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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 F 312; 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 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.

1.4 *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:*

F 302 Practice for Field Sampling of Aerospace Fluids in Containers[2]

F 303 Practices for Sampling Aerospace Fluids from Components[2]

F 311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters[3]

F 314 Test Method for Identification of Metallic and Fibrous Contaminants in Aerospace Fluids[3]

F 318 Practice for Sampling Airborne Particulate Contamination in Clean Rooms for Handling Aerospace Fluids[2]

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 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.

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[2] *Annual Book of ASTM Standards*, Vol 10.05.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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.

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 F 302, Practice F 311, Practice F 318, and Practices F 303.

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 = πr^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 F 311.

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

$$C_t = (C \times A_e)/(A_u \times N) \tag{1}$$

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 F 314.

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