Designation: D7321 − 16a

Standard Test Method for Particulate Contamination of Biodiesel B100 Blend Stock Biodiesel Esters and Biodiesel Blends by Laboratory Filtration[1]

This standard is issued under the fixed designation D7321; 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 of the mass of particulate contamination in B100 biodiesel in accordance with Specification D6751 and BXX blends that are prepared against all No. 1 and No. 2 grade fuels allowed within Specifications D396 and D975.

NOTE 1—Middle distillate fuels with flash points less than 38 °C have been ignited by discharges of static electricity when the fuels have been filtered through inadequately bonded or grounded glass fiber filter systems. See Guide D4865 for a more detailed discussion of static electricity formation and discharge.

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

1.3 *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]

D396 Specification for Fuel Oils
D975 Specification for Diesel Fuel Oils
D1193 Specification for Reagent Water
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

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.

3.1.2 *biodiesel blend, BXX, n*—a fuel composed of biodiesel blendstock with hydrocarbon-based diesel fuel.

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

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *filtered flushing fluids, n*—any of three solvents, methanol, propan-2-ol (isopropanol), or 2,2,4-trimethylpentane, filtered through a nominal 0.45 µm filter.

3.2.2 *glass fiber filter, n*—glass fiber filter used in this test method.

4. Summary of Test Method

4.1 *B100 Biodiesel Blend Stock Filtration:*

4.1.1 A measured volume of 400 mL to 450 mL of biodiesel ester (B100) is vacuum filtered through one 0.7 µm glass fiber filter. If upon visual inspection particulate contamination is observed, this may result in a high filtration time. In such cases, when the contamination is high or of a nature that induces slow filtration rates, two or more filtrations using a fresh filter each time may be required to complete filtration in a reasonable time.

4.1.2 After the filtration has been completed, the filter is washed with solvent, dried, and weighed. The particulate contamination level is determined by the mass gained by the glass fiber filter, and is reported in units of grams per cubic metre or its equivalent in milligrams per litre.

4.2 *BXX Biodiesel Blend Filtration:*

4.2.1 A measured volume of 800 mL to 900 mL of biodiesel blend (BXX) is vacuum filtered through one 0.7 µm glass fiber filter. If upon visual inspection particulate contamination is observed, this may result in a high filtration time. In such cases, when the contamination is high or of a nature that induces slow

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[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.

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

filtration rates, two or more filtrations using a fresh filter each time may be required to complete filtration in a reasonable time.

4.2.2 After the filtration has been completed, the filter is washed with solvent, dried, and weighed. The particulate contamination level is determined by the mass gained from the glass fiber filter, and is reported in units of grams per cubic metre or its equivalent in milligrams per litre.

5. Significance and Use

5.1 The mass of particulates present in a fuel is a significant factor, along with the size and nature of the individual particles, in the rapidity with which fuel system filters and other small orifices in fuel systems can become plugged. This test method provides a means of assessing the mass of particulates present in a fuel sample.

5.2 The test method can be used in specifications and purchase documents as a means of controlling particulate contamination levels in the fuels purchased.

6. Apparatus

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

6.1.1 *Funnel and Funnel Base,* with filter support for a 47 mm diameter glass fiber, and locking ring or spring action clip.

6.1.2 *Ground/Bond Wire,* 0.912 mm 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. 1.

NOTE 2—The electrical bonding apparatus shown in Fig. 1 or other suitable means of electrical grounding that 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 since copper ions catalyze gum formation during the stability test.

6.1.3 *Receiving Flask,* 1.5 L or larger borosilicate glass vacuum filter flask, into which the filtration apparatus fits, equipped with a sidearm to connect to the safety flask.

6.1.4 *Safety Flask,* 1.5 L or larger borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistance 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.

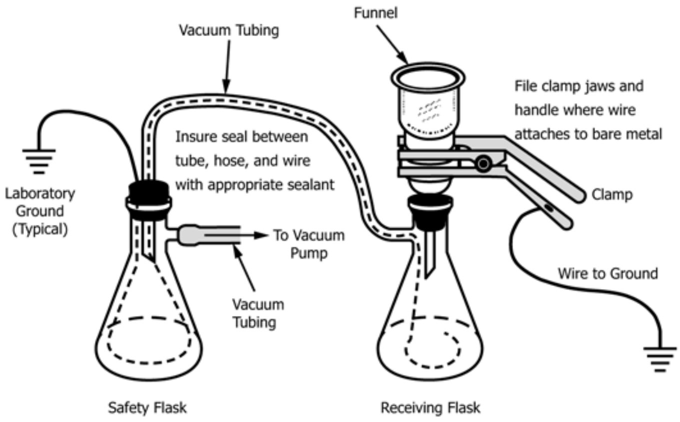

FIG. 1 Schematic of Filtration System

6.1.5 *Vacuum System,* either a water aspirated or a mechanical vacuum pump may be used if capable of producing a vacuum of 1 kPa to 100 kPa below atmospheric pressure when measured at the receiving flask.

6.2 *Other Apparatus:*

6.2.1 *Air Ionizer,* if used for the balance case. Air ionizers if used should be replaced annually.

NOTE 3—When using a solid-pan balance, the air ionizer may be omitted provided that, when weighing a glass fiber filter, it is placed on the pan so that no part protrudes over the edge of the pan.

6.2.2 *Analytical Balance,* single- or double-pan, with the precision standard deviation of 0.07 mg or less.

6.2.3 *Crucible Tongs,* for handling clean sample container lids.

6.2.4 *Drying Oven,* naturally convected (without fan-assisted air circulation), controlling to 90 °C ± 5 °C.

6.2.5 *Flushing Fluid Dispenser,* an apparatus for dispensing flushing fluid through a nominal 0.45 µm filter.

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

6.2.7 *Graduated Cylinders,* to contain at least 1 L of fluid and marked at 10 mL intervals. For samples that filter slowly, 100 mL graduated cylinders may be required.

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

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

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.[3] 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 *Purity of Water*—Unless otherwise indicated, references to water mean reagent water as defined by Types I, II and III of Specification D1193.

7.3 *Flushing Fluids:*

7.3.1 *Methanol,* (**Warning—** Flammable).

7.3.2 *2,2,4-trimethylpentane (isooctane),* (**Warning—** Flammable).

7.3.3 *propan-2-ol (2-propanol; isopropyl alcohol),* (**Warning—**Flammable).

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

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[3] *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 *Analar 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.

7.5 *Glass Fiber Filter,* plain, with a diameter of 47 mm and a nominal pore size of 0.7 µm.

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

8. Sampling

8.1 *Sampling for Procedure for Biodiesel Blend Stock (B100):*

8.1.1 The sample container should be 500 mL (±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 rinsing 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 they are less desirable since visual inspection of the interior of the container is more difficult.

8.1.1.1 It is imperative that the *entire* contents of the sample container are filtered during the B100 filtration. This includes not only all of the fuel but also all rinsings of the interior of the container with flushing fluid. Because of this, take care to protect the sample from any external contamination.

8.1.2 If it is not possible to sample in a 500 mL bottle, or the sample has already been received in a 1 L bottle, follow 8.1.2.1.

8.1.2.1 Vigorously shake the sample for 1 min, and transfer 400 mL to 450 mL (±15 mL) to a clean fresh 500 mL bottle. If a clean fresh 500 mL bottle is not available, use a clean fresh 1 L bottle.

8.1.3 Precautions to avoid sample contamination shall include selection of an appropriate sampling point. Samples should 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 taking the sample.

8.1.3.1 Where it is desirable or only possible to obtain samples from static storage, follow the procedures given in Practice D4057 or equivalent, taking precautions for cleanliness of all equipment used. Ensure that the sample has not passed through intermediate containers prior to placement in the prepared container.

NOTE 5—Samples obtained from static storage may 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.

8.1.4 Visually inspect the sample container before collecting the samples to verify that there are no visible particles present inside the container. Collect 800 mL to 900 mL (±15 mL), in a 1 L sample bottle or 400 mL to 500 mL (±15 mL) in a 500 mL (±15 mL) sample bottle and ensure that there is an ullage (vapor space) of at least 10 % of the container volume to allow for liquid expansion. 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 must be obtained.

8.1.5 Analyze fuel samples as soon as possible after sampling. When a fuel cannot be analyzed within one day, it should be blanketed with an inert gas such as oxygen-free nitrogen, argon, or helium. Store at a temperature no higher than 10 °C; samples with cloud points above 10 °C may be stored at room temperature.

8.2 *Sampling for Procedure for Biodiesel Blends (BXX):*

8.2.1 The sample container for BXX shall be 1 L (±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 rinsing 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 these are less desirable since visual inspection of the interior of the container is more difficult.

8.2.1.1 It is important to note that the *entire* contents of the sample container are filtered during the BXX filtration. This includes not only all of the fuel but also all rinsings of the interior of the container with flushing fluid. Because of this, take care to protect the sample from any external contamination. The expectation is that the volume in the 1 L sample bottle will be 800 mL to 900 mL, allowing for 10 % ullage.

8.2.2 Follow 8.1.3 – 8.1.5.

9. Preparation of Apparatus and Sample Containers

9.1 Clean all components of the filtration apparatus as described in 9.1.1 – 9.1.4.

9.1.1 Remove any labels, tags, and so forth.

9.1.2 Rinse apparatus thoroughly with propan-2-ol that has been filtered through a 0.45 µm glass fiber filter.

9.1.3 Rinse thoroughly with filtered flushing fluid and dry.

9.1.4 Keep a clean protective cover (the cover may be rinsed with filtered flushing fluid) 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.

9.1.5 Use clean sample containers.

10. Preparation of Glass Fiber Filter

10.1 For fuels containing little particulate materials, only one filter is required (as a control filter is not required). If the fuel is highly contaminated, more than one filter may be required (see Section 11). Clean all glassware used in preparation of glass fiber filter as described in Section 9.

10.2 Using forceps, place the glass fiber filter in a clean petri dish. To facilitate handling, the glass fiber filter should rest on clean glass support rods or watch glasses in the petri dish.

10.3 Place the petri dish with its lid slightly ajar, in a drying oven at 90 °C ± 5 °C, and leave it for 30 min.

10.4 Remove the petri dish from the drying oven, and place it near the balance. Keep the petri dish cover ajar, but such that the filter is still protected from contamination from the atmosphere. Allow 30 min for the filter to come to equilibrium with room air temperature and humidity.

10.5 Remove the control glass fiber filter from the petri dish with forceps, handling by the edge only, and place it centrally on the weighing pan of the balance. Weigh it, record the initial mass to the nearest 0.0001 g, and return it to the petri dish.

10.6 Place the filter on the funnel base and then 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.

11. Procedure

11.1 Thoroughly clean the outside of the sample container in the region of the cap by wiping it with a damp, lint-free cloth. Shake the container vigorously for about 1 min.

11.2 Remove the cap and remove any external contaminant that may be present in the threads.

11.3 Complete assembly of the receiving flask, preweighed filter, and funnel as a unit (see Fig. 1). To minimize operator exposure to fumes, the filtering procedure should be performed in a fume hood. **IMPORTANT**—The entire contents of the sample container shall be filtered through the glass fiber filter to ensure a correct measure of the particulate contamination in the sample.

NOTE 6—Some fuels may filter reasonably rapidly during transfer of the total contents of the sample container through a single glass fiber filter. However, some fuels, due to the quantity or nature of particulates, or both, may plug the filters during filtration and require use of multiple successive filtrations. To facilitate the latter, it is advisable to use smaller cleaned graduated transfer cylinders of 100 mL capacity.

11.4 Pour fuel from the sample container to the graduated cylinder, start the vacuum, and then transfer 100 mL of fuel to the filter funnel.

11.5 Continue transferring 100 mL increments of fuel to the filter funnel. When all the fuel from the sample container has been filtered, or if filtration slows so that 100 mL of sample requires greater than 10 min for complete filtration, then remove the filter support/filter funnel from the receiving flask, and pour the filtered fuel into a clean graduated cylinder and record the volume of fuel in mL that was filtered (V_f). Keep the fuel sample filtrate separate from the solvent washings filtrate. This allows the fuel to be used for additional analyses. If all the fuel has been filtered, thoroughly rinse the sample container and the graduated cylinder with one or more portions of filtered flushing fluid and pour the rinses into the funnel and proceed to 11.6. If all the fuel has not been filtered, then proceed to 11.6 and 11.8, and then repeat from 11.1.

11.6 Wash down the inside of the funnel and the outside of the joint between the funnel and filter base filtered with flushing fluid. Use two washes with approximately 50 mL each of washing flushing fluid. With the vacuum applied, carefully separate the funnel from the filter base. Wash the periphery of the glass fiber filter by directing a gentle stream of filtered flushing fluid from the edge to the center, exercising care not to wash any of the particulate from the surface of the glass fiber filter.

NOTE 7—The volume of the solvent required is recommended to be approximately 50 mL. However the rate of filtration may dictate the solvent volume. Take care not to wash particles from the filter particularly at the stage when the funnel has been removed.

11.7 The volume of the solvent used to wash the filter should be sufficient to rinse any particles on to the filter paper. A final wash of approximately 20 mL of methanol or isopropanol (propan-2-ol) is applied with the use of a pipette to remove trace of fuel, while taking care to minimize the risk of dislodging any particles on the filter. A Pasteur pipette has been found suitable for the final wash. Maintain vacuum after the final washing for 10 s to 15 s to remove excess filtered flushing fluid from the glass fiber filter.

11.7.1 If after washing and before placing in the drying oven to dry the filter, fuel is observed on the outer edges, gently rinse the filter with more solvent, rinsing gently from the outer edges inward, so as to not disturb any particulate matter filtered.

11.8 Using clean forceps, carefully remove the glass fiber filter from the filter base and place it on clean glass support rods or watch glass in a clean, covered petri dish. Dry and reweigh the glass fiber filter as described in 10.5, taking care not to disturb the particulate on the surface of the glass fiber filter. Record the glass fiber filter mass to the nearest 0.0001 g for each filtration.

11.9 If after removing the filter from the drying oven, the filter is observed to have discoloration on the filter indicating fuel on the outer edges, gently rinse the filter with more solvent, rinsing gently from the outer edges inward, so as to not disturb any particulate matter filtered, and repeat 11.8.

12. Calculation

12.1 The particulate contamination level is determined from the increase in the mass of the glass fiber filter and is reported in units of g/m^3 or its equivalent in mg/L.

12.2 If the entire fuel sample filtered through a single filter, then:

12.2.1 Calculate the mass on the glass fiber filter, M_{tm}, as $M_2 - M_1$, g.

M_2 = mass of the glass fiber filter after the filtration (11.8), and
M_1 = mass of the glass fiber filter before the filtration. (10.5).

12.2.2 Calculate total particulate contaminant in g/m^3 (mg/L) as follows:

$$[M_{tm}/V_f] \times 10^6 \tag{1}$$

where:

V_f = volume of fuel filtered, mL.

12.3 If the fuel sample required more than one glass fiber filter, then:

12.3.1 For each filter calculate the mass on the glass fiber filter, M_{tm}, as $M_{2(x)} - M_{1(x)}$, in g, where the subscripts 2 and 1 have the same meaning as in 12.2.1, and *x* indicates the number of the filtration.

12.4 Calculate the total contaminant mass and total volume of fuel filtered for each glass fiber filter as follows:

$$M_{tm(tot)} = M_{tm(1)} + M_{tm(2)} + \ldots + M_{tm(x)} \quad (2)$$

$$V_{tot} = V_{f(1)} + V_{f(2)} + \ldots + V_{f(x)} \quad (3)$$

$M_{tm(tot)}$ = total mass on glass fiber filter, g, and
V_{tot} = total volume of fuel filtered, mL.

NOTE 8—Subscripts 1 to *x* indicate the number of the filtration.

12.4.1 Calculate the total particulate contaminant in g/m^3 (mg/L) as follows:

$$\left[M_{tm(tot)}/V_{tot} \times 10^6\right] \quad (4)$$

13. Report

13.1 Report the particulate contamination to the nearest 0.1 g/m^3 (mg/L) and the volume of fuel filtered in m^3 (L).

13.2 Report the total particulate contamination to the nearest 0.1 g/m^3 (mg/L), the total volume of fuel filtered in cubic metres (litres), and the total number of filtrations (if more than one filter is required).

13.3 Report the B100 filtered as B100 particulate contamination as in g/m^3 (mg/L).

13.4 Report the BXX filtered as BXX particulate contamination as in g/m^3 (mg/L).

14. Precision and Bias

14.1 *Precision and Bias for B100 and BXX Biodiesel Blend Stock Filtration:*[4]

14.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material for B100 and BXX filtration would in the long run, in the normal and correct operation of this test method, exceed 0.9192 × $X^{0.5000}$ mg/L only in one case in twenty.

14.1.2 *Reproducibility*—The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material for B100 and BXX filtration would in the long run, in normal and correct operation of this test method, exceed 1.4625 × $X^{0.5000}$ mg/L only in one case in twenty.

NOTE 9—Table 1 shows precision for the range of 1 mg/L to 15 mg/L.

14.2 *Bias*—The procedure given for the determination of Test Method D7321 has no bias because the value of particulate contamination is defined in terms of this test method.

15. Keywords

15.1 biodiesel; biodiesel blend; diesel fuel; glass fiber filter; gravimetric determination; laboratory filtration; middle distillate fuel; particulate contamination

TABLE 1 Precision (1 mg/L to 15 mg/L)

Particulate Contamination, mg/L	1.0	2.0	3.0	4.0	5.0	10.0	15.0
Repeatability	0.92	1.30	1.59	1.84	2.06	2.91	3.56
Reproducibility	1.46	2.07	2.53	2.92	3.27	4.62	5.66

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7321 – 16) that may impact the use of this standard. (Approved July 1, 2016.)

(1) Revised definition in 3.1.2.

(2) Revised subsections 4.1.1, 4.2.1, 8.1.2.1, 8.1.4, and 8.2.1.1.

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7321 – 14) that may impact the use of this standard. (Approved June 1, 2016.)

(1) Revised subsections 4.1.1 and 4.2.1.

(2) Added new subsections 11.7.1 and 11.9.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1713.

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