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Standard Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters[1]

This standard is issued under the fixed designation F312; 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 These test methods cover the determination of the size distribution and quantity of particulate matter contamination from aerospace fluids isolated on a membrane filter. The microscopical techniques described may also be applied to other properly prepared samples of small particles. Two test methods are described for sizing particles as follows:

1.1.1 *Test Method A*—Particle sizes are measured as the diameter of a circle whose area is equal to the projected area of the particle.

1.1.2 *Test Method B*—Particle sizes are measured by their longest dimension.

1.2 The test methods are intended for application to particle contamination determination of aerospace fluids, gases, surfaces, and environments.

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

1.4 These test methods do not provide for sizing particles smaller than 5 µm.

NOTE 1—Results of these methods are subject to variables inherent in any statistical method. The use of these methods as a standard for initially establishing limits should be avoided unless ample tolerances are permissible.

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:*[2]

F302 Practice for Field Sampling of Aerospace Fluids in Containers

F303 Practices for Sampling for Particles in Aerospace Fluids and Components

F311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters

F314 Methods of Test for Identification of Metallic and Fibrous Contaminants in Aerospace Fluids (Withdrawn 1990)[3]

F318 Practice for Sampling Airborne Particulate Contamination in Cleanrooms for Handling Aerospace Fluids (Withdrawn 2013)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *unit area*—the area selected for counting particles. This may be the area of a reticle grid or some subdivision thereof, the area of one imprinted membrane grid, or any other accurately calibrated area.

3.1.2 *effective filter area*—the area of the membrane which entraps the particles to be counted.

3.1.3 *particle size*—the size of a particle as defined by area comparison or by its longest dimension.

4. Summary of Test Methods

4.1 The membrane is examined through a microscope and the particles counted according to size or size categories using a calibrated reticle. The total number of particles present is estimated by statistical methods from the actual number of particles counted. Either sizing Test Method A or B may be selected according to the preference and results expected.

5. Significance and Use

5.1 Reported particle size measurement is a function of both the actual particle dimension and shape factor, as well as the particular physical or chemical properties of the particle being measured. Caution is required when comparing data from

[1] These test methods are under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and are 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.

instruments operating on different physical or chemical parameters or with different particle size measurement ranges. Sample acquisition, handling, and preparation can also affect the reported particle size results.

6. Apparatus

6.1 *Microscope,* capable of resolving the smallest particles to be counted and producing a flat field of view.

6.1.1 The following optic combinations are recommended:

Magnification	Ocular	Objective	Minimum Numerical Aperture
50×	10×	5×	0.15
100×	10×	10×	0.25
200×	10×	20×	0.50

Similar ocular-objective combinations resulting in magnifications of 50 ± 10×, 100 ± 10×, and 200 ± 20× may be used. The optimum equipment is a compound binocular microscope. Conventional stereo microscopes will not meet these requirements.

6.2 *Mechanical Stage,* capable of traversing the entire effective filter area.

6.3 *Stage Micrometer,* with 0.1 and 0.01-mm subdivisions.

6.4 Provisions for variable high-intensity external oblique incident illumination and for a focusing condenser. A flexible or jointed arm is desirable.

6.5 *Reticles,* inscribed with reference markings that can be calibrated to represent the following dimensions:

Magnification	Size, µm	Tolerance, µm
200 ± 20×	5	±0.8
	15	±1.2
100 ± 10×	15	±1.5
	25	±2.0
50 ± 10×	50	±2.5
	50	±2.5
	100	±5.0

6.5.1 The reticles shall be as follows:

6.5.1.1 *Reticle A, Globe and Circle Pattern,* provides a means for correlation of the microscopic method with automatic counter methods. Reticle A uses the diameter of a circle for its comparison of a particle, and automatic counter methods use either a particle volume, projected area, or particle area measurements which are all directly related to the diameter.

6.5.1.2 *Reticle B, Linear Scale,* provides for measurement of the longest linear dimension technique.

NOTE 2—Some reticles combine both patterns in one reticle.

6.6 *Tally Counter,* hand operated, for recording particle counts.

6.7 Video imaging and software packages are allowed provided they meet the calibration requirements of 6.5.

7. Sampling

7.1 Collect and process the sample in accordance with the applicable methods of the American Society for Testing and Materials, as follows: Practice F302, Practice F311, Practice F318, and Practices F303.

8. Calibration

8.1 The sizing reticle shall be calibrated at each magnification by comparing the reference divisions noted in 6.3 with the rulings on the stage micrometer. Detailed calibration procedures and a discussion of errors are given in Appendix X1.

8.2 The area extrapolation factor used for statistical counting is determined by the ratio of the area counted to the total effective area of the membrane filter.

8.2.1 *Unit Area*—Measure the size of the appropriate unit area with the stage micrometer or previously calibrated reticle and calculate its area.

8.2.2 *Effective Filter Area* (Note 3)—Measure the diameter of the effective filter area with a scale, caliper, or calibrated mechanical stage and calculate the total area. Area = $\pi\ r^2$, where r is the radius of the effective circle.

NOTE 3—Where accurate effective filtering area measurements are required, a colored pigment solution should be filtered through the filtration apparatus as described in Practice F311.

8.2.3 *Area Extrapolation Factor*—The total particle count for a given size range is determined as follows:

$$C_t = (C \times A_e)/(A_u \times N) \quad (1)$$

where:

C_t = total extrapolated count,
C = actual particle count,
A_e = effective filter area,
A_u = unit area, and
N = number of unit areas counted.

9. Procedure

9.1 While exact details of the counting procedure depend partly on the specific equipment chosen, all procedures must conform to the requirements given in 9.1.1 – 9.1.9 to achieve reproducibility. Methods A and B differ only in the sizing of particles, and the detailed procedure given shall be used for either Reticle A or B.

9.1.1 Blank analysis counts, which are part of the normal processing procedure, must be used to determine the adequacy of environmental control.

9.1.2 Size and count the particles in the following order: particles greater than 100 µm (including fibers), 50 to 100 µm, 25 to 50 µm, 15 to 25 µm, and 5 to 15 µm. Particles smaller than 5 µm shall not be counted by this method. Fibers (particles with length-to-width ratio exceeding 10 to 1 and over 100 µm in length) may be identified additionally if desired. Identification may be made in accordance with Test Method F314.

9.1.3 Place the membrane filter (in a suitable holder) on the mechanical stage; adjust the lamp and microscope to achieve maximum particle definition.

9.1.4 Using 50× or lower magnification, scan the membrane surface to assure random particle distribution and to select the proper unit area to be used.

9.1.5 Select a unit area containing less than 20 particles in the size range counted at 50×. In most cases, this will represent the entire effective filtration area. Record all particles in the unit area exceeding 100 µm.

9.1.6 Move to a new unit area preselected as part of a uniform pattern designed to provide a representative sampling of the entire area and record all particles in the new unit area exceeding 100 µm.

9.1.7 Continue until either the entire effective filter area has been counted or until the complete count for the last unit area brings the total count above 100 particles. Record the number of unit areas utilized.

9.1.8 Repeat the procedure described in 9.1.5 – 9.1.7 for particles in the 50 to 100-µm size range at 50×, for the 25 to 50-µm size range at 100×, for the 15 to 25-µm size range at 100×, and for the 5 to 15-µm size range at 200×, using the appropriate reticle reference. For the purpose of sizing, a particle shall be counted in a given size range if its apparent size is equal to or greater than the reference. Particles whose apparent size is exactly that of the reference shall be assigned to the next highest range. Particles on the upper and left border of the unit area shall be counted. Particles on the lower and right hand border shall be omitted to avoid duplicate count.

NOTE 4—All size ranges counted at a given magnification may be counted simultaneously and recorded.

9.1.9 In counting particles on the membrane surface, a scheme of programmed randomness such as the following is suggested for uniformity, clarity, and speed: start a pass over the membrane from the top left corner until the edge of the effective filtering area is reached on the cross scan, then transverse downward in the same manner as above. Stop the scan point as 100 or more particles have been counted, making note of the area covered for the final calculation.

10. Calculation

10.1 Total particle counts are obtained by multiplying the particle count times the area extrapolation factor. Since the factor will usually vary according to the size range considered, this calculation must be repeated for each size range. The necessary extrapolation shall be made in accordance with 8.2.

11. Report

11.1 Total extrapolated particle counts in each particle size category shall be reported as follows:

Over 100 µm	50 to 100 µm	25 to 50µ m	15 to 25 µm	5 to 15 µm
C_{+1}	C_{+2}	C_{+3}	C_{+4}	C_{+5}

11.2 Fibers or the identification of particles may be reported in addition to particle counting.

12. Precision and Bias

12.1 Precision and reproducibility are intended to be adequate for use as a monitoring method, but the color of the membrane filter (white, black, green) has to be specified because the number of translucent and contrasting particles detected will vary depending upon the background medium.

12.2 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Mean Particle Count per Slide	Repeatability Counts
2500	650
280	94
33	13
6	3

13. Keywords

13.1 aerospace environments; aerospace fluids; aerospace gases; aerospace surfaces; membrane filter; microscopical counting particles; microscopical sizing particles; particulate matter contamination

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION

NOTE X1.1—The procedures outlined in this appendix have proven satisfactory for calibration within the tolerances required by the method. They should not be considered mandatory. Alternative procedures are equally acceptable, provided the end result is equivalent.

X1.1 Calibration of Reticles

X1.1.1 Usually it will not be possible to calibrate directly the references to be used within the required tolerances of the method due to limitations of available stage micrometers (smallest division 10 µm). It is necessary, therefore, to calibrate an even multiple of 10µ m and calculate the smaller unmeasurable size references.

X1.2 Calibration of Effective Filter Area

X1.2.1 Effective filter area, the area of the membrane covered with the particles to be counted, is dependent upon the area of the filter funnel, but is not identical, since a small quantity of particles will generally be deposited underneath the funnel walls.

X1.2.2 On very dirty samples it will be possible to measure the area directly. On most samples from aerospace systems, however, a simulated sample will be necessary.

X1.2.3 Disperse a small quantity of pigment such as iron oxide in a sample of the fluid to be tested. Approximately ¼ g of one of the MIL-G-25013 greases also is satisfactory.

X1.2.4 Average the measured diameters and calculate the area.

X1.2.5 While it is necessary to determine the effective filter area (EFA) exactly when using the method for referee purposes, certain shortcuts may be made when it is to be used for quality control purposes. If the EFA of all of the funnels to be used is known, it will be found that all funnels deviate 6 to 7 % from the average.

NOTE X1.2—*Example*—Twenty funnels selected at random from stock had a measured EFA averaging 1018 mm^2 and ranging from 984 to 1081 mm^2. However, by eliminating the five extreme examples, 15 funnels averaged 1018 mm^2 with a range from 1012 to 1035 mm^2.

X1.3 Calibration of Unit Area

X1.3.1 It will usually be necessary to calibrate several unit areas to conform to the requirement of the method that not more than 20 particles in a given size range be counted per unit area. It should be emphasized that the 20-particle limit is a maximum and that to avoid operation confusion a unit area containing fewer particles is highly desirable. There is less chance of an operator overlooking a particle or counting a particle twice if a smaller number of particles is present.