



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

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 (ϵ) 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:*²

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:*

¹ 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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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.

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 $4.5 \pm 0.5^\circ\text{C}$ ($40 \pm 1^\circ\text{F}$) for 16 h, allowed to warm to $25 \pm 1^\circ\text{C}$ ($77 \pm 2^\circ\text{F}$), 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

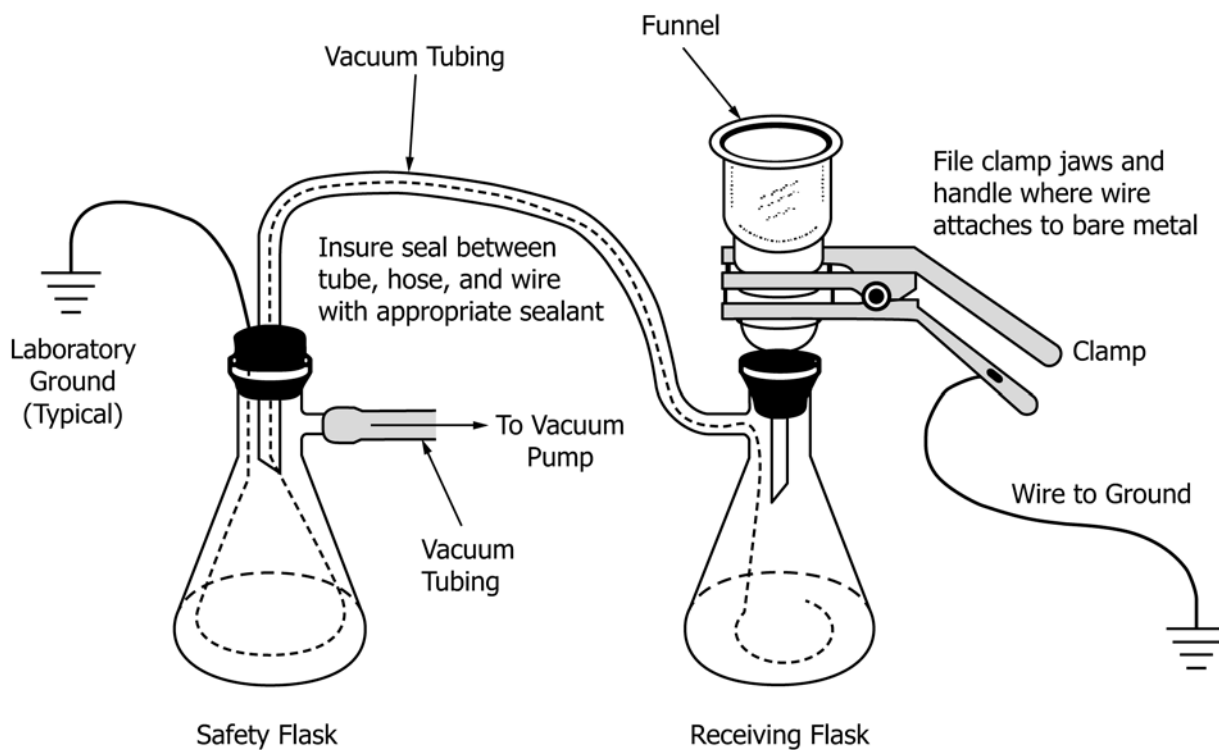


FIG. 1 Schematic of Filtration System

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)*,³ plain, 47-mm diameter, nominal pore size 0.7- μ 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 ± 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.

³ Whatman filters were used in the ILS. See Section **13**.

10. Preparation of Glass Fiber Filter

10.1 Each filtration uses one filter as specified in 6.1.9. The glass fiber filter used for each individual test may be identified by marking the petri dish used to hold and transport the filter.

10.2 Clean all glassware used in preparation of glass fiber filter as described in 9.1.

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

10.4 Place the petri dish, with its lid slightly ajar, in a drying oven at $90 \pm 2^\circ\text{C}$ ($194 \pm 3.6^\circ\text{F}$) and leave it for 30 min.

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.

10.6 Using clean forceps, place the filter centrally on the filter support of the filtration apparatus (see Fig. 1). Install the funnel and secure with locking ring or spring clip.

10.7 Protect the funnel opening of the assembled filtration apparatus with a clean protective cover until ready for use.

11. Procedure

11.1 Place 300 mL of sample in a 500 mL glass bottle and set in a liquid or air chamber at $4.5 \pm 0.5^\circ\text{C}$ ($40 \pm 1^\circ\text{F}$) for 16 ± 0.25 h. A refrigerator is *not* suitable for this procedure.

11.2 After the 16-h cold soak at $4.5 \pm 0.5^\circ\text{C}$ ($40 \pm 1^\circ\text{F}$) is completed, remove sample from bath and place in a circulating water bath with the temperature set to $25 \pm 0.5^\circ\text{C}$ ($77 \pm 1^\circ\text{F}$) according to 11.2.1 or 11.2.2. The sample shall be completely liquid before filtration.

11.2.1 If the sample is completely liquid after the 16-h cold soak, heat the sample at $25 \pm 0.5^\circ\text{C}$ ($77 \pm 1^\circ\text{F}$) for 2 ± 0.25 h. Otherwise follow 11.2.2.

11.2.2 If the sample is solid or contains any solids or haze after the 16-h cold soak, heat the sample at $25 \pm 0.5^\circ\text{C}$ ($77 \pm 1^\circ\text{F}$) for 4 ± 0.25 h.

11.2.3 Verify that the sample is at $25 \pm 0.5^\circ\text{C}$ ($77 \pm 1^\circ\text{F}$) by dipping *without stirring or agitating the sample* with an RTD sensor or thermometer.

11.3 Filter the samples as quickly as possible upon removal from the warming bath once the desired warming time has elapsed according to 11.2.1 or 11.2.2, whichever applies.

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

11.5 Start the vacuum system. (The vacuum controls have to be preset to run the test correctly. Since vacuum cannot be achieved with an open filter, a dry run with biodiesel may be used to set the controls when manual needle valves and analog gauges are used. Automatic digital vacuum controllers can be used to preset the final vacuum/pressure.)

11.6 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 for about 2-3 s to dislodge any particles that might have adhered to the walls of the container.

11.6.1 Take care not to shake the sample vigorously after the cold soak as this could cause some of the solids to go back into solution.

11.7 Immediately after swirling, remove cap and 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.

11.7.1 The *entire contents* of the sample container is defined as everything that will flow out of the sample container when it is poured into the filtration funnel and held for 10 s after the bulk of the sample has been poured out.

11.7.2 Record the vacuum in the system within 1 min of starting the filtration. The vacuum shall be between 70 to 85 kPa (21 and 25 in. of Hg). If the vacuum is not within the specified range, make adjustments to the vacuum system.

11.8 When filtration is complete, as evidenced by no sample remaining on the filter, immediately turn off the vacuum system and record the duration of the filtration, rounding to the nearest second.

11.9 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.

12. Report

12.1 Report the time for the 300 mL B100 to be completely filtered as B100 CSFT time by ASTM D7501 in seconds to the nearest second.

12.2 If the filtration of the 300 mL failed to be completed after 720 s, report the result as “B100 CSFT time greater than 720 s by ASTM D7501,” and report the volume which was filtered in 720 s in mL to the nearest mL.

13. Precision and Bias⁴

13.1 *Precision*—The formal precision of this test method for B100 filtration is given below. The 2009 ILS on which this precision is based included samples with mean CSFT results from 77.5 to 188.6.

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, would in the long run, in the normal and correct operation of this test method, exceed $0.2959(X - 4.5000E+01)$ time(s) only in one case in twenty. The degrees of freedom for this estimate are 55. Repeatability for samples falling outside the range of the ILS samples may be estimated with the above equation, but will represent an extrapolation of the repeatability relationship. The actual repeatability may deviate from these estimates.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1672.

TABLE 1 Precision Range from 2009 CSFT ILS

TABLE 1A				
from ILS data in accordance with Practice D6300				
Time, s	80	120	160	200
Repeatability, r	10	22	34	46
Reproducibility, R	19	40	62	83

TABLE 1B					
extrapolated, not compliant with Practice D6300					
Time, s	240	280	320	360	400
Repeatability, r	58	70	81	93	105
Reproducibility, R	105	127	148	170	191

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 would, in the long run, in normal and correct operation of this test method, exceed $0.5387(X - 4.5000E+01)$ time(s) only in one case in twenty. Reproducibility for samples falling outside the range of the ILS samples may be estimated with the above equation, but will represent an extrapolation of the reproducibility relationship. The actual reproducibility may deviate from these estimates. The degrees of freedom for this estimate are 28.

13.1.2.1 The degrees of freedom associated with the reproducibility estimate from this round robin study are 28. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual reproducibility may be significantly different than this estimate.

13.1.2.2 Based on the above, the precision estimates from the 2009 CSFT ILS for the specification limits from the ranges of 120 to 400 s would be as shown in Table 1. This Table is provided as a convenience for the user of the test method. Both r and R above 200 seconds are estimates that do not conform to the requirements of Practice D6300.

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.

14. Keywords

14.1 biodiesel; biodiesel blend; diesel fuel; filter blocking potential; glass fiber filter; laboratory filtration; low temperature operability; middle distillate fuel

APPENDIX

(Nonmandatory Information)

X1. PRECISION AND BIAS FROM THE 2007 COLD SOAK FILTRATION TEST (CSFT) RESEARCH REPORT (ILS0328)

X1.1 Precision⁵

X1.1.1 The reproducibility for this test method for B100 filtration has not yet been determined.

X1.1.2 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, 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.

X1.1.3 *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.

X1.1.4 *Interim Precision*—Repeatability and reproducibility determinations were made using data from the ASTM Biodiesel Low Temperature Operability Task Force (see Table X1.1.) The analysis of this data from ILS0328 is the subject of Research Report RR:D02-1649.⁵ 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.

NOTE X1.1—Interim precision repeatability and reproducibility determinations were made using data from the ASTM Operability Task Force. The degree of freedom associated with the repeatability/reproducibility estimate from this round robin study is 25 for repeatability and 10 for reproducibility. 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.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1649.

TABLE X1.1 Precision Range from 2007 Operability TF Data

	160 s	200 s	240 s	280 s	320 s	360 s
Repeatability	27	34	41	47	54	61
Reproducibility	93	116	139	162	185	208

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7501–12) that may impact the use of this standard.

- (1) Additional clarification added to **6.1.6**.
- (2) Temperature tolerance changed for consistency in **11.2**.
- (3) Additional clarification added to **11.5**.
- (4) Text in original 11.5 moved to **11.7.2** for better clarity.

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