Standard Test Method for
Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration[1]

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 The precision of this test method is applicable to particulate contaminant levels between 0 to 25 g/m³ 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 standard. No other units of measurement are included in this 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 appro-priate safety and health practices and determine the applica-bility of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
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 Oils
D1193 Specification for Reagent Water
D1655 Specification for Aviation Turbine Fuels
D2069 Specification for Marine Fuels[3]
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
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
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

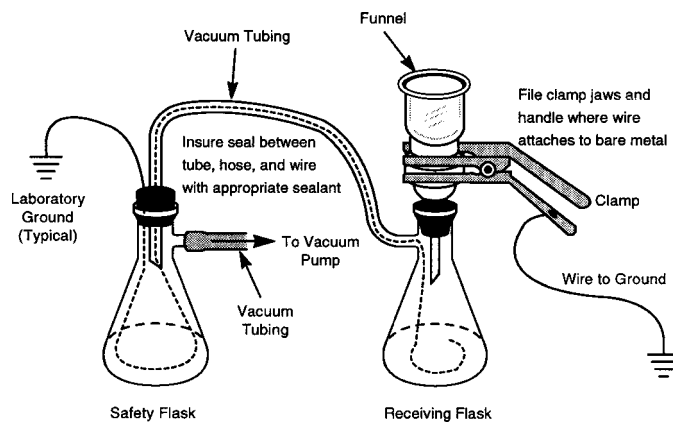## 3. Terminology

3.1 *Definitions:*

FIG. 1 Schematic of Filtration System

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

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

3.1.3 *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.

3.2 *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/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 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*, 0.912-2.59 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 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.

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.
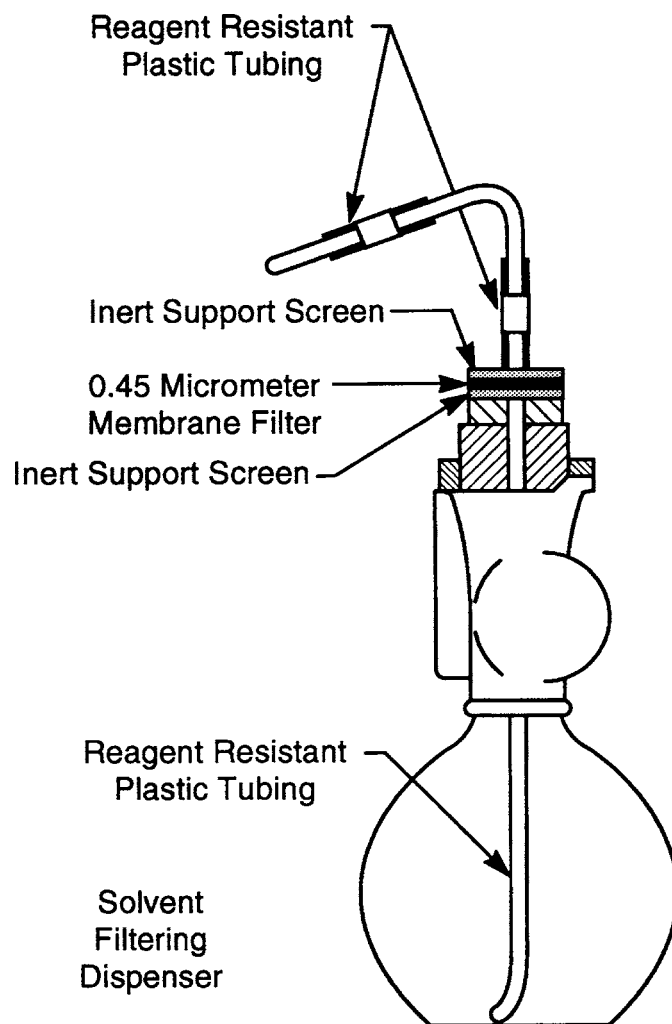
FIG. 2 Apparatus for Filtering and Dispensing Flushing Fluid

Labels in figure:
Reagent Resistant Plastic Tubing
Inert Support Screen
0.45 Micrometer Membrane Filter
Inert Support Screen
Reagent Resistant Plastic Tubing
Solvent Filtering Dispenser

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 ± 5°C.

6.2.5 *Flushing Fluid Dispenser*, an apparatus for dispensing flushing fluid through a nominal 0.45 µm membrane filter.[4]

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

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.[5] 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*:

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[4] Supporting data (a membrane approval procedure) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1012.

[5] *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 *Annual 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.

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

7.3.2 *2,2,4-trimethylpentane (isoctane)*, (**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 6), 47-mm diameter, nominal pore size 0.8-μm.

NOTE 6—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.

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 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 samples with cloud points above 10°C which are to be stored at a temperature 2°C above their cloud point.

10. Preparation of Membrane Filters

10.1 Each set of test filters consists of one test membrane filter and one control membrane filter. For fuels containing little particulate materials, only one set of filters is required. If the fuel is highly contaminated, more than one set of filters may be required (see Section 11). The two membrane filters used for each individual test shall be identified by marking the petri dishes used to hold and transport the filters. Clean all glassware used in preparation of membrane filters as described in 8.1.

10.1.1 Using forceps place the test and control membrane filters side by side in a clean petri dish. To facilitate handling, the membrane filters should rest on clean glass support rods, or watch glasses, in petri dish.

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

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

10.1.4 Remove the control membrane 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.

10.1.5 Repeat 10.1.4 for the test membrane filter.

10.1.6 Using clean forceps, place the weighed control membrane filter centrally on the membrane filter support of the filtration apparatus (see Fig. 1). Place the weighed test membrane filter on top of the control membrane filter. 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 ½ min.

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

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

NOTE 7—Some fuels may filter reasonably rapidly during transfer of the total contents of the sample container through a single set of filter membranes. However, some fuels, due to the quantity or nature of particulates, or both, may plug the membrane filter 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.

11.4.1 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. 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 11.4.2. If all the fuel has not been filtered, then proceed to 11.4.2 and 11.4.3 and then repeat from 11.4.1.

11.4.2 Wash down the inside of the funnel and the outside of the joint between the funnel and filter base filtered with flushing fluid. With the vacuum applied, carefully separate the funnel from the filter base. Wash the periphery of the membrane 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 membrane filter. Main-

tain vacuum after the final washing for 10 to 15 s to remove excess filtered flushing fluid from the membrane filter.

11.4.3 Using clean forceps, carefully remove the test and control membrane filters from the filter base and place them side by side on clean glass support rods or watch glasses in a clean, covered petri dish. Dry and reweigh the membrane filters as described in 10.1.5, taking care not to disturb the particulate on the surface of the test membrane filter. Record the final control membrane filter mass and the final test membrane filter mass to the nearest 0.0001 g for each filtration.

12. Calculation and Report

12.1 If the entire fuel sample filtered through a single set of filters then:

12.1.1 Calculate the mass on the test membrane filter, M_{tm}, as $M_2 - M_1$ in, g.

where:
M_2 = mass of the test membrane filter after the filtration (11.4.3) and
M_1 = mass of the test membrane filter before the filtration (10.1.5).

12.1.2 Calculate the mass on the control membrane filter, M_{cm}, as $M_4 - M_3$, g.

where:
M_4 = mass of the control membrane filter after the filtration (11.4.3) and
M_3 = mass of the control membrane filter before the filtration (10.1.4).

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

$$[(M_{tm} - M_{cm})/V_f] \times 10^6 \qquad (1)$$

where:
V_f = volume of fuel filtered, mL.

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

12.2 If the fuel sample required more than one set of membrane filters then:

12.2.1 For each set of filters calculate the mass on the test membrane filter, M_{tm}, as $M_{2(x)} - M_{1(x)}$, in g, where the subscripts 2 and 1 have the same meaning as in 12.1.1 and x indicates the number of the filtration.

12.2.2 For each set of filters, calculate the mass on the control membrane filter, $M_{cm\ (x)}$, as $M_{4(x)} - M_{3(x)}$, in g, where the subscripts 4 and 3 have the same meaning as in 12.1.1 and x indicates the number of the filtration.

12.2.3 Calculate the total contaminant mass and total volume of fuel filtered for each set of filters as follows:

$$M_{tm(tot)} = M_{tm(1)} + M_{tm(2)} + ... + M_{tm(x)} \qquad (2)$$

$$M_{cm(tot)} = M_{cm(1)} + M_{cm(2)} + ... + M_{cm(x)} \qquad (3)$$

$$V_{tot} = V_{f(1)} + V_{f(2)} + ... + V_{f(x)} \qquad (4)$$

where:
$M_{tm(tot)}$ = total mass on test membrane filters, g,
$M_{cm(tot)}$ = total mass on control membrane filters, g, and

V_{tot} = total volume of fuel filtered, mL.

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

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

$$[(M_{tm(tot)} - M_{cm(tot)} / V_{tot}] \times 10^6 \qquad (5)$$

12.2.5 Report the total particulate contamination to the nearest 0.1 g/m^3(mg/L), the total volume of fuel filtered in m^3(L), and the total number of filtrations (sets of membranes required).

13. Precision and Bias

13.1 *Precision*—The precision of this test method[6] was determined in accordance with currently accepted guidelines in Committee D02 Research Report D02-1007.[7] The cooperative test involved 13 laboratories and nine test fuels. Both grade 1 and grade 2 diesel fuels were used in the testing. The precision data for this procedure were developed by round robin participants using both water aspirated and mechanical vacuum systems with vacuums ranging from 1 to 100 kPa. The information on the precision of this test method was developed with fuels ranging in particulate contamination from approximately 0.3 g/m^3 to approximately 25 g/m^3. The precision data were obtained by statistical examination of interlaboratory test results using fuels samples of approximately 1 L volume. Results obtained when analyzing samples with volumes significantly different than 1 L may have different precision values.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1419.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1007.

TABLE 1 Statistical Information for Particulate Contamination

Result, g/m^3	0.3	1.0	2.0	5.0	10.0	15.0	20.0	25.0
Repeatability	0.4	0.7	1.0	1.5	2.2	2.6	3.0	3.4
Reproducibility	0.6	1.1	1.6	2.5	3.6	4.4	4.9	5.7

13.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.68 \ (X)^{0.5} \qquad (6)$$

where:
X = the test result, measured to the nearest 0.1 g/m^3

13.1.2 *Reproducibility*—The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 1.13 \ (X)^{0.5} \qquad (7)$$

where:
X = the test result, measured to the nearest 0.1 g/m^3

13.1.3 Repeatability and reproducibility values for various values of X are given in Table 1.

13.2 *Bias*—The procedure given for the determination of Test Method D6217 has no bias because the value of particulate contamination is defined in terms of this test method.

14. Keywords

14.1 diesel fuel; gravimetric determination; kerosine; laboratory filtration; membrane filter; middle distillate fuel; particulate contamination