INTERNATIONAL

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Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration¹

This standard is issued under the fixed designation D 6217; 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 a middle distillate fuel by filtration. This test method is suitable for all No. 1 and No. 2 grades in Specifications D 396, D 975, D 2880 and D 3699 and for grades DMA and DMB in Specification D 2069.
- 1.2 This test method is not suitable for fuels whose flash point as determined by Test Methods D 56, D 93 or D 3828 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 D 2276 and D 5452 for means of determining particulate contamination in Specification D 1655 aviation turbine fuels and other similar aviation fuels. See Guide D 4865 for a more detailed discussion of static electricity formation and discharge.

- 1.3 The precision of this test method is applicable to particulate contaminant levels between 0 to $25~\mathrm{g/m^3}$ provided that 1 L samples are used and the 1 L is filtered completely. Higher levels of particulate contaminant can be measured, but are subject to uncertain precision.
- 1.4 The values stated in SI units are to be regarded as the standard.
- 1.5 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:
- D 56 Test Method for Flash Point by Tag Closed Tester²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester²
- D 396 Specification for Fuel Oils²

- D 975 Specification for Diesel Fuel Oils²
- D 1193 Specification for Reagent Water³
- D 1655 Specification for Aviation Turbine Fuels²
- D 2069 Specification for Marine Fuels²
- D 2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling²
- D 2880 Specification for Gas Turbine Fuel Oils²
- D 3699 Specification for Kerosine⁴
- D 3828 Test Methods for Flash Point by Small Scale Closed Tester⁴
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴
- D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems⁴
- D 5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration⁵

3. Terminology

- 3.1 Definitions:
- 3.1.1 *membrane filter*, *n*—a thin medium of closely controlled pore size through which a liquid is passed and on which particulate matter in suspension is retained.
 - 3.1.2 *ground*, *v*—to connect electrically with earth.
- 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 *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

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.14on Stability and Cleanliness of Liquid Fuels.

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 05.02.

⁵ Annual Book of ASTM Standards, Vol 05.03.

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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/m^3 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 D 5452 and its predecessor Test Method D 2276 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*, 0.912-2.59 mm (No. 10 thru 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 described in Test Method D 5452 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.
- 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*, for the balance case. Air ionizers shall be replaced within one year of manufacture.
- Note 3—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 fanassisted air circulation), controlling to 90 ± 5 °C.
- 6.2.5 *Flushing Fluid Dispenser*, an apparatus for dispensing flushing fluid through a nominal 0.45 μm membrane filter.⁶

Note 4—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

⁶ A membrane approval procedure may be obtained from ASTM Headquarters. Request RR:D02-1012.

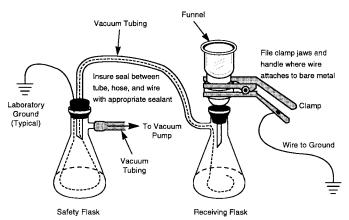


FIG. 1 Schematic of Filtration System