



Designation: D7501 – 12a

# Standard Test Method for Determination of Fuel Filter Blocking Potential of Biodiesel (B100) Blend Stock by Cold Soak Filtration Test (CSFT)<sup>1</sup>

This standard is issued under the fixed designation D7501; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method covers the determination by filtration time after cold soak of the suitability for a biodiesel (B100) blend stock that meets all other requirements of Specification D6751 and has a cloud point below 20°C (68°F) to provide adequate low temperature operability performance to at least the cloud point of the finished blend.

1.2 The formal precision of this test method is included in 13.1 on Precision.

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

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.*

## 2. Referenced Documents

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

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

## 3. Terminology

3.1 *Definitions:*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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

3.1.3 *BXX blend, n*—fuel blend consisting of up to 20 volume percent biodiesel designated as up to B20 conforming to the requirements of Specification D6751 with the remainder being a light middle or middle distillate grade diesel fuel and meeting the requirements of this test method.

3.1.3.1 *Discussion*—The abbreviation BXX represents a specific blend concentration in the range B2 to B20, where XX is the percent volume of biodiesel in the fuel blend.

3.1.4 *ground, v*—to connect electrically with earth.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air chamber, n*—unit to control temperature of sample for cooling with maximum 0.5°C variation from set point. The unit shall use a microprocessor temperature controller with digital set point and readout. A food-grade refrigerator does not provide adequate temperature stability for this test method.

3.2.2 *minor component, n*—material present in B100, typically at concentrations well below 1% by mass.

3.2.3 *thermal history, n*—range of temperatures that a batch or sample of B100 has experienced which can result in separation or precipitation of minor components.

3.2.3.1 *Discussion*—Cooling some biodiesel fuels can result in precipitation or separation of minor components that have limited solubility in B100. Heating these biodiesel fuels above 40°C is believed to redissolve most of these minor components and thus erase the thermal history of the sample.

3.3 *Abbreviations:*

3.3.1 *CSFT*—Cold Soak Filtration Test

## 4. Summary of Test Method

4.1 In this test method, 300 mL of biodiesel (B100) is stored at 4.5 ± 0.5°C (40 ± 1°F) for 16 h, allowed to warm to 25 ± 1°C (77 ± 2°F), and vacuum filtered through a single 0.7 μm glass fiber filter at controlled vacuum levels of ~70–85 kPa (21–25 in. of Hg).

4.2 The filtration time is reported in seconds.

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

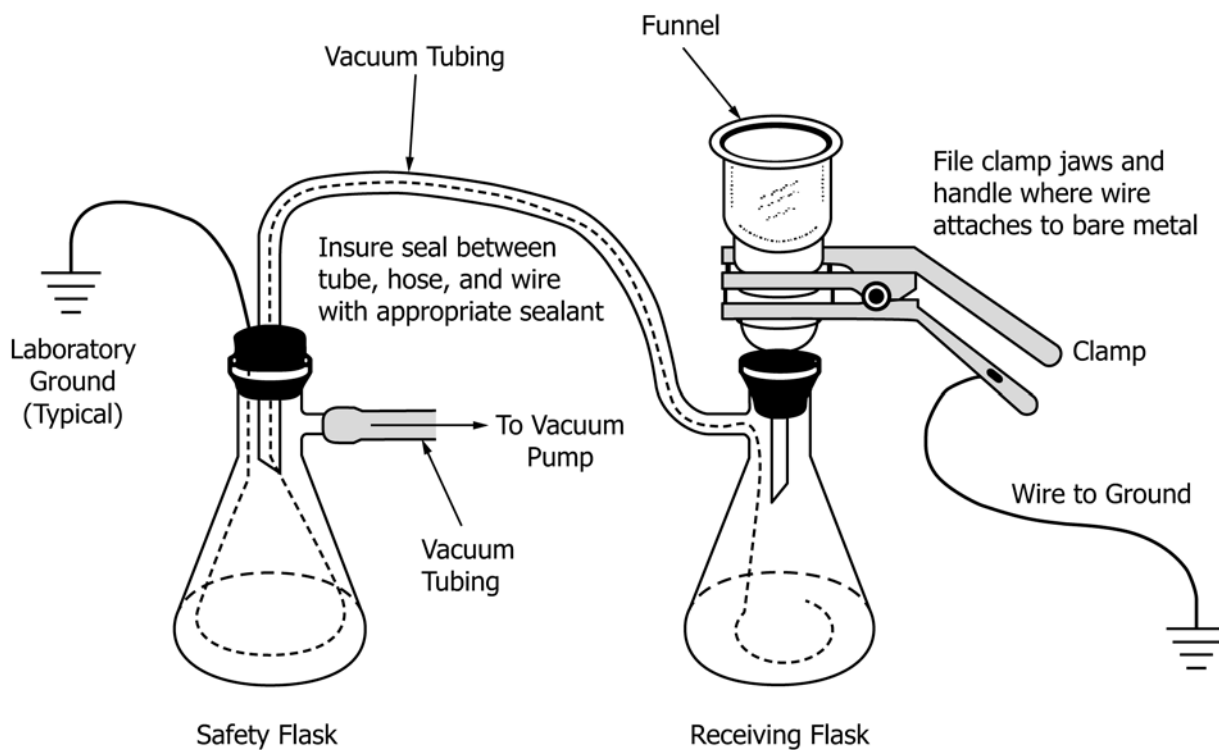


FIG. 1 Schematic of Filtration System

## 5. Significance and Use

5.1 Some substances that are soluble or appear to be soluble in biodiesel (B100) at room temperature will, upon cooling to temperatures above the cloud point or standing at room temperature for extended periods, come out of solution. This phenomenon has been observed in both B100 and BXX blends. These substances can cause filter plugging. This method provides an accelerated means of assessing the presence of these substances in B100 and their propensity to plug filters.

5.1.1 B100 biodiesel fuels that give short filtration times are expected to give satisfactory operation of BXX blends at least down to the cloud point of the biodiesel blends.

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.

## 6. Apparatus

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

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

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

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

6.1.3 *Electrical Bonding Apparatus*, as described in Test Method D6217 or by using other suitable means of electrical grounding that ensure safe operation of the filtration apparatus

and flask. This may not be necessary in filtering biodiesel B100 because of the higher electrical conductivity.

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

6.1.5 *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 is connected to the sidearm of the receiving flask to the tube, passes through the rubber stopper in the top of the safety flask.

6.1.6 *Vacuum System*, capable of producing a vacuum of ~70 to 85 kPa (21 to 25 in. of Hg) when measured at the safety flask. A mechanical vacuum pump may be used if it has this capability. A vacuum pump with free air displacement capacity of 25 to 30 L/min at 60 Hz has been found to be suitable to pull the vacuum down to the required level within 30 to 40 s when guard and filtration flasks are present in the assembly.

NOTE 2—Water aspirated vacuum will not provide relative vacuum within the prescribed range.

6.1.7 *Chilling Apparatus*, either 6.1.7.1 or 6.1.7.2 may be used.

6.1.7.1 *Circulating Water Bath*, capable of sustaining a temperature of  $4.5 \pm 0.5^\circ\text{C}$  ( $40 \pm 1^\circ\text{F}$ ).

6.1.7.2 *Air Chamber*, capable of sustaining a temperature of  $4.5 \pm 0.5^\circ\text{C}$  ( $40 \pm 1^\circ\text{F}$ ). The unit shall use microprocessor temperature control and have a digital temperature control/display.

6.1.8 *Circulating Water Bath*, capable of sustaining a temperature of  $25 \pm 0.5^\circ\text{C}$  ( $77 \pm 1^\circ\text{F}$ ).

6.1.9 *Whatman Glass Fiber Filters (GF/F)*,<sup>3</sup> plain, 47-mm diameter, nominal pore size 0.7- $\mu$ m. Acid treated low metal TCLP filters are not suitable for this purpose.

#### 6.2 Other Apparatus:

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

6.2.2 *Graduated Cylinders*, to contain at least 0.5 L of fluid and marked at 10-mL intervals. Samples that filter slowly may require 100-mL graduated cylinders.

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

6.2.3.1 *Small Watch Glasses*, approximately 5 to 7 cm in diameter, have been found suitable to support the glass fiber filters.

NOTE 3—B100 will dissolve some plastics. This can cause the filters to adhere to the plastic.

6.2.4 *Protective Cover*, polyethylene film or clean aluminum foil.

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

6.2.6 *Thermometer or RTD Sensor*, for measuring sample temperature, should be capable of measurement  $\pm 0.5^{\circ}\text{C}$  ( $\pm 1^{\circ}\text{F}$ ).

## 7. Reagents and Materials

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. 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.

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.

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

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

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

## 8. Sampling

8.1 A separate representative sample shall be collected for this test method. Samples may preferentially be obtained 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. Alternatively, circulate or agitate the contents of the tank in order to collect a representative sample that includes any precipitates that might have settled in the tank. One option is to sample the tank immediately after filling, before the contents have time to settle.

8.1.1 Where it is necessary to obtain samples from static storage using a sampling container or intermediate containers, follow the procedures given in Practice D4057 or equivalent, taking precautions for cleanliness of all equipment used. This is interpreted to mean a representative sample in accordance with Practice D4057, and refers to an “all-levels” sample. (**Warning**—Samples obtained from static storage can give results which are not representative of the bulk contents of the tank because of particulate matter settling.)

8.2 *Precautions*—to avoid sample contamination include:

8.2.1 Selection of an appropriate sampling point.

8.2.2 Use of clean sample containers. Visually inspect the sample container before taking the samples to verify that there are no visible particles present inside the container.

8.2.3 Keeping a clean protective cover over the top of the sample container until the cap is replaced after filling.

8.2.4 If the primary sample container cannot be selected such that it is suitable for this test procedure, the sample shall pass through only a minimum number of intermediate containers prior to placement in the final container.

8.2.5 After filling the sample container with 300 mL of sample, 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.

8.3 If 300 mL of sample is not received in a 500-mL bottle or the sample has been received in a container not suitable for this test, follow the sampling condition procedure in 8.4.

8.4 *Sample Conditioning*—for samples received that might have been cooled to temperatures below 20°C (68°F):

8.4.1 Upon receipt of a biodiesel blend stock (B100) sample, the entire sample is to be heated to 40°C (104°F) 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. After heating for the required time, allow the sample to sit for 24 h at a temperature no lower than 20°C (68°F).

8.4.2 This sample conditioning step may be omitted if the sample was collected from a heated tank at a production facility. In this case, proceed directly to 8.5.

8.4.3 If there is any doubt about the thermal history of the sample, the sample conditioning step shall be performed.

8.5 Analyze fuel samples as soon as possible after sampling.

8.6 Shake the sample vigorously for 1 min. If the sample is not already in a 500-mL bottle suitable for this test procedure, transfer 300 mL to a clean, fresh 500  $\pm$  15-mL bottle. If a 300-mL sample was received is already in such a bottle, proceed with the test procedure in Section 11.

## 9. Preparation of Apparatus and Sample Containers

9.1 Clean all components of the filtration apparatus using the reagents described in 7.2 and 7.3.

9.1.1 Filtration apparatus shall be clean and dry prior to use.

9.1.2 Remove any labels, tags, and so forth.

<sup>3</sup> Whatman filters were used in the ILS. See Section 13.