Designation: F311 – 08 (Reapproved 2013)

Standard Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters[1]

This standard is issued under the fixed designation F311; 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 practice covers the processing of liquids in preparation for particulate contamination analysis using membrane filters and is limited only by the liquid-to-membrane filter compatibility.

1.2 The practice covers the procedure for filtering a measured volume of liquid through a membrane filter. When this practice is used, the particulate matter will be randomly distributed on the filter surface for subsequent contamination analysis methods.

1.3 The practice describes procedures to allow handling particles in the size range between 2 and 1000 µm with minimum losses during handling.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D1078 Test Method for Distillation Range of Volatile Organic Liquids

D1193 Specification for Reagent Water

D1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products

D1836 Specification for Commercial Hexanes

D2021 Specification for Neutral Detergent, 40 Percent Alkylbenzene Sulfonate Type (Withdrawn 2000)[3]

F302 Practice for Field Sampling of Aerospace Fluids in Containers

F303 Practices for Sampling for Particles in Aerospace Fluids and Components

F312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters

3. Definition

3.1 *filtered liquid dispenser*—as used in this practice, a dispenser capable of delivering rinse liquid through a filter with pore size no larger than half the size of the smallest particle being considered for measurement.

4. Significance and Use

4.1 This practice provides for the processing of liquid samples obtained in accordance with Practice F302 and Practices F303. It will provide the optimum sample processing for visual contamination methods such as Method F312, and Test Method F314.

5. Apparatus and Materials

5.1 *Filtration Funnel*—The funnel opening in contact with the membrane shall be approximately 35.0 mm in inside diameter. The effective area shall be calibrated. If the funnel is to be used for measuring the sample volume, the funnel shall be calibrated within ±2 % of the required volume.

5.2 *Membrane Filter Support*—Either a fritted-glass, sintered-metal, polyphenyl-sulfone or stainless steel screen may be used. The support shall be so designed as to enable attachment to a vacuum flask.

5.3 *Vacuum Flask.*

5.4 *Funnel-Holding Device*—A provision should be made for the dissipation of static electricity from the funnel.

5.5 A clean bench or hood, supplied with unidirectional flow filtered air, in which uncovered components may be placed.

5.6 *Vacuum Source*—Minimum vacuum gage reading of 61 kPa (or other metric units acceptable to ASTM).

5.7 *Forceps,* unserrated tips.

5.8 *Filtered Liquid Dispenser.*

[1] This practice is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

5.9 *Membrane Filter,* pore size no greater than half the size of the smallest particle being considered for measurement. The filter shall have an imprinted grid on 3.10 ± 0.02-mm centers. The color shall be chosen to provide maximum contrast with the particulate contamination to be observed.

NOTE 1—When testing military hydraulic fluids, a white cellulose membrane of 0.45-µm pore size, capable of filtering 80 mL of distilled water per square centimeter per minute at 93 kPa (or other metric units acceptable to ASTM) and 25°C is required.

5.10 *Petri Dishes,* glass. Plastic Petri dishes may be used only if the selected plastic is known to be compatible with the filtered liquid.

5.11 *Glass Bottles,* small-mouth, screw-capped, permanently marked to indicate sample volume.

5.12 *Plastic Film,* 0.002-in. (0.05-mm) minimum thickness.

5.13 *Contrasting Pigment Suspension,* to be used in calibration of the funnel.

5.14 *Slides,* glass, 50 by 50-mm.

5.15 *Tape,* transparent, pressure-sensitive.

6. Reagents and Materials

6.1 *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.[4] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type III or clearer.

6.3 *Detergent,* free-rinsing. Material conforming to Specification D2021 is suitable.

6.4 *Isopropyl Alcohol,* acetone-free.[5]

6.5 *Solvent,* conforming to the following requirements (Note 2):

6.5.1 *Distillation Range,* initial boiling point 27°C (80°F) min; dry point 71°C (160°F) max, as determined in accordance with Test Method D1078.

6.5.2 *API Gravity,* 70 deg min, as determined in accordance with Test Method D287.

6.5.3 *Residue,* 0.004 g/100 mL max, as determined in accordance with Method D1353.

NOTE 2—Commercial hexanes conforming to Specification D1836 meet this requirement. Other chlorinated solvents, conforming to the Montreal conference on control of chlorinated fluorocarbons may be substituted where solvent presents a safety hazard, providing the substitute solvent is compatible with the membrane filter and adequate ventilation is used to avoid inhalation of the vapors.

6.6 *Rinse Liquid*—This liquid shall be specified by the user and shall be compatible with the sample liquid the plastic film (see section 5.12) and the membrane filter.

7. Preparation of Apparatus

7.1 Wash the apparatus thoroughly in a solution of liquid detergent and hot water and rinse with hot tap water (Note 3). Rinse with filtered isopropyl alcohol to remove the water; then rinse twice with filtered solvent.

NOTE 3—Distilled or deionized water shall be used in areas where hardness or contamination increase the blank count over the allowable level.

7.2 *Sample Bottle Preparation*—Repeat the procedure of 7.1 for both the sample bottles and caps, then allow them to drip dry. Place a piece of plastic film that has been rinsed with filtered liquid over the mouth of the bottle. Hold the film while screwing on the cap to prevent the film from rotating.

NOTE 4—It is important to hold the film when applying and removing the cap to prevent serration.

8. Funnel Calibration

8.1 Disperse a small quantity of pigment into 100 mL of liquid in a glass bottle. Prepare for a filtration as described in 9.1. Filter the suspension through the membrane filter and allow to dry. Release the vacuum and remove the membrane filter. Place the membrane on a flat surface and measure the pigment stain at three diameters 0, 45 and 90°, with a vernier caliper to the nearest 0.1 mm.

8.2 Average the three measurements and calculate the area as follows:

$$\text{Area} = \pi D^2/4 \quad (1)$$

8.3 Mark the funnel to allow identification with calculated area. Rinse the filter funnel three times with clean liquid to remove remaining pigment from the funnel surface.

9. Procedure

9.1 *Blank Analysis Filtration:*

9.1.1 Remove a membrane from the container and rinse with filtered liquid. Place the membrane on the support, lower the funnel, and secure with the holding device. Cover the funnel.

9.1.2 Place an amount of filtered liquid equal to the amount of fluid used in the routine sample filtration in a sample bottle and agitate. Remove the cover and pour the contents of the bottle into the funnel. Place approximately 50 mL of rinse liquid into the bottle and agitate. Pour rinse liquid into the funnel and cover.

9.1.3 Turn on the vacuum and allow the liquid to filter until approximately one third remains. Lift the cover and carefully wash down the funnel walls with rinse liquid (Note 5). Replace the cover and filter until the membrane is dry. Remove the holding device and funnel (see Note 6 and Note 7), then turn off the vacuum immediately. Remove the membrane using the forceps and place it in a petri dish labeled "Blank." The user may choose to subtract the blank from his sample counts, or may establish a maximum standard for particles in the blank.

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

[5] Material conforming to USP XVII, 1965, p. 995 is suitable.

NOTE 5—When the fluid filtration rate is excessive, the vacuum should be released to allow adequate rinsing of the funnel walls.

NOTE 6—Do not slide the funnel during this operation.

NOTE 7—When filtration is completed the rate of repressurization shall be such that no fluid agitation shall occur which could re-wet the filter membrane.

9.2 *Sample Filtration:*

9.2.1 Repeat the procedure described in 9.1.1.

9.2.2 Thoroughly agitate the sample bottle (Note 8), and then remove the cap.

NOTE 8—Other containers may be substituted providing they are identified in the results. The sample bottle will of necessity remain the standard container.

9.2.3 Immediately remove the funnel cover, pour the sample contents into the funnel, and recover. Place approximately 50 mL of rinse liquid in the bottle, recap, and agitate. Pour the rinse liquid into the funnel and cover.

NOTE 9—Where a filtration time is required as for military hydraulic fluids, apply suction of 47 to 57 kPa (356 to 436 mm Hg) before rinsing, and record the time required to filter the specimen. The bottle is then rinsed twice into the funnel as described.

9.2.4 Turn on the vacuum and allow the sample to filter until one third remains. Lift the cover and carefully wash the funnel walls with rinse liquid (Note 5). Replace the cover and filter until the membrane is dry. Remove the cover, holding device, and funnel, then release the vacuum immediately. Using the forceps, transfer the membrane filter to the petri dish. Label the petri dish giving the sample volume and identification. The filter is now ready for particulate contamination analysis.

9.2.5 For permanent storage, required on military hydraulic fluids, sandwich the membrane between clean glass slides hinged together with transparent tape and seal the remaining sides with tape.

10. Keywords

10.1 liquid sample handling; membrane filters; particulate contamination