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Methods of testing petroleum and associated products

Cold soak filter blocking tendency of biodiesel (B100)

ICS 77.060



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Cold soak filter blocking tendency of biodiesel (B100)

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Methods of testing petroleum and associated products

Cold soak filter blocking tendency of biodiesel (B100)

1 Scope

This method evaluates the cold soak filter blocking tendency (CSFBT) of biodiesel (B100).

Minor components of some biodiesel esters, including saturated monoglycerides, can separate above the cloud point of a biodiesel fuel blend. The CSFBT quantifies the propensity of these materials to separate from a blend of biodiesel and isoparaffinic solvent after a cold soak cycle.

The testing and evaluation of a product against this method may require the use of materials and equipment that could be hazardous. This document does not purport to address all the safety aspects associated with its use. Anyone using this method has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any applicable regulatory requirements prior to its use.

2 Normative references

The following normative documents contain provisions that, through reference in this text, constitute provisions of this method. The referenced documents may be obtained from the sources noted below.

NOTE The addresses provided below were valid at the date of publication of this standard.

An undated reference is to the latest edition or revision of the reference or document in question, unless otherwise specified by the authority applying this method. A dated reference is to the specified revision or edition of the reference or document in question. However, parties to agreements based on this method are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below.

2.1 Canadian General Standards Board (CGSB)

CAN/CGSB-3.0 — *Methods of testing petroleum and associated products*

No. 28.8 — *Visual haze rating of distillate fuel oils*

CAN/CGSB-3.524 — *Biodiesel (B100) for blending in middle distillate fuels.*

2.1.1 Source

The above may be obtained from the Canadian General Standards Board, Sales Centre, Gatineau, Canada K1A 1G6. Telephone 819-956-0425 or 1-800-665-2472. Fax 819-956-5740. E-mail ncr.cgsb-ongc@tpsgc-pwgsc.gc.ca. Web site www.tpsgc-pwgsc.gc.ca/ongc-cgsb.

2.2 Alberta Innovates Technology Futures

2012/2013 Inter-laboratory Study (ILS) for cold coak filter blocking tendency draft CAN/CGSB-3.0 No. 142.0

2.2.1 Source

The above may be obtained from Alberta Innovates Technology Futures, 250 Karl Clark Road, Edmonton, Alberta T6N 1E4. Telephone 780-450-5111. Fax 780-450-5333. Web site www.albertatechfutures.ca

2.3 ASTM International

Annual Book of ASTM Standards (Annex A)

2.3.1 Source

The above may be obtained from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, U.S.A., telephone 610-832-9585, fax 610-832-9555, Web site www.astm.org, or from IHS Global Canada Ltd., 200-1331 MacLeod Trail SE, Calgary, Alberta T2G 0K3, telephone 613-237-4250 or 1-800-267-8220, fax 613-237-4251, Web site www.global.ihs.com.

3 Summary of test method

3.1 A sample of biodiesel is first conditioned to erase its thermal history.

3.2 Then a blend of 20% by volume of this biodiesel sample and a specified isoparaffinic solvent is prepared and “cold soaked” at 1°C for 16 h.

3.3 The 20% blended sample is then warmed to 25°C for 2-4 h.

3.4 After warming, the “cold soaked” 20% blended sample is then passed at a constant rate of flow (20 mL/min) through a glass fibre filter medium (1.6 µm pore size).

3.4.1 The pressure drop across the filter is monitored until 300 mL of the 20% blend has passed through the filter, or

3.4.2 If a maximum pressure drop of 105 kPa is reached before the total volume of 20% blend is filtered, the actual volume filtered at the time of maximum pressure drop is recorded and used to calculate the CSFBT result.

3.5 Results of the CSFBT test can range from 1 for a biodiesel with very good filterability (essentially no separated materials under the test conditions), to more than 10 for a fuel with poor filterability (a relatively high level of separated materials under test conditions).

4 Terms and definitions

For the purposes of this method, the following terms and definitions apply.

4.1

20% blend

a blend of 20% by volume biodiesel with 80% by volume standard isoparaffinic solvent.

4.2

biodiesel (B100)

mono-alkyl esters of long-chain fatty acids derived from renewable sources. In its neat form, biodiesel is commonly designated as B100. In the context of this test method, the biodiesel is intended as a blending component in a finished diesel fuel or heating oil.

4.3

cold soak filter blocking tendency (CSFBT)

a dimensionless value that defines the tendency of a biodiesel fuel to plug or block a filter, based on pressure increase or volume filtered. See 12.1 for the equations to calculate the CSFBT number.

**4.4
middle distillate fuel**

a fuel boiling in the range of 130-400°C composed of hydrocarbons, including naturally occurring, petroleum-derived, non-hydrocarbons.

**4.5
standards isoparaffinic solvent**

a dearomatized heavy naphtha, isoparaffinic solvent as specified in 7.1.

5 Interferences

Water and inorganic sediment can interfere with the measurement of CSFBT, however biodiesel samples that meet the requirements of CAN/CGSB-3.524 generally will not have interference from water and sediment. High particulate contamination of the biodiesel sample as measured by ASTM D7321 can increase the CSFBT test result. If desired, ASTM D6304 may be used to determine the dissolved water content.

6 Apparatus

6.1 The apparatus is shown as a diagram in Figure 1. It is available as a commercial instrument or may be assembled from the following parts:

6.1.1 Piston pump: capable of delivering and maintaining the 20% blend or standard isoparaffinic solvent at a constant flow rate of 20 ± 1 mL/min.

6.1.2 Pulse damper: a mechanism to maintain constant flow of the 20% blend or standard isoparaffinic solvent through the filter until up to the maximum pressure of the test (105 kPa).

6.1.3 Pressure gauge: gauge or equivalent pressure measuring device calibrated and graded covering the range 0 – 150 kPa pressure, with an accuracy of ± 1 kPa.

6.1.4 Pressure relief valve: used to relieve the pressure if the filter becomes blocked and during verification of the pressure gauge. Set the relief pressure to a minimum of 125 kPa.

6.1.5 Filter unit/media: glass fibre filter, nominal pore size 1.6 μm , 13 mm diameter. Whatman Puradisc 13 mm diameter GF/A glass microfiber syringe filters are required (Part No. 6820-1316 or 6806-1316). Whatman Puradisc syringe filter orientation is shown in Figure 2.

NOTE Batch-to-batch variability of filter units may be reduced by Stanhope-Seta certified Whatman Puradisc filters (part No. 91616-00).

6.1.6 Fuel collection container: used to collect fuel from the pump outlet.

6.1.7 Temperature measuring device: with a range that includes 0 to 60°C, and an accuracy of $\pm 1^\circ\text{C}$.

6.1.8 Measuring cylinder: nominal capacity 25 mL, glass with graduations every 0.5mL, for verifying the flow rate.

6.1.9 Measuring cylinder: nominal capacity 500 mL, glass with graduations every 5 mL, for verifying the flow rate, and measuring the volume of fuel in the fuel collection container.

6.1.10 Filter adaptor: an adaptor is required to connect the tapered side of the filter (see 6.1.5) to the filtration apparatus outlet.

6.1.11 Electrical grounding:

6.1.11.1 Ground/bond wire: 0.912-2.59 mm (#10-#19) bare stranded flexible, stainless steel or copper installed as described below.

6.1.11.2 The fuel inlet reservoir is placed on a grounded surface for the duration of the test.

6.1.11.3 The fuel inlet tube is grounded. If the tube is non-conductive, a grounding wire is placed at the tube inlet opening and along the length of the tube until it exits the inlet reservoir (see Figure 1).

6.1.11.4 The fuel outlet tube is grounded. If the tube is non-conductive, a grounding wire is placed along the tube (see Figure 1).

6.1.11.5 An electrically grounded wire is used to connect the filter outlet to the bottom of the fuel receiver such that the fuel will flow along the wire (see Figure 1).

6.1.12 Stopwatch: manual or electronic, nominal accuracy 1 s, required for verifying the flow rate, sample shaking time, and sample standing time.

6.1.13 Bath: liquid circulating bath or forced air chamber capable of sustaining a temperature of $1 \pm 1.0^\circ\text{C}$ for at least 16 h and able to accommodate the required number of 500 mL sample containers. A commercial appliance grade refrigerator is incapable of meeting this requirement.

6.1.14 Liquid circulating bath: capable of sustaining a temperature of $25 \pm 1.0^\circ\text{C}$ for 4 h and able to accommodate the required number of 500 mL sample bottles. Alternatively the same bath as in 6.1.13 may be used if it meets the requirements of this section.

6.1.15 20% Blend fuel inlet reservoir: 500 mL clear glass bottle (see 6.2) or 500 mL glass beaker.

6.2 Sample container: 500 mL in volume, for the coal soak conditioning step, clear glass with a screw-on cap having an inert liner (see 11.1).

6.2.1 Clear glass containers facilitate a visual inspection of the contents and the container before and after filling. Clear glass containers also allow for visual inspection of the container, after the sample is emptied, to confirm complete emptying of the container. Container should be rinsed with approximately 50 mL of the specified isoparaffinic solvent or similar clean solvent prior to use.

6.2.1.1 Protect biodiesel samples in clear glass containers from excessive exposure to light, which can lead to photo-induced degradation. Avoid plastic containers from which esters can extract antioxidants or plasticizers.

6.3 Filter media: 0.45 μm nylon, for filtering standard isoparaffinic solvent (see 7.1).

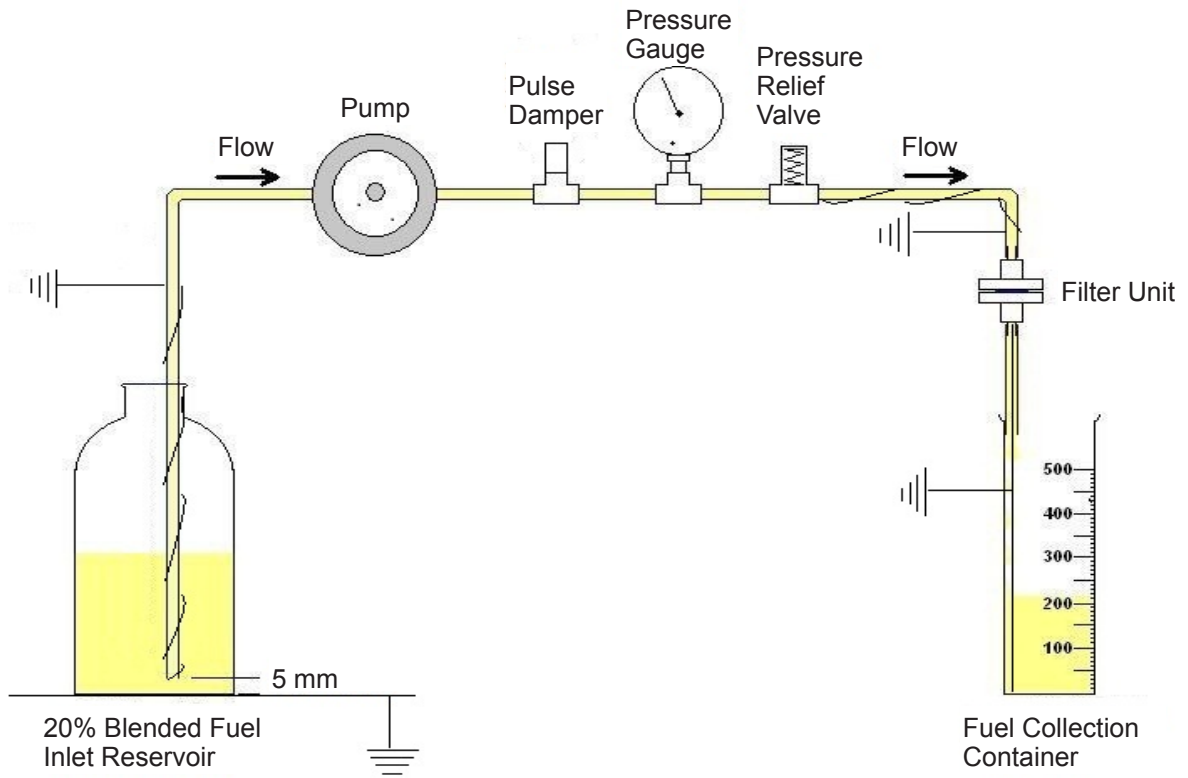


Figure 1 — Flow diagram of filtration test apparatus

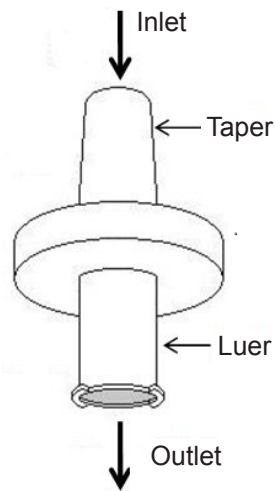


Figure 2 — Whatman Puradisc syringe filter orientation

7 Reagents

7.1 Standard isoparaffinic (see 4.5)

An isoparaffinic solvent meeting the following specifications is suitable for this test.

Property	Test method	Minimum	Maximum
Appearance	CAN/CGSB-3.0 No. 28.8 OR ASTM D4176	Clear and bright	
Aromatics, volume%	ASTM D1319		0.25
Boiling range, °C	ASTM D86	171	210
Flash point, °C	ASTM D93	43	
Sulphur, mg/kg	ASTM D5453		1
Water, mg/kg	ASTM D6304, Procedure A		105
Kinematic viscosity @ 25°C, mm ² /s	ASTM D445	1.5	2.1
NOTE Suitable solvents meeting these requirements are ISOPAR K, ISOPAR L, SHELLSOL OMS, and ASHPAR L.			

7.1.1 Filter the solvents through a 0.45 µm nylon filter. Use only solvent that has reached a temperature of 20-25°C.

8 Precautions

8.1 Follow the manufacturer's instructions to ensure the filtration apparatus is properly bonded and grounded. Refer to ASTM D4865 *Standard Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems*. As a minimum, the sample inlet and sample outlet shall be grounded.

8.2 Pressure increases in the apparatus as the test filter becomes plugged. A pressure relief valve is in place. Monitor pressure rise and do not allow increasing pressure beyond that required by the test and safe limit of the apparatus.

8.3 Do not test gasoline or lighter distillates that would present a hazard due to their higher volatility and lower flash point (<40°C).

8.4 Use appropriate solvent-resistant gloves and minimize exposure by working within a well ventilated area.

8.5 Use cut-resistant gloves for handling glassware at risk of breaking.

8.6 Biodiesel soaked cleaning materials (rags and similar material) can spontaneously combust. Take appropriate precautions for the timely and safe disposal of such waste material. This includes washing rags with soap and water and drying in a ventilated area before disposal.

9 Apparatus preparation

9.1 Pressure and temperature: Verify the pressure gauge reading, at 0 kPa (ambient atmospheric pressure) and 100 kPa, at least every 6 months. Verify the temperature reading devices (digital or glass thermometer), at 25°C and at 0°C for the bath at least once a year.

9.2 Flow rate and system pressure check: Verify monthly that the flow rate is 20 mL ± 1 mL/min through a filter assembly installed according to 11.7, measuring the volume of standard solvent pumped and recording the pressure at 20 s, and then after every 60 s during a 15-minute period. The measured volume should be between 285 and 315 mL. The pressure should be 5 ± 2 kPa. Flow rate may be checked more frequently by measuring the volume pumped during a 1-minute period. In this case, the volume should be between 19 and 21 mL. Adjust the flow control on the pump following manufacturer's instructions if flow rate checks are outside 20 mL ± 1 mL/min.

9.3 Apparatus assembly: Assemble the apparatus as shown in Figure 1 without the filter unit connected.

10 Sampling

10.1 The sample shall be obtained in accordance with Practices ASTM D4057, ASTM D4177 or equivalent site-specific practices.

10.1.1 Precautions to avoid sample contamination shall include selection of an appropriate sampling point. It is preferable 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 product before collecting the biodiesel sample.

10.1.2 Use clean sample containers that are impervious to biodiesel. Flush the containers three times with the product to be sampled before collecting the sample.

10.1.3 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, obtain a new sample.

10.1.4 Where it is desirable or only possible to obtain samples from static storage, follow the procedures given in ASTM 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.

10.1.5 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.

10.1.6 If any free water is visually apparent (as may be determined by test methods ASTM D4176 or CAN/CGSB-3.0 No. 28.8), discard and replace with fresh sample.

10.1.7 Where possible, store the biodiesel sample under an inert atmosphere.

10.2 Heat the entire biodiesel sample, preferably under an inert atmosphere, to 60°C for 3 h before analysis to remove all thermal history and ensure that any separated materials are dissolved in the sample.

NOTE Ruggedness testing by several laboratories has shown that sample conditioning all biodiesel according to 10.2 minimized discrepancies in results.

10.2.1 To minimize degradation, do not heat the biodiesel unnecessarily, especially if the sample cannot be placed under an inert atmosphere.

10.2.2 After heating at 60°C for 3 h, allow the biodiesel to cool down to 24 ± 3°C. A liquid bath at 20 to 25°C may be used.

10.2.2.1 This sample conditioning step may be omitted if the biodiesel sample was collected from a heated tank at a production facility. In this case, proceed directly to 11.1.

10.2.2.2 If there is any doubt about the thermal history of the biodiesel sample, the conditioning step (see 10.2) shall be performed.

11 Procedure

11.1 Shake the biodiesel vigorously for one minute. Aliquot 70 mL into a 500 mL clear glass container (see 6.2) and heat the container in an oven or bath at $60 \pm 1^\circ\text{C}$ for 45 min. Allow to cool to $24 \pm 4^\circ\text{C}$.

11.1.1 This heating may be omitted if step 10.2.2 was performed within 10 h or if 10.2.2.1 applies.

11.1.2 The 60°C heating is intended to dissolve separated materials that form in the biodiesel at room temperature. If the biodiesel has been cooled to below 20°C then repeat 10.2 before starting the procedure.

11.2 Add 280 mL of standard isoparaffinic solvent (see 7.1) to the 70 mL of biodiesel to make a 20% blend by volume of biodiesel and 80% by volume standard isoparaffinic solvent. Ensure a homogeneous solution by mixing.

11.3 Put the container in a liquid bath at $1 \pm 1^\circ\text{C}$ for 16 ± 1 h.

11.4 Remove the 20% blend from the bath. If any free (undissolved) water is apparent in the fuel at this stage, abandon the test and report the presence of water.

11.5 Swirl gently and place the 20% blend in a circulating bath $25 \pm 1^\circ\text{C}$ for 2 ± 0.25 h. Alternatively the same bath may be used if the setting is changed to 25°C immediately after swirling. If the 20% blend contains any solids or haze after the 2-hour warming period, shake vigorously for 20 ± 10 s and continue warming for an additional 2 h. If solids or haze are still visible after a total of 4-hour warming, then report "solids or haze visible" and continue with the test.

11.5.1 Remove the 20% blend from the $25 \pm 1^\circ\text{C}$ bath and perform the rest of section 11 as soon as possible and within 2 h.

11.5.2 Shake the 20% blend vigorously for 60 ± 10 s and pour into the blend fuel inlet reservoir (see 6.1.15). Alternatively the bottle used for the cold soak conditioning may be used as the inlet reservoir. Place the reservoir on an electrically grounded surface for the remainder of the test.

11.6 Place the pump suction pipe into the 20% blend such that the opening is approximately 5 mm above the bottom of the pump inlet reservoir. Allow the sample to stand on a vibration free surface for 120 ± 15 s prior to filtration. Run the pump without the filter attached until the 20% blend flows into the collection container. Collect approximately 20 mL of 20% blend. Stop the pump and discard any fuel from the collection container. Ensure there is no air in the lines prior to attaching filter unit.

11.7 Attach the tapered end of the filter unit to the adaptor (see 6.1.10) on the system.

11.7.1 Restart the pump and start the timer.

NOTE An adaptor constructed with flexible material, such as plastic tubing or luer adaptor, is prone to deformation or cracking after repeated use. Change frequently, and ensure a proper pressure seal is made, such as by performing the leak test (see 11.10).

11.8 Record the pressure after 20 s. The pressure shall be equal or greater than the system pressure check (see 9.2). A lower pressure could be due to a leak in the filter media. Record the pressure after 60 s and thereafter

every 60 s until the end of the test¹. When the pressure rises to 105 kPa, stop the pump immediately. Do not allow additional sample to flow into the collection container, as that could affect the volume result. Measure and record the volume collected, rounding off the figure to the nearest 10 mL.

11.9 When 300 mL of 20% blend has been pumped without the pressure of 105 kPa, stop the pump. Record the maximum pressure reached, to at least the nearest 5 kPa.

11.10 If the graph in 12.2 indicates an abnormal curve, then before dismantling and cleaning, check for leaks within the system which could have affected the test result.

11.10.1 Do not remove the filter unit used in the test from the system. Leave the fuel inlet and collection reservoirs in place.

11.10.2 Plug the outlet of the filter, and pump liquid until a pressure of approximately 105 kPa is reached.

11.10.3 Turn off the pump and observe the pressure. The pressure should not drop more than 3 kPa during a 30 s interval.

11.11 Pump at least 50 mL of filtered standard isoparaffinic solvent (see 7.1) through the lines of the filter plugging apparatus to remove contamination that could cause plugging on the next sample.

11.12 Apparatus cleanliness and restrictiveness can be checked by attaching a filter as described in 11.6 and running the pump with standard isoparaffinic solvent. The pressure should be within the system pressure check value (see 9.2).

12 Calculations

12.1 Calculate the CSFBT by either of the following equations:

$$\text{CSFBT} = \sqrt{1 + (P / 105)^2} \quad (1) \text{ When 300 mL of the 20\% blend has passed through the filter and the pressure did not reach 105 kPa.}$$

$$\text{CSFBT} = \sqrt{1 + (300 / V)^2} \quad (2) \text{ When the pressure reached 105 kPa and the test was discontinued.}$$

P is the maximum pressure reading obtained for 300 mL of 20% blend to pass the filter (kPa).

V is the volume of 20% blend passed through the filter before the pressure reaches 105 kPa (mL).

12.2 For a CSFBT results greater or equal to 1.07 (≥ 40 kPa maximum pressure) plot the pressure versus volume pumped data on a graph similar to that in Figure 3 (see 11.8).

12.2.1 The points should fit an easily defined continuously increasing smooth curve with linear or positively curved slope (see Figure 3).

12.2.2 Particularly in the region $P \geq 40$ kPa, a plateau or drop in pressure ($P_v \geq P_{v+20\text{ml}}$), or pronounced negative curvature, could indicate a leak in the system or apparent filter breakthrough. Verify the instrument is working properly and return to check result.

¹ Automated data collection may be suitable for this step.

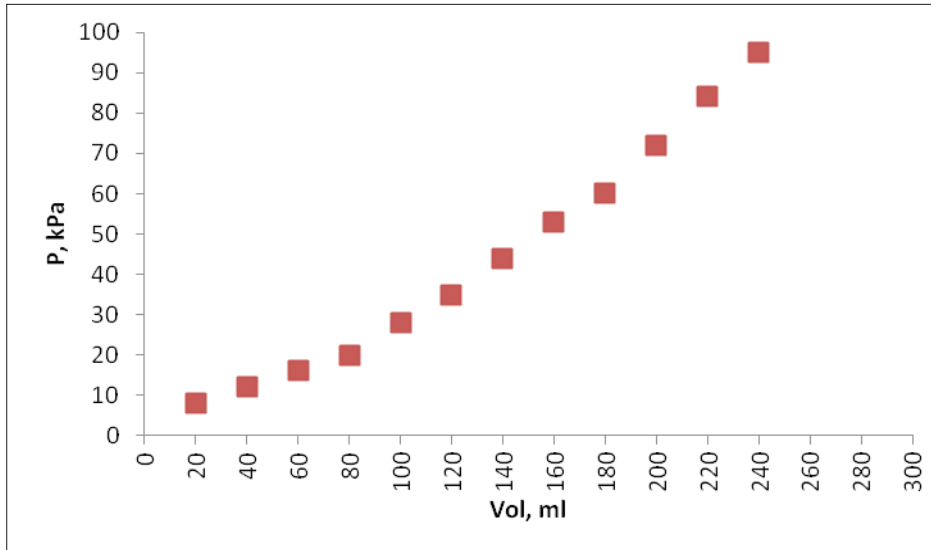


Figure 3 — Filter plugging pressure vs volume pumped

13 Report

13.1 Report the results of the test in one of the following forms, referencing this test method:

13.1.1 If free water is found (see 11.4), report “Free (undissolved) water present, test not performed”.

13.1.2 If solids or haze are visible (see 11.5) after 4 h of warming, report “solids or haze visible” and the CSFBT result.

13.2 Report the following:

- CSFBT to the nearest 0.1 as calculated in 12.1.
- Maximum pressure (P) reading (see 11.8) obtained for 300 mL of fuel to pass the filter, rounded to the nearest 5 kPa.
- Volume of fuel (V) passed at a pressure reading of 105 kPa (see 11.8), rounded to the nearest 10 mL.

14 Precision and bias

14.1 The precision of the method was developed in a 2012 interlaboratory test program involving 13 laboratories and ten test samples (see 2.2.1). Each sample was analyzed twice. Commercial and site-assembled instrument types were used. Repeatability and reproducibility analysis for all instruments as one group was analyzed in accordance with ASTM D6300. Precision and bias between instrument types was analyzed in accordance with ASTM D6708.

14.1.1 One set of sample data was excluded due to essentially zero variation from 1.0 CSFBT.²

² One sample gave results of 1.0 by all laboratories, so there was no variation, and current statistical treatment of interlaboratory test data requires some variation. This data set was excluded because it gave excellent, unvarying results, not because it was poor or suspect data.

14.1.2 Repeatability

Duplicate results by the same operator should be considered suspect if they differ by more than the amount given by the equation $(r) = 0.284*(X)^{1.67}$, which is valid over the range $>1.0 - 3.5$, where $X = \text{CSFBT result}$.

14.1.3 Reproducibility

Results submitted by each of two laboratories should be considered suspect if they differ by more than the amount given by the equation $(R) = 0.49*(X)^{1.67}$, which is valid over the range $>1.0 - 3.5$, where $X = \text{CSFBT result}$.

14.1.4 Agreement between instrument types

There is no statistically distinguishable difference in precision performance between the commercial and site-assembled instrument types. There is a small but distinguishable constant bias between the two types, with the commercial type higher by an average of 0.17 CSFBT.

Annex A (normative)

Referenced ASTM publications (see 2.3)

Annual Book of ASTM Standards

D86 — Standards test method for distillation of petroleum products at atmospheric pressure

D93 — Standards test method for flash point by Pensky-Martens closed cup tester

D445 — Standard test method for kinematic viscosity of transparent and opaque liquids (and calculation of dynamic viscosity)

D1319 — Standard test method for hydrocarbon types in liquid petroleum products by fluorescent indicator adsorption

D4057 — Standard practice for manual sampling of petroleum and petroleum products

D4176 — Standard test method for free water and particulate contamination in distillate fuels (visual inspection procedures)

D4177 — Standard practice for automatic sampling of petroleum and petroleum products

D4865 — Standard guide for generation and dissipation of static electricity in petroleum fuel systems

D5453 — Standard test methods for determination of total sulfur in light hydrocarbons, spark ignition engine fuel, diesel engine fuel, and engine oil by ultraviolet fluorescence

D6300 — Standard practice for determination of precision and bias data for use in test methods for petroleum products and lubricants

D6304 — Standard test method for determination of water in petroleum products, lubricating oils, and additives by coulometric Karl Fischer titration

D6708 — Standard practice for statistical assessment and improvement of expected agreement between two test methods that purport to measure the same property of a material

D7321 — Standard test method for test method for particulate contamination of biodiesel B100 blend stock. Biodiesel esters and biodiesel blends by laboratory filtration