



Designation: D6217 – 21



Designation: 415/98

Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration^{1,2}

This standard is issued under the fixed designation D6217; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers the determination of the mass of particulate contamination in a middle distillate fuel by filtration. This test method is suitable for all No. 1 and No. 2 grades in Specifications [D396](#), [D975](#), [D2880](#) and [D3699](#) and for grades DMA and DMB in Specification [D2069](#).

1.2 This test method is not suitable for fuels whose flash point as determined by Test Methods [D56](#), [D93](#) or [D3828](#) is less than 38 °C.

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 membrane filter systems. See Test Methods [D2276](#) and [D5452](#) for means of determining particulate contamination in Specification [D1655](#) aviation turbine fuels and other similar aviation fuels. See Guide [D4865](#) for a more detailed discussion of static electricity formation and discharge.

1.3 This test method has not been validated for testing biodiesel, such as meeting Specification [D6751](#) or blends of middle distillates and biodiesel, such as meeting Specification [D7467](#), or both. Test Method [D7321](#) has been determined to be suitable for testing B100 and all blends of middle distillates and biodiesel.

NOTE 2—No. 1 and No. 2 grades in Specifications [D396](#) or [D975](#) currently allow up to 5 % biodiesel meeting Specification [D6751](#). Samples containing biodiesel can result in partial dissolution or compromise of the membrane filters and give erroneous results.

1.4 The precision of this test method is applicable to particulate contaminant levels between 0 g/m³ to 25 g/m³ provided that 1 L samples are used and the 1 L is filtered

¹ This test method is under the jurisdiction of ASTM International Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of ASTM Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels. The technically equivalent standard as referenced is under the jurisdiction of the Energy Institute Subcommittee SC-B-5.

Current edition approved Oct. 1, 2021. Published October 2021. Originally approved in 1998. Last previous edition approved in 2018 as D6217 – 18. DOI: 10.1520/D6217-21.

² This test method has been developed through the cooperative effort between ASTM and the Energy Institute, London. ASTM and IP standards were approved by ASTM and EI technical committees as being technically equivalent but that does not imply both standards are identical.

completely. Higher levels of particulate contaminant can be measured, but are subject to uncertain precision.

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

1.6 *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.*

1.7 *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:³

- [D56 Test Method for Flash Point by Tag Closed Cup Tester](#)
- [D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester](#)
- [D396 Specification for Fuel Oils](#)
- [D975 Specification for Diesel Fuel](#)
- [D1193 Specification for Reagent Water](#)
- [D1655 Specification for Aviation Turbine Fuels](#)
- [D2069 Specification for Marine Fuels \(Withdrawn 2003\)⁴](#)
- [D2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling](#)
- [D2880 Specification for Gas Turbine Fuel Oils](#)
- [D3699 Specification for Kerosine](#)
- [D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester](#)

³ 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.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

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

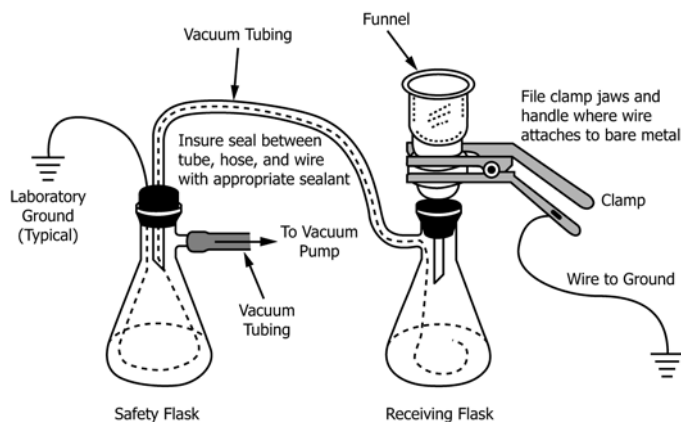


FIG. 1 Schematic of Filtration System

- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
- D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
- D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
- D7321 Test Method for Particulate Contamination of Biodiesel B100 Blend Stock Biodiesel Esters and Biodiesel Blends by Laboratory Filtration
- D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 control membrane, *n*—the lower of the two stacked membrane filters used in this test method.

3.2.2 filtered flushing fluids, *n*—either of two solvents, heptane or 2,2,4-trimethylpentane, filtered through a nominal 0.45 µm membrane filter.

3.2.3 test membrane, *n*—the upper of the two stacked membrane filters used in this test method.

4. Summary of Test Method

4.1 A measured volume of about 1 L of fuel is vacuum filtered through one or more sets of 0.8 µm membranes. Each membrane set consists of a tared nylon test membrane and a tared nylon control membrane. When the level of particulate contamination is low, a single set will usually suffice; when the contamination is high or of a nature that induces slow filtration rates, two or more sets may be required to complete filtration in a reasonable time.

4.2 After the filtration has been completed, the membranes are washed with solvent, dried, and weighed. The particulate contamination level is determined from the increase in the

mass of the test membranes relative to the control membranes, and is reported in units of g/m³ or its equivalent mg/L.

5. Significance and Use

5.1 This is the first ASTM standard test method for assessing the mass quantity of particulates in middle distillate fuels. Test Method D5452 and its predecessor Test Method D2276 were developed for aviation fuels and used 1 gal or 5 L of fuel sample. Using 1 gal of a middle distillate fuel, which can contain greater particulate levels, often required excessive time to complete the filtration. This test method used about a quarter of the volume used in the aviation fuel methods.

5.2 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.

5.3 The test method can be used in specifications and purchase documents as a means of controlling particulate contamination levels in the fuels purchased. Maximum particulate levels are specified in several military fuel specifications.

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 membrane, 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 3—The electrical bonding apparatus described in Test Method D5452 or other suitable means of electrical grounding which 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 as copper ions catalyze gum formation during the stability test.

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

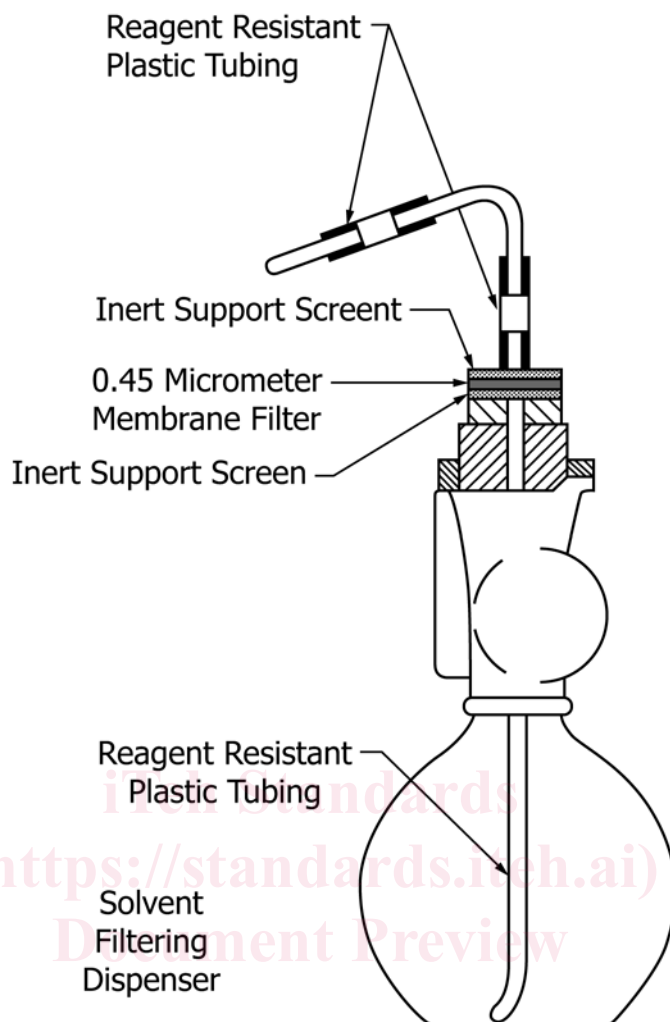


FIG. 2 Apparatus for Filtering and Dispensing Flushing Fluid

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*, for the balance case. Air ionizers shall be replaced within one year of manufacture.

NOTE 4—When using a solid-pan balance, the air ionizer may be omitted provided that, when weighing a membrane 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, the precision standard deviation of which must be 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 $90\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

6.2.5 *Flushing Fluid Dispenser*, an apparatus for dispensing flushing fluid through a nominal 0.45 μm membrane filter.⁵

NOTE 5—An apparatus such as pictured in Fig. 2 has been found suitable for this task. A standard laboratory wash bottle can also be used provided the flushing fluid is pre-filtered through a 0.45 μm pore size membrane filter and precautions are taken to maintain appropriate cleanliness of the interior of the wash bottle

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. 100 mL graduated cylinders may be required for samples which filter slowly.

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

NOTE 6—Small watch glasses, approximately 5 cm to 7 cm in diameter, have also been found suitable to support the membrane filters.

⁵ Supporting data (a membrane approval procedure) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1012. Contact ASTM Customer Service at service@astm.org.

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.⁶ 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 Type III of Specification D1193.

7.3 Flushing Fluids:

7.3.1 *Heptane*, (**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.

7.6 *Nylon Test Membrane Filters*, plain, 47 mm diameter, nominal pore size 0.8 μm.

7.7 *Nylon Control Membrane Filters* (see Note 7), 47 mm diameter, nominal pore size 0.8 μm.

NOTE 7—Membrane filters with a grid imprinted on their surface, may be used as control membrane filters for identification.

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

8. Preparation of Apparatus and Sample Containers

8.1 Clean all components of the filtration apparatus, sample containers, their caps and Petri dishes as described in 8.1.1 – 8.1.7.

8.1.1 Remove any labels, tags, and so forth.

8.1.2 Wash with warm tap water containing detergent.

8.1.3 Rinse thoroughly with warm tap water.

8.1.4 Rinse thoroughly with reagent water. Container caps should be handled only externally with clean laboratory crucible tongs during this and subsequent washings.

8.1.5 Rinse thoroughly with propan-2-ol that has been filtered through a 0.45 μm membrane filter.

8.1.6 Rinse thoroughly with filtered flushing fluid and dry.

8.1.7 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.

⁶ 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.

9. Sampling

9.1 The sample container shall be 1 L (± 0.15 L) in volume and have a screw on cap. 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 are less desirable since visual inspection of the interior of the container is more difficult. (**Warning**—It is important to note that the *entire* contents of the sample container are filtered during the conduct of this test method. 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.)

9.2 All containers and their caps, sampling lines, and other equipment used in obtaining the sample for analysis shall be thoroughly cleaned as described in Section 8. When it is not practical to clean the sample containers in this manner, the containers shall be rinsed three times with the fuel to be sampled. When it is not practical to clean the sampling lines, rinse them thoroughly with the fuel to be sampled.

9.3 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.

9.3.1 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. (**Warning**—Samples obtained from static storage may give results which 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 performed shortly after a tank has been filled.)

9.4 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 95 % by 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.

9.5 Analyze fuel samples as soon as possible after sampling. When a fuel cannot be analyzed within one day, blanket it with an inert gas such as oxygen-free nitrogen, argon, or helium and store it at a temperature no higher than 10 °C, except for