



Designation: D7321 – 11<sup>ε1</sup>

# Standard Test Method for Test Method for Particulate Contamination of Biodiesel B100 Blend Stock Biodiesel Esters and Biodiesel Blends by Laboratory Filtration<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Editorial changes were made to Section 8 in July 2013.

## 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:<sup>2</sup>

- 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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

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*—blend of biodiesel fuel with petroleum based biodiesel 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  $\mu\text{m}$  filter.

3.2.2 *test 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 about 400 mL of biodiesel ester (B100) is vacuum filtered through one 0.7  $\mu\text{m}$  glass fiber filter. When the contamination is high or of a nature that induces slow filtration rates, two or more filtrations using a fresh test filter each time may be required to complete filtration in a reasonable time.

4.1.2 After the filtration has been completed, the test filter is washed with solvent, dried, and weighed. The particulate contamination level is determined by subtracting the blank filter mass before filtration from the mass gained by the test filter, and is reported in units of  $\text{g}/\text{m}^3$  or its equivalent in  $\text{mg}/\text{L}$ .

### 4.2 BXX Biodiesel Blend Filtration:

4.2.1 A measured volume of about 800 mL of biodiesel blend (BXX) is vacuum filtered through one 0.7  $\mu\text{m}$  glass fiber filter. When the contamination is high or of a nature that

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

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

4.2.2 After the filtration has been completed, the test filter is washed with solvent, dried, and weighed. The particulate contamination level is determined by subtracting the mass gain from the test filter, and is reported in units of  $\text{g}/\text{m}^3$  or its equivalent in  $\text{mg}/\text{L}$ .

## 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*, 0.912-2.59 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.

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 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  $90 \pm 5^\circ\text{C}$ .

6.2.5 *Flushing Fluid Dispenser*, an apparatus for dispensing flushing fluid through a nominal  $0.45 \mu\text{m}$  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 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.<sup>3</sup> 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**.

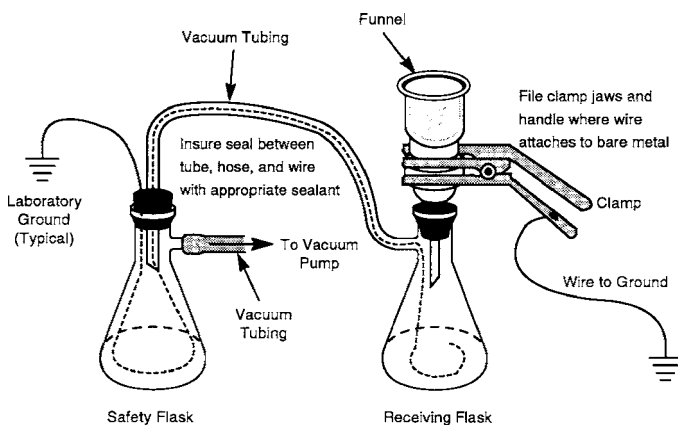
### 7.3 Flushing Fluids:

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

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

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

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



**FIG. 1 Schematic of Filtration System**

<sup>3</sup> *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.

7.6 *Test Glass Fiber Filter*, plain, 47-mm diameter, nominal pore size 0.7- $\mu\text{m}$ .

7.7 *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 ( $\pm 0.015$  L) 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 a clean fresh 500 mL ( $\pm 0.015$  L) bottle. If a clean fresh 500 mL ( $\pm 0.015$  L) bottle is not available, use a clean fresh 1 L ( $\pm 0.015$  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 taking the samples to verify that there are no visible particles present inside the container. Fill the sample container 90 volume % full, leaving space for vapor 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 ( $\pm 0.015$  L) 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, allowing for ullage, that the volume in the 1-L sample bottle will be 800 mL ( $\pm 0.015$  L).

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  $\mu\text{m}$  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. If the fuel is highly contaminated, more than one set of filters 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 test and glass fiber filter side by side 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  $90 \pm 5^\circ\text{C}$ , 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 test filter is still protected from contamination from the