Designation: D7321 – 21

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel

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 homogeneous mixture of hydrocarbon oils and mono-alkyl esters of long chain fatty acids.

3.1.2.1 *Discussion*—In the abbreviation BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.2.2 *Discussion*—The mono-alkyl esters of long chain fatty acids (that is, biodiesel) used in the mixture shall meet the requirements of Specification D6751.

3.1.2.3 *Discussion*—Diesel fuel, fuel oil, and non-aviation gas turbine oil are examples of hydrocarbon oils.

3.1.2.4 *Discussion*—B0 is synonymous with a hydrocarbon oil to which no biodiesel has been added.

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

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

[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, Cleanliness and Compatibility 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.

4.1.2 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 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.

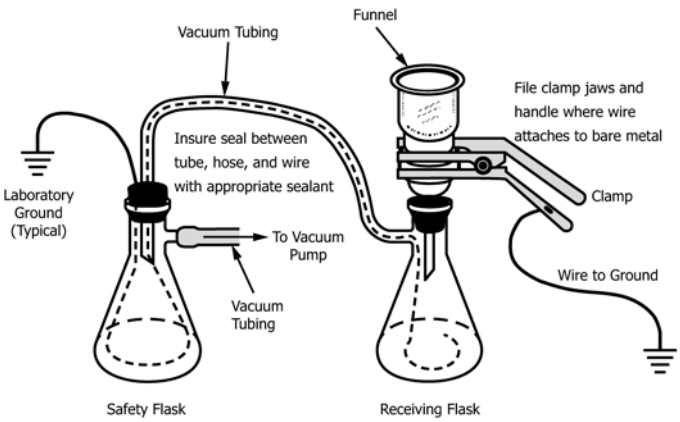

FIG. 1 Schematic of Filtration System

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.

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

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.