Designation: D 6751 – 08

An American National Standard

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

This standard is issued under the fixed designation D 6751; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (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 D 4865.

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]

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D 189 Test Method for Conradson Carbon Residue of Petroleum Products

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

D 613 Test Method for Cetane Number of Diesel Fuel Oil

D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D 874 Test Method for Sulfated Ash from Lubricating Oils and Additives

D 974 Test Method for Acid and Base Number by Color-Indicator Titration

D 975 Specification for Diesel Fuel Oils

D 976 Test Method for Calculated Cetane Index of Distillate Fuels

D 1160 Test Method for Distillation of Petroleum Products at Reduced Pressure

D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)

D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)

D 2500 Test Method for Cloud Point of Petroleum Products

D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry

D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge

D 2880 Specification for Gas Turbine Fuel Oils

D 3117 Test Method for Wax Appearance Point of Distillate Fuels

D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry

D 3242 Test Method for Acidity in Aviation Turbine Fuel

D 3828 Test Methods for Flash Point by Small Scale Closed Cup Tester

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

[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.

Current edition approved Oct. 1, 2008. Published October 2008. Originally approved in 1999 as PS 121–99. Adopted as a standard in 2002 as D 6751–02. Last previous edition approved in 2007 as D 6751–07b$^{\varepsilon 1}$.

[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.**

D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
D 4530 Test Method for Determination of Carbon Residue (Micro Method)
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D 4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
D 5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
D 6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
D 6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester
D 6469 Guide for Microbial Contamination in Fuels and Fuel Systems
D 6584 Test Method for Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters By Gas Chromatography
D 6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
D 7039 Test Method for Sulfur in Gasoline and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry

2.2 *Government Standard:*

40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act[3]

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 content[5]
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 D 6751 and having a sulfur specification of 15 ppm maximum.

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

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

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

4. Requirements

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

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

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

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

[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)	D 93	93 min	93 min	°C
Alcohol control				
One of the following must be met:				
1. Methanol content	EN 14110	0.2 max	0.2 max	mass %
2. Flash point	D 93	130 min	130 min	°C
Water and sediment	D 2709	0.050 max	0.050 max	% volume
Kinematic viscosity, 40°C	D 445	1.9-6.0[B]	1.9–6.0[B]	mm^2/s
Sulfated ash	D 874	0.020 max	0.020 max	% mass
Sulfur[C]	D 5453	0.0015 max (15)	0.05 max (500)	% mass (ppm)
Copper strip corrosion	D 130	No. 3 max	No. 3 max	
Cetane number	D 613	47 min	47 min	
Cloud point	D 2500	Report[D]	Report[D]	°C
Carbon residue[E]	D 4530	0.050 max	0.050 max	% mass
Acid number	D 664	0.50 max	0.50 max	mg KOH/g
Cold soak filterability	Annex A1	360 max[F]	360 max[F]	seconds
Free glycerin	D 6584	0.020 max	0.020 max	% mass
Total glycerin	D 6584	0.240 max	0.240 max	% mass
Phosphorus content	D 4951	0.001 max	0.001 max	% mass
Distillation temperature, Atmospheric equivalent temperature, 90 % recovered	D 1160	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 mm^2/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.

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

5. Test Methods

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

5.1.1 *Flash Point*—Test Methods D 93, except where other methods are prescribed by law. Test Methods D 3828 or D 6450 can also be used. The precision and bias of Test Methods D 3828 and D 6450 with biodiesel is not known and is currently under investigation. Test Methods D 93 shall be the referee method.

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

5.1.3 *Viscosity*—Test Method D 445.

5.1.4 *Sulfated Ash*—Test Method D 874.

5.1.5 *Oxidation Stability*—Test Method EN 14112.

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

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

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

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

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

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

5.1.12 *Total Glycerin*—Test Method D 6584.

5.1.13 *Free Glycerin*—Test Method D 6584.

5.1.14 *Phosphorus Content*—Test Method D 4951.

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

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

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

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]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D 5452 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 µm glass fiber filter.

A1.3.2.2 *glass fiber filter, n*—the 0.7 µm 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 µm 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 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

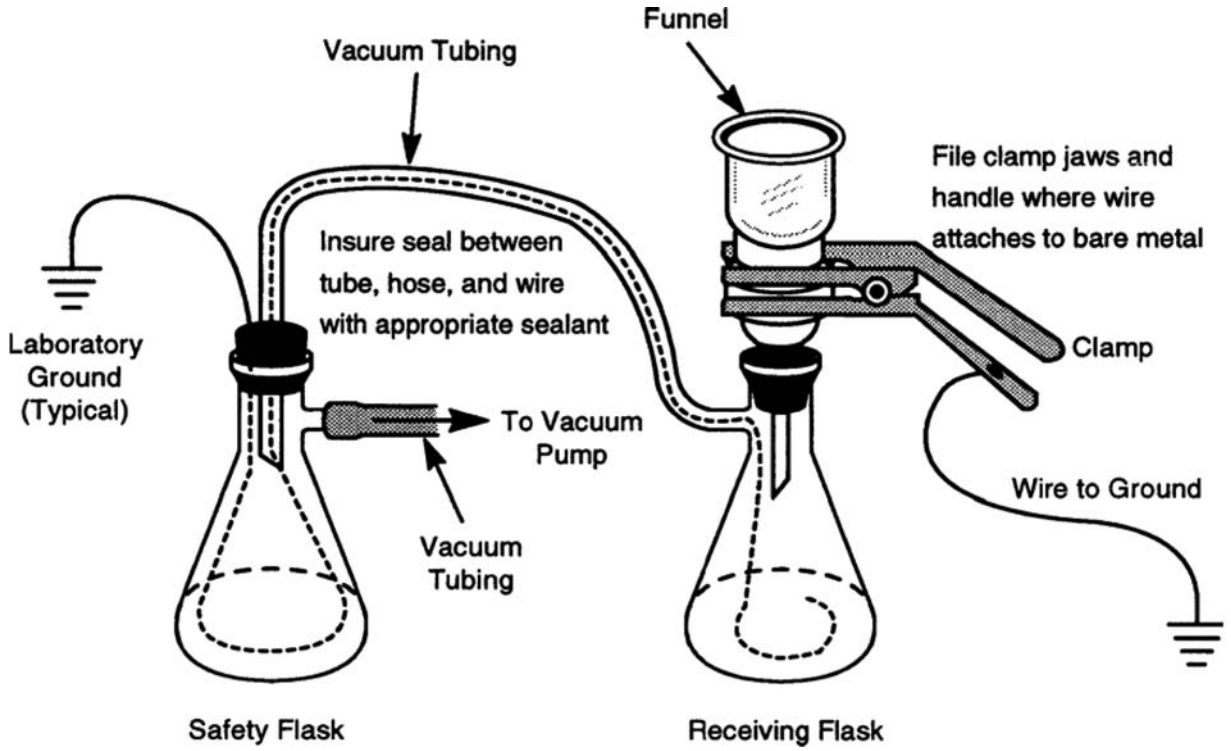

FIG. A1.1 Schematic of Filtration System

Method D 5452 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 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-µm.

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 ± 1.1°C (40 ± 2°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).

[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.

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

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 D 4057 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 ± 5°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 ± 1.1°C (40 ± 2°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.