3 The quantity of insoluble fuel contaminants present in biodiesel can be determined using Test Method D6217 with glass fiber filters and abundant washing although no precision or bias testing has been performed with biodiesel using Test Method D6217.

X2.6.4 The acid value of biodiesel appears to exceed its specified maximum before other deleterious fuel property changes occur. A conscientious program of measuring the acid value of biodiesel may be sufficient for monitoring biodiesel stability.

X2.7 Fuel Storage Conditions

X2.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Refer to Guide D6469 for a more complete discussion. Underground or isothermal storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel.

X2.7.2 Copper and copper-containing alloys should be avoided with biodiesel due to increased sediment and deposit formation. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

X2.7.3 Appendix X3 of Specification D2880 discusses fuel contaminants as a general topic. The discussion in Specification D2880 pertains to gas turbine combustion which may or may not be applicable to diesel engine combustion.

X3. LOW TEMPERATURE OPERABILITY OF BIODIESEL BLENDS

X3.1 General Considerations

X3.1.1 The cold flow properties of biodiesel (B100) meeting Specification D6751 depend mainly on the number of carbons in the fatty acid chains, the degree of saturation of the fatty acid chains, and the alcohol to which the fatty acid chains are esterified. Residual constituents from biodiesel raw materials and production processes can affect fuel filter operation with biodiesel and biodiesel blends as fuel temperatures become colder, as can contaminants that accumulate during fuel storage and distribution. The concentration of biodiesel inclusion can also affect the cold flow properties of the finished blend.

X3.1.2 It is unrealistic to specify low temperature properties of biodiesel blends that will ensure satisfactory operation at all ambient conditions in all storage situations. In general, cloud point, Low Temperature Flow Test (LTFT), and Cold Filter Plugging Point (CFPP) might be used as estimates of operating temperature limits for biodiesel blends, although precision data may not be available for biodiesel blends in all of these test methods. However, equipment design, operating conditions, and the use of flow-improver additives can allow satisfactory operation of the biodiesel blend below its cloud point.

X3.1.3 Due to fuel delivery system, engine design, and test method differences, low temperature operability tests may not provide the same degree of protection in all biodiesel blend applications or storage situations. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use of the biodiesel blend and the expected ambient temperatures.

X3.2 Influence of Residual Constituents and Contaminants

X3.2.1 Residual non-mono-alkyl ester constituents in biodiesel that remain from biodiesel raw materials and production processes can contribute to fuel filter fouling over time especially if biodiesel blends are stored at low temperatures. Certain residual constituents have been found on fouled biodiesel blend filters in the field. For example, sterol glucosides, monopalmitin, and monostearin have been identified in elevated quantities on biodiesel blend dispenser filters that fouled at temperatures above the cloud point of the blended fuel. Water in poorly maintained diesel distribution systems can interact with the residual constituents in biodiesel to increase the likelihood of filter clogging.

X3.2.2 Specification D6751 on B100 blend stock restricts some residual constituents and contaminants either directly or indirectly. The limit on Test Method D6584 total glycerin indirectly controls monoglycerides and related derivatives. Limits on Test Method D874 sulfated ash and EN 14538 for combined calcium/magnesium and combined sodium/potassium indirectly control soap levels. Test Method D2709 limits water and sediment content. Additionally, Test Method D7501 is a performance based filtration test that provides an assessment of the aggregate impact of residual constituents and contaminants on the filter clogging potential of the finished blend.

X3.2.3 Fuel contaminants found in distribution systems can also contribute to low temperature filter clogging with biodiesel and biodiesel blends.

X3.3 Low Temperature Operability Considerations

X3.3.1 In establishing low temperature operability requirements for biodiesel and biodiesel blends, consideration should be given to the following factors:

X3.3.1.1 Temperature guidelines such as those in Specification D975, Appendix X5, Tenth Percentile Minimum Ambient Air Temperatures for the United States (Except Hawaii).

X3.3.1.2 Short term local weather conditions (unusual cold periods do occur).

X3.3.1.3 Elevation (high locations are usually colder than surrounding lower areas).

X3.3.1.4 Specific engine design or other application.

X3.3.1.5 Fuel system design (for example, fuel recirculation rate, filter location, filter capacity, filter porosity).

X3.3.1.6 Fuel viscosity at low temperatures.

X3.3.1.7 The specific characteristics of the middle distillate fuel with which the biodiesel will be blended.

X3.3.1.8 Type of blending that will be used (in-line or splash).

X3.3.1.9 Temperature and volume of both the diesel and biodiesel to be blended.

X3.3.1.10 Equipment add-ons (engine heaters, radiator covers, fuel line and fuel filter heaters, exterior fuel filters, etc.).

X3.3.1.11 Types of operation (extensive idling, engine shutdown, or unusual operation).

X3.3.1.12 Low temperature flow improver additive effectiveness.

X3.3.1.13 Geographic area for fuel use and movement between geographic areas.

X3.3.1.14 General housekeeping (dirt, water, or both, in fuel or fuel supply system).

X3.3.1.15 Impact of failure for engine to start or run effectively (critical vs. non-critical application).

X3.3.2 Some biodiesel residual constituents might not come out of solution in a biodiesel blend unless exposed to cold temperature for an extended period of time. These constituents may come out of solution above the measured cloud point of the blend and might or might not go back into solution when the blend is warmed to a higher temperature. Specification D6751 contains requirements such as cold soak filterability and total glycerin to reduce the potential influence of such constituents on low temperature operability.

X3.3.3 Work in the area of low temperature operability is ongoing by several organizations in 2010, such as the Coordinating Research Council (CRC) Diesel Performance Group and the ASTM Biodiesel Task Force. These groups include representatives from the engine and filter equipment manufacturers, fuel injection equipment manufacturers, fuel producers and additive suppliers. These groups are investigating field reports involving filter plugging of distribution system filters and vehicle filters, causal factors for filter plugging with biodiesel blends, and controlled laboratory analysis of vehicle performance under cold conditions.

SUMMARY OF CHANGES

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751 – 15b) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Added Test Method D7344 to Section 2, Referenced Documents, and to subsection 5.1.16.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751 – 15a) that may impact the use of this standard. (Approved Oct. 1, 2015.)

(1) Added Test Method D7345 to Section 2, Referenced Documents, and to subsection 5.1.16.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751 – 15) that may impact the use of this standard. (Approved June 1, 2015.)

(1) Added Test Method D7689 to Section 2, Referenced Documents, and to subsection 5.1.10.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751 – 14) that may impact the use of this standard. (Approved Jan. 1, 2015.)

(1) Revised subsection 5.1.9 to include Test Method D7668.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751 – 12) that may impact the use of this standard. (Approved Oct. 1, 2014.)

(1) Revised footnote C of Table 1.