



Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels¹

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.

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 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.

1.4.1 *Exception*—In Annex A1, 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: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)
- 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
- 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

¹ 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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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.

- 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
- D5452** Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
- D5453** Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
- 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
- D6300** Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
- 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 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 and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry
- D7397** Test Method for Cloud Point of Petroleum Products (Miniaturized Optical Method)
- 2.2 *Government Standard:*
- 40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act³
- 2.3 *Other Documents:*⁴
- UOP 389** Trace Metals in Oils by Wet Ashing and ICP-OES
- UOP 391–91** Trace Metals in Petroleum Products or Organics by AAS
- EN 14112** Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of oxidation stability (Accelerated oxidation test)⁵
- EN 14110** Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of methanol content⁵

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)⁵

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. 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 *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

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

⁴ 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.

⁵ 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) (All Sulfur Levels)

Property	Test Method ^A	Grade S15 Limits	Grade S500 Limits	Units
Calcium and Magnesium, combined	EN 14538	5 max	5 max	ppm (µg/g)
Flash point (closed cup)	D93	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	mass %
2. Flash point	D93	130 min	130 min	°C
Water and sediment	D2709	0.050 max	0.050 max	% volume
Kinematic viscosity, 40°C	D445	1.9–6.0 ^B	1.9–6.0 ^B	mm ² /s
Sulfated ash	D874	0.020 max	0.020 max	% mass
Sulfur ^C	D5453	0.0015 max (15)	0.05 max (500)	% mass (ppm)
Copper strip corrosion	D130	No. 3 max	No. 3 max	
Cetane number	D613	47 min	47 min	
Cloud point	D2500	Report ^D	Report ^D	°C
Carbon residue ^E	D4530	0.050 max	0.050 max	% mass
Acid number	D664	0.50 max	0.50 max	mg KOH/g
Cold soak filterability	Annex A1	360 max ^F	360 max ^F	seconds
Free glycerin	D6584	0.020 max	0.020 max	% mass
Total glycerin	D6584	0.240 max	0.240 max	% mass
Phosphorus content	D4951	0.001 max	0.001 max	% mass
Distillation temperature, Atmospheric equivalent temperature, 90 % recovered	D1160	360 max	360 max	°C
Sodium and Potassium, combined	EN 14538	5 max	5 max	ppm (µg/g)
Oxidation stability	EN 14112	3 minimum	3 minimum	hours

^A The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.

^B See X1.3.1. The 6.0 mm²/s upper viscosity limit is higher than petroleum based diesel fuel and should be taken into consideration when blending.

^C Other sulfur limits can apply in selected areas in the United States and in other countries.

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

^E Carbon residue shall be run on the 100 % sample (see 5.1.11).

^F B100 intended for blending into diesel fuel that is expected to give satisfactory vehicle performance at fuel temperatures at or below –12°C shall comply with a cold soak filterability limit of 200 s maximum.

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 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 *Sulfated Ash*—Test Method D874.

5.1.5 *Oxidation Stability*—Test Method EN 14112.

5.1.6 *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.

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

5.1.8 *Cetane Number*—Test Method D613. Test Method D6890 may also be used. Test Method D613 shall be the referee method.

5.1.9 *Cloud Point*—Test Method D2500. Test Method D5773 or D7397 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.

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

5.1.11 *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.

5.1.12 *Total Glycerin*—Test Method D6584.

5.1.13 *Free Glycerin*—Test Method D6584.

5.1.14 *Phosphorus Content*—Test Method D4951.

5.1.15 *Distillation Temperature, Reduced Pressure*—Test Method D1160.

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.

5.1.18 *Cold Soak Filterability*—The test method in Annex A1 shall be used to determine the cold soak filterability. 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. A cold soak filterability standard test method is under development.

NOTE 4—Interim precision information is provided in Annex A1 for the cold soak filterability test to give the user some indication of the repeatability and reproducibility expected.

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

ANNEX

(Mandatory Information)

A1. DETERMINATION OF FUEL FILTER BLOCKING POTENTIAL OF BIODIESEL (B100) BLEND STOCK BY COLD SOAK LABORATORY FILTRATION

A1.1 Scope

A1.1.1 This test method covers the determination by filtration time after cold soak of the suitability for a Biodiesel (B100) Blend Stock for blending with middle distillates to provide adequate low temperature operability performance to at least the cloud point of the finished blend.

A1.1.2 The interim precision of this test method has been determined.

A1.1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A1.1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

A1.2 Referenced Documents

A1.2.1 *ASTM Standards:*²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration

A1.3 Terminology

A1.3.1 *Definitions:*

A1.3.1.1 *biodiesel, n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

A1.3.1.2 *bond, v*—to connect two parts of a system electrically by means of a conductive wire to eliminate voltage differences.

A1.3.1.3 *ground, v*—to connect electrically with earth.

A1.3.2 *Definitions of Terms Specific to this Standard:*

A1.3.2.1 *filtered flushing fluids, n*—either of two solvents, heptane or 2,2, 4–trimethylpentane, filtered through a nominal $0.45\ \mu\text{m}$ glass fiber filter.

A1.3.2.2 *glass fiber filter, n*—the $0.7\ \mu\text{m}$ glass fiber filters used in this test method.

A1.3.3 *Abbreviations:*

A1.3.3.1 *CSFT*—cold soak filtration test.

A1.4 Summary of Test Method

A1.4.1 In this test method, 300 mL of biodiesel (B100) is stored at 4.4°C (40°F) for 16 h, allowed to warm to 20 to 22°C (68 to 72°F), and vacuum filtered through a single $0.7\ \mu\text{m}$ glass fiber filter.

A1.4.2 In this test method, the filtration time is reported in seconds.

A1.5 Significance and Use

A1.5.1 Some substances that are soluble or appear to be soluble in biodiesel at room temperature will, upon cooling or standing at room temperature for extended periods, come out of solution. These substances can cause filter plugging. This test method provides an accelerated means of assessing the propensity for these substances to plug filters.

A1.5.1.1 Fuels that give short filtration times are expected to give satisfactory operation down to the cloud point of biodiesel blends.

A1.5.2 The test method can be used in specifications as a means of controlling levels of minor filter plugging components in biodiesel and biodiesel blends.

A1.6 Apparatus

A1.6.1 *Filtration System*—Arrange the following components as shown in Fig. A1.1.

A1.6.1.1 *Funnel and Funnel Base*, with a stainless steel filter support for a 47-mm diameter glass fiber filter and a locking ring or spring action clip capable of receiving 300 mL.

NOTE A1.1—Sintered glass supports were found to give much higher filtration times during initial studies and should not be used.

A1.6.1.2 *Ground/Bond Wire*, 0.912 to $2.59\ \text{mm}$ (No. 10 through No. 19) bare-stranded flexible stainless steel or copper installed in the flasks and grounded as shown in Fig. A1.1.

NOTE A1.2—The electrical bonding apparatus described in Test Method D5452 or other suitable means of electrical grounding which ensure safe operation of the filtration apparatus and flask can be used. If the filtrate is to be subsequently tested for stability it is advisable not to use

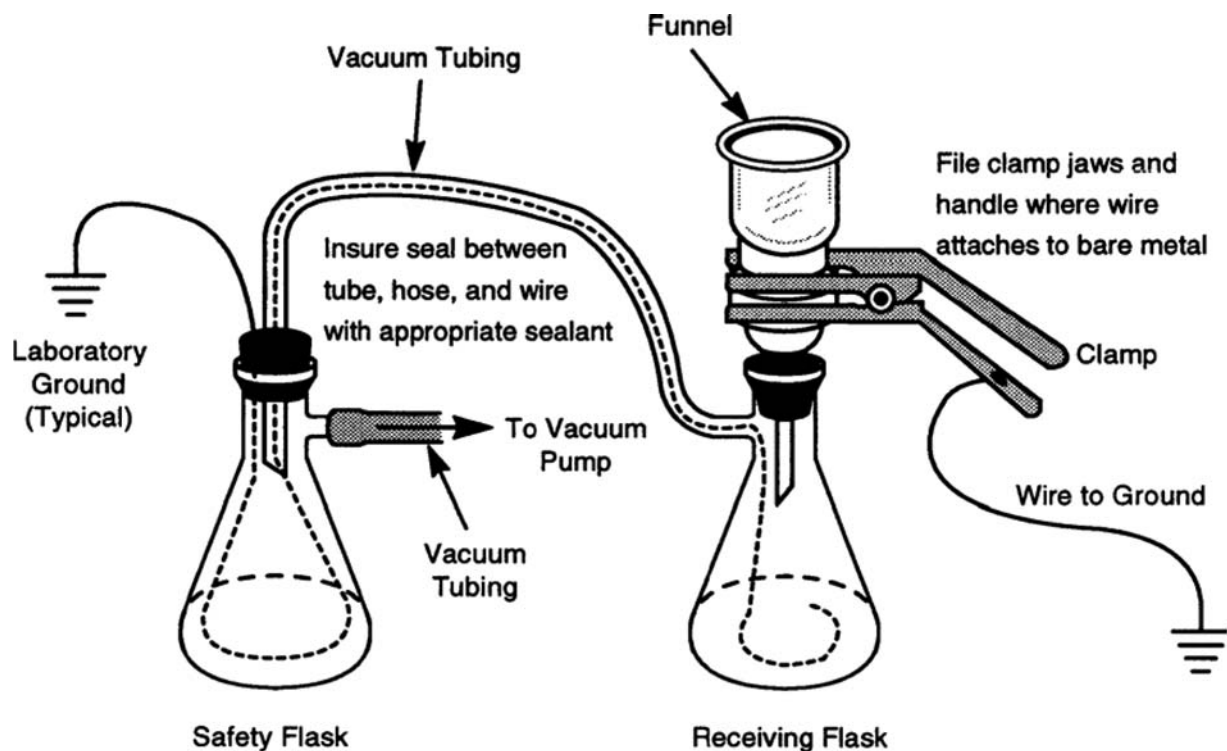


FIG. A1.1 Schematic of Filtration System

copper as copper ions catalyze gum formation during the stability test.

A1.6.1.3 *Receiving Flask*, 1-L borosilicate glass vacuum filter flask, into which the filtration apparatus fits, equipped with a sidearm to connect to the safety flask.

A1.6.1.4 *Safety Flask*, 1-L borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistant rubber hose, through which the grounding wire passes, shall connect the sidearm of the receiving flask to the tube passing through the rubber stopper in the top of the safety flask.

A1.6.1.5 *Vacuum System*, a vacuum system capable of producing a vacuum of 70 to 100 kPa below atmospheric pressure when measured at the receiving flask. A mechanical vacuum pump may be used if it has this capability.

NOTE A1.3—Water aspirated vacuum will not provide relative vacuum within the prescribed range.

A1.6.2 Other Apparatus:

A1.6.2.1 *Forceps*, approximately 12-cm long, flat-bladed, with non-serrated, non-pointed tips.

A1.6.2.2 *Graduated Cylinders*, to contain at least 0.5 L of fluid and marked at 10-mL intervals. Graduated cylinders, 100-mL, may be required for samples which filter slowly.

A1.6.2.3 *Petri Dishes*, approximately 12.5 cm in diameter, with removable glass supports for glass fiber filters.

NOTE A1.4—Small watch glasses, approximately 5 to 7 cm in diameter, have also been found suitable to support the glass fiber filters.

NOTE A1.5—B100 will dissolve some plastics. This can cause the filters to adhere to the plastic.

A1.6.2.4 *Glass Fiber Filters*, plain, 47-mm diameter, nominal pore size 0.7- μ m.

A1.6.2.5 *Protective Cover*, polyethylene film or clean aluminum foil.

A1.6.2.6 *Liquid or Air Bath or Chamber*, capable of sustaining a temperature of $4.4 \pm 1.1^\circ\text{C}$ ($40 \pm 2^\circ\text{F}$) for 16 h.

A1.6.2.7 *Timer*, capable of displaying elapsed times of at least 900 s to the nearest 0.1 s.

A1.7 Reagents and Materials

A1.7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

A1.7.2 *Flushing Fluids*—Flushing fluids are not required for the test as the filter is not weighed. However, heptane or isooctane may be used to wash the apparatus after filtration to remove any residue. Alternatively soap and water may be used in accordance with A1.7.3.

A1.7.2.1 *Heptane*, (**Warning**—Flammable).

A1.7.2.2 *2,2,4-trimethylpentane (isooctane)*, (**Warning**—Flammable).

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

A1.7.3 *Liquid or Powder Detergent*, water-soluble, for cleaning glassware.

A1.8 Preparation of Apparatus and Sample Containers

A1.8.1 Clean all components of the filtration apparatus using the reagents described in [A1.7.2](#) and [A1.7.3](#).

A1.8.1.1 Remove any labels, tags, and so forth.

A1.9 Sampling

A1.9.1 The sample container should be 500 ± 15 mL in volume and have a screw-on cap with an inert liner. Glass containers are preferred to facilitate a visual inspection of the contents and the container before and after filling. Glass containers also allow for visual inspection of the container, after the sample is emptied, to confirm complete emptying of the container. Epoxy-lined sample cans, polytetrafluoroethylene (PTFE) bottles, and high density linear polyethylene bottles have also been found suitable as sample containers but are less desirable since visual inspection of the interior of the container is more difficult.

A1.9.2 Precautions to avoid sample contamination shall include selection of an appropriate sampling point. It is preferred to obtain samples dynamically from a sampling loop in a distribution line, or from the flushing line of a field sampling kit. Ensure that the line to be sampled is flushed with fuel before collecting the sample.

A1.9.2.1 Use clean sample containers.

A1.9.2.2 Keep a clean protective cover over the top of the sample container until the cap is installed. Similarly protect the funnel opening of the assembled filtration apparatus with a clean protective cover until ready for use.

A1.9.2.3 Where it is desirable or only possible to obtain samples from static storage, follow the procedures given in Practice [D4057](#) or equivalent, taking precautions for cleanliness of all equipment used. The sample should pass through a minimum number of intermediate containers prior to placement in the prepared container.

A1.9.2.4 Samples obtained from static storage can give results that are not representative of the bulk contents of the tank because of particulate matter settling. Where possible, the contents of the tank should be circulated or agitated before sampling, or the sampling should be performed shortly after a tank has been filled.

A1.9.3 Visually inspect the sample container before taking the samples to verify that there are no visible particles present inside the container. Fill the sample container to contain 300 mL. Protect the fuel sample from prolonged exposure to light by wrapping the container in aluminum foil or storing it in the dark to reduce the possibility of particulate formation by light-promoted reactions. Do not transfer the fuel sample from its original sample container into an intermediate storage container. If the original sample container is damaged or leaking, then a new sample shall be obtained.

A1.9.3.1 If a 500-mL bottle is not available, or the sample has already been received in a container not suitable for this test, follow [A1.9.5](#).

A1.9.4 Analyze fuel samples as soon as possible after sampling.

A1.9.4.1 Upon receipt of a Biodiesel Blend Stock (B100) sample, the entire sample shall be heated to 40°C for at least 3 h under an inert atmosphere to erase any thermal history and to dissolve any solids that might have precipitated during transit unless it is known that the sample has never been cooled below 20°C. If the sample has never been exposed to temperatures below 20°C then proceed to [A1.9.5](#).

A1.9.4.2 After heating for the required time, allow the sample to sit for 24 h at a temperature no lower than 20°C.

A1.9.5 Shake the sample vigorously for 1 min, and transfer 300 mL to a clean fresh 500 ± 15 mL bottle.

A1.10 Preparation of Glass Fiber Filter

A1.10.1 Each filtration uses one filter. The glass fiber filter used for each individual test shall be identified by marking the petri dishes used to hold and transport the filters.

A1.10.2 Clean all glassware used in preparation of glass fiber filter as described in [A1.8.1](#).

A1.10.3 Using forceps, place the filters on clean glass support rods or watch glasses in petri dish.

A1.10.4 Place the petri dish with its lid slightly ajar in a drying oven at $90 \pm 5^\circ\text{C}$, and leave it for 30 min.

A1.10.5 Remove the petri dish from the drying oven. Keep the petri dish cover ajar, such that the filter is protected from contamination from the atmosphere. Allow 30 min for the filter to come to equilibrium with room air temperature and humidity.

A1.10.6 Using clean forceps, place the filter centrally on the filter support of the filtration apparatus (see [Fig. A1.1](#)). Install the funnel and secure with locking ring or spring clip. Do not remove the plastic film from the funnel opening until ready to start filtration.

A1.11 Procedure

A1.11.1 Place 300 mL of sample in a glass 500-mL bottle, and set in a liquid or air bath or chamber at $4.4 \pm 1.1^\circ\text{C}$ ($40 \pm 2^\circ\text{F}$) for 16 ± 0.5 h.

A1.11.2 After the 16-h cold soak is completed, allow the sample to come back to room temperature at 20 to 22°C (68 to 72°F) on its own without external heating. The sample shall be completely liquid before filtration. The sample shall be filtered within 1 h after reaching 20 to 22°C (68 to 72°F).

A1.11.3 Complete assembly of the receiving flask, 0.7 μm glass fiber filter and funnel as a unit (see [Fig. A1.1](#)) before swirling the sample. To minimize operator exposure to fumes, the filtering procedure should be performed in a fume hood.

A1.11.4 Start the vacuum system. Record the pressure in the system after 1 min of filtration. The vacuum shall be between 71.1 and 84.7 kPa (21 and 25 in. Hg) below atmospheric pressure. If the vacuum is not within the specified range, make adjustments to the vacuum system.

A1.11.5 Thoroughly clean the outside of the sample container in the region of the cap by wiping it with a damp, lint-free cloth. Swirl the container vigorously for about 2 to 3 s to dislodge any particles that may have adhered to the walls of the container.

A1.11.6 Immediately after swirling, pour the entire contents of the sample container into the filtration funnel and simultaneously start the timer. The entire contents of the sample

container shall be filtered through the glass fiber filter to ensure a correct measure of the contamination in the sample.

NOTE A1.6—Take care not to shake the sample vigorously, as this could cause some of the solids to go back into solution.

A1.11.7 If the filtration is not complete when 720 s (12 min) has elapsed, turn off the vacuum system and record the duration of the filtration to the nearest second. Record the pressure in the system and the volume filtered just before the termination of the filtration.

A1.12 Reporting

A1.12.1 Report the time for the 300-mL B100 to be completely filtered as B100 filtration time in seconds.

A1.12.2 If the filtration of the 300 mL failed to be completed after 720 s, report the volume that was filtered after 720 s.

A1.13 Precision and Bias

A1.13.1 *Precision*—The precision of this test method for B100 filtration has not yet been determined.

A1.13.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material for B100 filtration has not yet been determined.

A1.13.1.2 *Reproducibility*—The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material for B100 filtration has not yet been determined.

A1.13.1.3 *Interim Precision*—Repeatability and reproducibility determinations were made using data from the ASTM Biodiesel Low Temperature Operability Task Force. The analysis of the data is the subject of a research report, RR:D02-

1649.⁷ The report is an attempt to supply such an analysis based on well-established methodologies. Subsequent to test method publication a more thorough round robin is planned.

	200 s	360 s
Repeatability	34.0	61.0
Reproducibility	115.9	208.1

NOTE A1.7—The degree of freedom associated with the repeatability estimate from this round robin study is 25 for repeatability which is below 30 but acceptable. The degree of freedom associated with the reproducibility estimate from this round robin study is 10 and below acceptable limits. For that reason only the repeatability is included in A1.13.1.4. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual repeatability/reproducibility may be significantly different than these estimates. An ASTM ILS will be conducted in the future.

A1.13.1.4 The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material for B100 filtration would in the long run, in the normal and correct operation of this test method, exceed $0.1689(X + 1.2018)$ time(s) only in one case in twenty.

A1.13.2 *Bias*—The procedure given for the determination of B100 filtration time has no bias because the value of the filtration time is defined in terms of this test method.

A1.14 Keywords

A1.14.1 biodiesel; diesel fuel; glass fiber filter; biodiesel; filter blocking potential, cold soak filtration test, CSFT, biodiesel blend; laboratory filtration; glass fiber filter; low temperature operability, middle distillate fuel.

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1649.

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 mm²/s at 40°C) is higher than the maximum allowable viscosity in Specification **D975** Grade 2-D and 2-D low sulfur (4.1 mm²/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:D02-1480⁸, *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. 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 Phosphorus Content

X1.13.1 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

⁸ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1480.

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.

X1.14 Reduced Pressure Distillation

X1.14.1 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.

X1.15 Alcohol Control

X1.15.1 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.

X1.15.2 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.

X1.16 Calcium and Magnesium

X1.16.1 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.

X1.17 Sodium and Potassium

X1.17.1 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.

X1.18 Oxidation Stability

X1.18.1 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.

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, corre-

lation 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.

SUMMARY OF CHANGES

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–08) that may impact the use of this standard. (Approved April 15, 2009.)

(1) Added Test Method **D7397** cloud point as an option to Test Method **D2500** in **5.1.9** and to the Referenced Documents.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–07b^{e1}) that may impact the use of this standard. (Approved Oct. 1, 2008.)

(1) Added **Annex A1**.

(3) Added **5.1.18**.

(2) Revised **Table 1**.

(4) Added **Note 4**.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–07a) that may impact the use of this standard. (Approved Sept. 15, 2007.)

(1) Added Test Method **D6890** to Referenced Documents and **5.1.8**.

(3) Revised **1.2**.

(2) Added Test Method **D7039** to Referenced Documents and **5.1.6**.

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