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An American National Standard



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## Standard Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration<sup>1</sup>

This standard is issued under the fixed designation D 5452; 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.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This test method covers the gravimetric determination by filtration of particulate contaminant in a sample of aviation turbine fuel delivered to a laboratory.

1.1.1 The sample is filtered through a test membrane and a control membrane using vacuum. The mass change difference identifies the contaminant level per unit volume.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* For specific hazard statements, see 4.2, 7.3, 7.5, 11.3, and X1.7.2. Before using this standard, refer to supplier's safety labels, material safety data sheets, and technical literature.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 56 Test Method for Flash Point by Tag Closed Tester<sup>2</sup>
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>3</sup>
- D 1535 Practice for Specifying Color by the Munsell System<sup>4</sup>
- D 1655 Specification for Aviation Turbine Fuels<sup>2</sup>
- D 2244 Test Method for Calculation of Color Differences

from Instrumentally Measured Color Coordinates<sup>4</sup>

D 2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling<sup>2</sup>

D 3828 Test Methods for Flash Point by Small Scale Closed Tester<sup>5</sup>

D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination<sup>5</sup>

D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems<sup>5</sup>

### 3. Terminology

#### 3.1 Definitions:

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

3.1.2 *ground*, *vt*—to connect electrically with ground (earth).

3.1.3 *membrane filter*, *n*—a porous article of closely controlled pore size through which a liquid is passed to separate matter in suspension.

3.1.3.1 *Discussion*—RR:D02-1012 contains information on membrane filters that meet the requirements therein.

3.1.4 *particulate*, *adj*—of or relating to minute separate particles.

3.1.4.1 *Discussion*—Solids generally composed of oxides, silicates, and fuel insoluble salts.

3.1.5 *volatile fuels*—relatively wide boiling range volatile distillate.

3.1.5.1 *Discussion*—These are identified as Jet B in Specification D 1655 or the military grade known as JP-4. Any fuel or mixture having a flash point less than 38°C must be considered volatile.

### 4. Summary of Test Method

4.1 A known volume of fuel is filtered through a pre-weighed test membrane filter and the increase in membrane filter mass is weight determined after washing and drying. The change in weight of a control membrane located immediately

<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.J0 on Aviation Fuels.

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This standard has been separated from D 2276 and has been modified primarily to establish improved safety measures. Last previous edition was D 2276-89.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 06.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 05.02.

below the test membrane filter is also determined. The particulate contaminant is determined from the increase in mass of the test membrane relative to the control membrane filter.

4.2 In order to ensure safety in handling, the test method requires that volatile fuels be transferred from the sample container to the funnel without pouring. Fuels having a verified flash point greater than 38°C (refer to Test Method D 56 or Test Methods D 93 or D 3828) may be transferred by pouring the sample from the sample container directly into the funnel. Bonding the sample container to the funnel is required. (Warning—Volatile fuels such as JP-4 and Jet B or mixtures having flash points below 38°C have been ignited by electrostatic discharges when poured through membrane filters.)

4.3 Appendix X1 describes safety precautions to avoid static discharge in filtering fuel through membranes.

### 5. Significance and Use

5.1 This test method provides a gravimetric measurement of the particulate matter present in a sample of aviation turbine fuels delivered to a laboratory for evaluation. The objective is to minimize these contaminants to avoid filter plugging and other operational problems. Although tolerable levels of particulate contaminants have not yet been established for all points in fuel distribution systems, the total contaminant measurement is normally of most interest.

### 6. Apparatus

6.1 Analytical Balance, single- or double-pan, the precision standard deviation of which must be 0.07 mg or better.

6.2 Oven, of the static type (without fan-assisted air circulation), controlling to 90 ± 5°C.

6.3 Petri Dishes, approximately 125 mm in diameter with removable glass supports for membrane filters.

6.4 Forceps, flat-bladed with unserrated, non-pointed tips.

6.5 Vacuum System.

6.6 Test Membrane Filters, <sup>6,7</sup> plain, 47-mm diameter, nominal pore size 0.8 µm (see Note 1).

6.7 Control Membrane Filters, <sup>6,7</sup> 47-mm diameter, nominal pore size 0.8 µm. (Gridded control membrane filters may be used for purpose of identification.)

NOTE 1—Matched weight membrane filters, <sup>6</sup> 47-mm diameter, nominal pore size 0.8 µm, may be used as test and control membrane filters if so desired. Use of matched-weight membrane filters precludes the necessity for carrying out subsequently the procedures detailed in Section 10.

6.8 Dispenser for Flushing Fluid, 0.45-µm membrane filters to be provided in the delivery line (see Fig. 1).

6.9 Air Ionizer, for the balance case. See Note 2 and Note 3.

NOTE 2—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.

NOTE 3—Air ionizers should be replaced within 1 year of manufacture.

6.10 Filtration Apparatus, of the type shown in Fig. 2. It consists of a funnel and a funnel base with a filter support such that a membrane filter can be gripped between the sealing surface and the base by means of a locking ring. Use a metal funnel with at least a 70-mm diameter at the top.

6.11 Support Apparatus, as shown in Fig. X3.1, having adjustable height, integral spill collection pan at the base, and an edge on the can shelf to prevent the can from slipping off. The shelf is slotted. Refer to Appendix X2 for fabrication details.

6.12 Dispensing Cap or Plug, with approximately 9.5-mm inside diameter hose barb 32-mm long on which a 75 to 100-mm long piece of fuel resistant, flexible, plastic tubing is installed (see Fig. 2). The closure gasket shall be made of a fuel resistant material. A paper composition material is not acceptable.

6.13 Feed Container, shall be a 3.8 to 5-L (1-gal) epoxy lined sample can, preferably the same container in which the sample was collected.

6.14 Receiving Flask, shall be glass or metal. A graduated glass flask is preferred so that the space remaining for fuel can be observed. The filtration apparatus is fitted to the top of the flask. The flask shall be fitted with a side arm to connect the vacuum system. The flask should be large enough to contain the sample and flushing fluids.

6.15 Safety Flask, shall be glass containing a sidearm attached to the receiving flask with a fuel and solvent resistant rubber hose and shall be connected to the vacuum system.

6.16 Ground/Bond Wire, #10 thru #19 (0.912–2.59 mm) bare stranded flexible, stainless steel or copper installed in the flasks and grounded as shown in Fig. 2. If a metallic flask(s) is used instead of glass, the flask(s) must be grounded.

6.17 Plastic Film, polyethylene or any other clear film not adversely affected by flushing fluids.

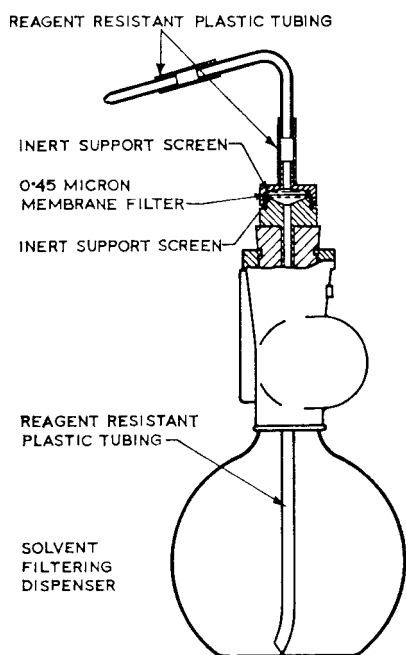


FIG. 1 Apparatus for Filtering and Dispensing Flushing Fluid

<sup>6</sup> All available membrane filters are not suitable for this application. Apparatus considered for this application shall be checked by the user for suitability in accordance with the requirements of RR:D02-1012, 1994 revision.

<sup>7</sup> A list of suppliers who have provided data indicating their membranes, field monitors, and field monitor castings are in accordance with the requirements of RR:D02-1012 is available from ASTM Headquarters. To obtain the research report, data, and list of suppliers request RR:D02-1012, 1994 revision.

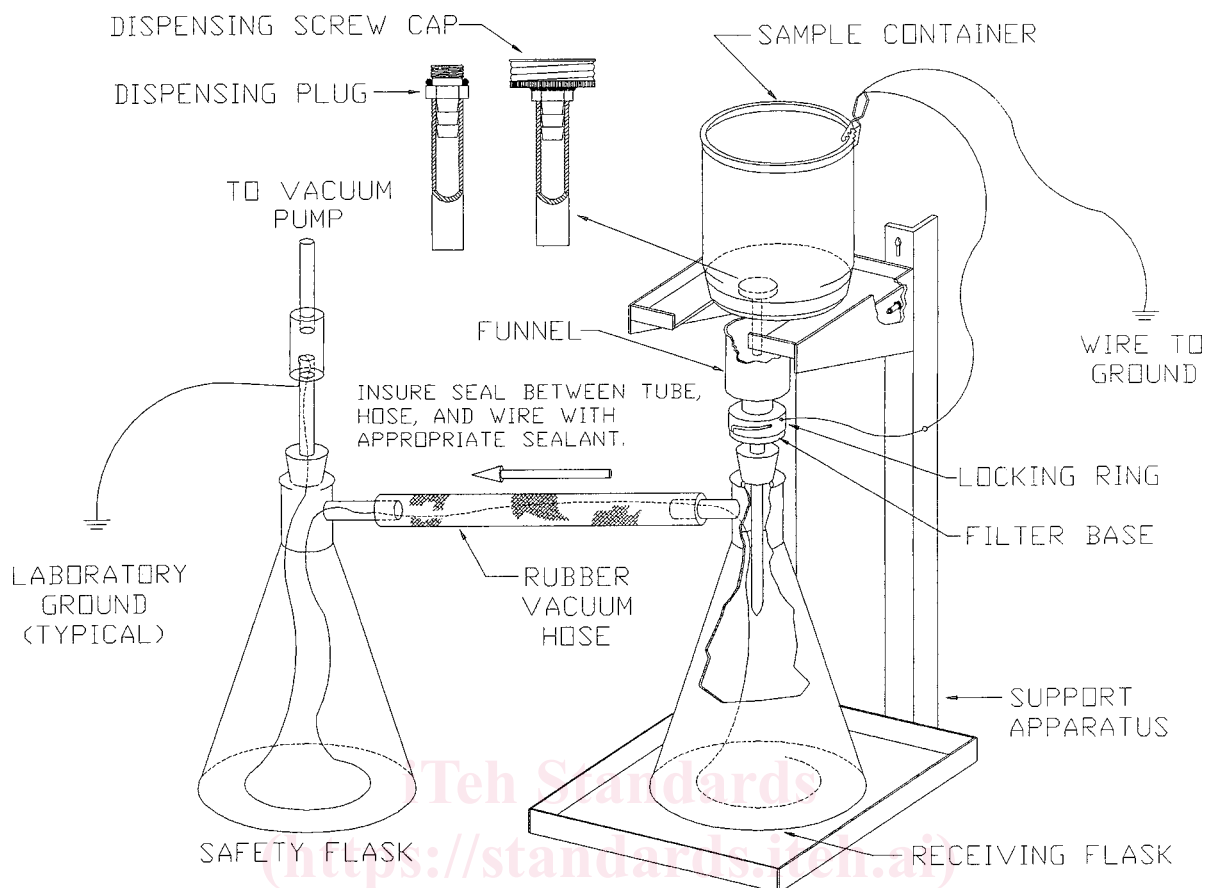


FIG. 2 Apparatus for Determining Total Contaminant

6.18 *Multimeter/VOM*, used for determining whether electrical continuity is 10 ohms or less between 2 points.

## 7. Reagents

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>8</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

7.3 *Isopropyl Alcohol*, (**Warning**—Flammable.)

7.4 *Liquid Detergent*, water-soluble.

7.5 *Flushing Fluids*—Petroleum spirit (also known as petroleum ether or IP Petroleum Spirit 40/60) (**Warning**—Extremely flammable. Harmful if inhaled. Vapors are easily

ignited by electrostatic discharges, causing flash fire.), having boiling range from 35 to 60°C.

7.6 *Filtered Fluids*—Filtered fluids are fluids filtered through a nominal 0.45- $\mu\text{m}$  membrane filter. Filtered fluids are most conveniently obtained by means of the solvent filtering dispenser described in Fig. 1.

## 8. Sampling

8.1 All containers and their closures, sampling lines, and other equipment used in obtaining the sample for analysis must be thoroughly cleaned as described in Section 9. Containers used must conform to the criteria set forth in Practice D 4306.

8.2 To obtain a representative sample from a fuel stream and to avoid external contamination, the sample may be drawn from the flushing line of a field sampling kit. Ensure that the line is first flushed with the fuel to be sampled and that the line is externally clean.

8.3 Whether or not a sampling kit is available, suitable precautions must be taken to avoid sample contamination by the use of a suitable sampling point in accordance with Test Method D 2276. If the quick-disconnect sampling connection is not used, a stainless steel ball or plug type valve should be selected as its internal design avoids the possibility of trapping or generating solid contaminant. Samples that are collected for general laboratory or chemical analysis are not necessarily suitable for this test method because insufficient care may have been taken to avoid particulate contamination.

<sup>8</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.

8.4 Where possible a 3.8 to 5-L (1-gal) fuel sample should be taken, preferably in the same container that will be used in the test to avoid the need to transfer from one container to another with increased possibility of contamination. Results obtained by taking other sample volumes may have different precisions.

8.5 The sample volume must be quoted with the results. If the sample was transferred from one container to another before the test was performed, this must also be noted with the test results.

## 9. Preparation of Apparatus and Sample Containers

9.1 Clean all components of the filtration apparatus, sample containers, and their closures as described in 9.1.1-9.1.7. Clean petri dishes as described in 9.1.2-9.1.7.

9.1.1 Remove any labels, tags, and so forth.

9.1.2 Wash with warm tap water containing detergent.

9.1.3 Rinse thoroughly with warm tap water.

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

9.1.5 Rinse thoroughly with filtered isopropyl alcohol.

9.1.6 Rinse thoroughly with filtered flushing fluid.

9.1.7 Keep a clean piece of plastic film rinsed with filtered flushing fluid over the top of the sample container until the closure is installed. Similarly protect the funnel opening of the assembled filtration apparatus with clean plastic film until ready for use.

## 10. Preparation of Test and Control Membrane Filters

10.1 Two 47-mm membrane filters of nominal pore size 0.8- $\mu\text{m}$  are required: a test and a control membrane filter. Matched-weight membrane filters may be used if so desired (see Note 1). If matched-weight membrane filters are used, it is unnecessary to carry out the procedures detailed in this section because they had been carried out previously by the membrane filter supplier. The two membrane filters used for each individual test should be identified by marking the petri dishes used as containers. Glassware used in preparation of membrane filters must be cleaned as described in 9.1.

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 in the petri dish.

10.1.2 Place the petri dish with its lid slightly ajar, in an oven at  $90 \pm 5^\circ\text{C}$  and leave it for 30 min.

10.1.3 Remove the petri dish from the oven and place it near the balance. The petri dish cover should be ajar but still protecting the membrane filters from contamination from the atmosphere. Allow 30 min for the membrane filters to come to equilibrium with the ambient 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. Weigh it and return it to the petri dish.

10.1.5 Repeat 10.1.4 for the test membrane filter. Record the membrane filter masses.

10.1.6 Using clean forceps, place the weighed control membrane filter centrally on the membrane filter support of the

filtration apparatus (see Fig. 2). Place the weighed test membrane filter on top of the control membrane filter. Install the funnel. Do not remove the plastic film from the funnel opening until ready to start filtration.

## 11. Procedure

11.1 Take precautions to minimize apparatus contamination from airborne dust. Use a protective hood or cover. Clean all equipment used for handling samples and membrane filters before use as described for petri dishes in 9.1.

11.2 With the membrane filter in place, perform a continuity test using a multimeter between the funnel and the filter holder. The meter shall read 10 ohms or less.

11.3 The assembled apparatus shall be grounded as shown in Fig. 2, by connecting a wire to a laboratory ground from the locking ring of the filtration assembly to the support stand and to the sample container. Another ground wire is required from the inside of the receiving and safety flasks and from the sample container.

11.4 Thoroughly clean the outside of the sample container in the region of the closure by washing with detergent in water and rinsing with tap water and filtered isopropyl alcohol. Shake the container vigorously for about  $\frac{1}{2}$  min. Remove the closure; remove any external contaminant that may be present in the threads on the sample container by washing with filtered flushing fluid, ensuring that none of the washings enter the container.

11.5 Transfer the fuel in the container to the filter funnel.

11.5.1 After removing the closure from the sample container and cleaning the threads, screw the closure from 6.12 on the container. If the closure is not clean, rinse thoroughly with filtered flushing fluid and dry with compressed air before use. Slip a length of plastic tubing mentioned in 6.12 snugly over the hose barb. If the closure will not fit the original sample container, shake the original container briefly and quickly transfer the sample to an appropriate container that has been rinsed as described in Section 9. Tighten the dispensing cap or plug on this container.

11.5.2 Assemble the receiving flask, preweighed filter(s), and funnel as a unit, and place on the pan of the support. Snap the support shelf on the support apparatus described in 6.11. The shelf should be positioned approximately 25 to 50 mm above the top of the funnel. Secure a grounding clip and wire to the container support and electrically ground in series as shown in Fig. 2.

NOTE 4—All of this apparatus should be mounted in an exhaust hood to minimize operator exposure to fumes.

11.5.3 Remove any labels from the container top, fold down any container handle that is present, and double over or crimp the plastic tubing installed on the cap in 11.5.1 above. Grasp the container firmly and shake gently or agitate the container briefly and carefully release vapor pressure from the container by venting through the plastic tubing away from the face. Quickly crimp the tubing again and hold in place.

11.5.4 Carefully invert the container and position it over the shelf and funnel, releasing the tubing into the funnel just before resting it on the shelf.

NOTE 5—A small clamp, pinchcock, or fingertip pressure may be used



to assist in crimping the tubing.

11.5.5 As the funnel is filling, secure a grounding clip and wire to the container and electrically ground in series with all other grounds. When the fuel has ceased flowing into the funnel, switch on vacuum source, stand well clear, and allow the fuel to filter. Agitate the container every minute during filtration by grasping firmly on both sides of the container and lifting the can approximately 12 mm off of the base and briefly shake the container for approximately 5 s.

11.5.6 Move (swirl) the container about in order to drain any residual fuel into the funnel.

11.5.7 After fuel has filtered completely, disconnect the vacuum, and record the volume of the filtered sample.

11.5.8 Rinse the inside and outside of cap or plug and tubing with approximately 30 mm of filtered flushing fluid, flushing directly into the funnel. Also rinse the test container with four 50-mL quantities of filtered flushing fluid to complete transference of the contaminant to the membrane filter. Reapply vacuum as necessary.

11.5.9 If the sample had been transferred from another container, rinse the original container with four 50-ml quantities of filtered flushing fluid to complete transference of the contaminant to the membrane filter. Attach ground wires as specified in 11.5.2. Reapply vacuum as needed to the funnel/flask assembly to flush rinse fluid through the filters. If a funnel was used to transfer to an appropriate container, rinse the funnel with 30 mL of filtered flushing fluid and on to the membrane filter.

11.5.10 Wash down the inside of the funnel and the outside joint between the funnel and filter base with filtered flushing fluid. With the vacuum applied, carefully separate the funnel from the filter base by releasing the locking ring. Wash the periphery of the membrane filter with filtered flushing fluid by directing a gentle stream of filtered flushing fluid from the edge to the center, taking great care not to wash any of the contaminant from the surface of the membrane filter. Maintain vacuum after the final washing only for the few seconds necessary to remove excess filtered flushing fluid from the membrane filter.

11.6 Using clean forceps, carefully remove the test and control membrane filters from the filter base, and place them in a clean, covered petri dish. Dry and reweigh the membrane filters as described in 10.1.2-10.1.5, taking great care not to disturb the contaminant on the surface of the test membrane filter.

## 12. Calculation and Report

12.1 Subtract the initial mass of the test membrane filter,  $W_1$ , from the final mass,  $W_2$ .

12.2 Subtract the initial mass of the control membrane filter,  $W_3$ , from the final mass,  $W_4$ .

12.3 Calculate total contaminant in milligrams per litre as follows:

$$\frac{(W_2 - W_1) - (W_4 - W_3)}{\text{Volume filtered, } L} \quad (1)$$

NOTE 6—If matched-weight membranes have been used for the test (see Note 1), then  $W_1 = W_3$  and the corrected weight of contaminant in 12.3 becomes  $W_2 - W_4$ .

12.4 Report the particulate contamination to the nearest 0.01 mg/L, and also report the sample volume used in the test.

## 13. Precision and Bias

13.1 The precision of this test method as determined by interlaboratory results<sup>9</sup> is as follows:

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 in only one case in twenty:

| Range         | Repeatability         |
|---------------|-----------------------|
| 0 to 0.6 mg/L | 0.415x <sup>0.5</sup> |

where:

$x$  = the mean of the two results.

13.1.2 *Reproducibility*—The difference between 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 in only one case in twenty:

| Range         | Reproducibility       |
|---------------|-----------------------|
| 0 to 0.6 mg/L | 0.726x <sup>0.5</sup> |

where:

$x$  = the mean of the two results.

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

**TABLE 1 Statistical Information for Particulate Contaminant**

| Average Results, mg/L | 0.1  | 0.2  | 0.3  | 0.4  | 0.5  | 0.6  |
|-----------------------|------|------|------|------|------|------|
| Repeatability         | 0.13 | 0.18 | 0.22 | 0.26 | 0.29 | 0.32 |
| Reproducibility       | 0.23 | 0.32 | 0.40 | 0.46 | 0.52 | 0.56 |

13.2 *Bias*—The procedure given for the determination of particulate contamination in aviation turbine fuel by laboratory filtration has no bias since this property can be defined only in terms of a test method.

## 14. Keywords

14.1 aviation fuel; gravimetric contaminant; membrane color; membrane filter; particulate

<sup>9</sup> A report of the interlaboratory results and statistical analysis is available from ASTM by requesting RR:D02-1384.

**X1. FILTER MEMBRANE COLOR RATINGS OF AVIATION FUELS BY LABORATORY FILTRATION**
**X1.1 Scope**

X1.1.1 This practice describes how to determine the filter membrane color rating of a sample of aviation fuel delivered to a laboratory. The filter membrane color rating may be used for the qualitative assessment of contaminant levels in the fuel or of changes in other visual characteristics.

X1.1.1.1 No quantitative relationship exists between gravimetric results obtained by Test Method D 5452 and membrane color ratings obtained by this practice.

X1.1.1.2 This practice is not a substitute for gravimetric procedures to determine particulate contaminant.

**X1.2 Summary of Practice**

X1.2.1 A known volume of fuel is passed through a 0.8- $\mu\text{m}$  filter membrane. The color on the filter membrane is compared with the ASTM color standards and assigned a rating letter and number.

**X1.3 Significance and Use**

X1.3.1 The filter membrane color rating provides a simple means of detecting changes in the fuel. Changes in membrane color may be indicative of changes in fuel contaminant level, contaminant type, the fuel handling system, or refinery process conditions. Membranes may be rated in a dry or wet condition. The advantage of rating in a dry condition is that the membrane will not change color in the dry state. Differences between dry and wet may be as great as five numbers; therefore, comparison based on mixed wet and dry ratings should not be made. Only dry color ratings should be reported when color ratings are employed as a communications tool.

X1.3.2 The sample size must be reported with the color rating because smaller samples reduce the sensitivity and the color developed is not necessarily proportional to sample volume.

NOTE X1.1—Wet color ratings or smaller than recommended sample size, or both, may be of value to a trained observer familiar with local

conditions. When performing a gravimetric test simultaneously with a color rating test, it must be recognized that the recommended 3.8 to 5-L test volume for the gravimetric test is substantially less than the 10-L color rating test sample of field Test Method D 2276. Research report RR: D02-1437 shows that the volume throughput has an influence on membrane color. Therefore, color ratings based different sample sizes should be compared only with other test results that are obtained using the same volume of fuel. A 10-L sample improves sensitivity and when using dry color ratings, enables the detection of subtle changes that may precede a major problem.

**X1.4 Apparatus**

X1.4.1 The apparatus required to filter the aviation fuel through a membrane is described in Test Method D 5452. If the color membrane only is required, without the gravimetric determination, then only a single, white plain unweighed 0.8- $\mu\text{m}$  membrane is required.

**X1.5 Color Standards<sup>10</sup>**

X1.5.1 The ASTM color standards consist of three stepwise-graded scales intended to bracket in hue the color ordinarily encountered on jet fuel filter membranes. There are two color strips and one gray strip, each divided into eleven steps and assigned rating numbers from 0 to 10. The Munsell system notations for the individual colors are listed in **Table X1.1**. The Munsell system notation values are those used historically for preparation of these color standards, and are the referee values; however, since many color rating facilities are equipped to use the CIELAB system, **Table X1.1** includes those notations. The color standards shall meet the requirements of RR:D02-1145.

NOTE X1.2—Test Method **D 2244** and Practice **D 1535** describe this test method of color designation.

<sup>10</sup> An approved "Specification for Color Rating Booklet" incorporating the ASTM Color Standards is available from ASTM Headquarters. Request RR:D02-1145. Booklets conforming to this specification are available from Gammon Technical Products, Inc., 2300 Highway 34, Manasquan, NJ 08736.