Designation: F 25 – 68 (Reapproved 1999)

Standard Test Method for Sizing and Counting Airborne Particulate Contamination in Clean Rooms and Other Dust-Controlled Areas Designed for Electronic and Similar Applications[1]

This standard is issued under the fixed designation F 25; 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 test method covers counting and sizing airborne particulate matter 5 µm and larger. The sampling areas are specifically those with contamination levels typical of clean rooms (white and gray rooms) and dust-controlled areas designed for electronic work. It is not a test method for dust counting in which isokinetic sampling is a factor (Appendix X1).

1.2 The values stated in inch-pound 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.*

NOTE 1—Information is included relative to modifications to the referee techniques which will make the test method more suitable for specific routine monitoring, such as described in Appendix X4.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *Major Projected Dimension* of a particle is designated as the particle size.

2.1.2 *Standard Unit of Length* for sizing purposes is the micrometer, µm, which is 0.001 mm or 0.000 04 in. Only particles with a measurable length greater than 5 µm are to be counted.

2.1.3 *Fiber* is considered a particle, no distinction being made with respect to length to width ratios.

3. Summary of Test Method

3.1 The test method is based on the microscopical examination of particles impinged upon a membrane filter with the aid of a vacuum. The number of sampling points is proportional to the floor area of the enclosure to be checked. The apparatus and facilities required are typical of a laboratory for the study of microparticle contamination. The operator must have adequate basic training in microscopy and the techniques of particle sizing and counting.

4. Apparatus

4.1 *Filter Holder*,[2] aerosol open type having an effective filtering area of 960 ± 25 mm^2.

4.2 *Vacuum Pump*, capable of producing a vacuum of 500 torr (500 mm Hg) while pumping at a rate of 10 L/min.

4.3 *Flowmeter*, calibrated and having a capacity in excess of 10 L/min, or a limiting orifice,[2] calibrated with the pump, filter holder, and filter used for this method at a flow rate of 10 ± 0.5 L/min. Ensure visually that the orifice is free of restricting matter before each test.

4.4 *Membrane Filters*,[2] black, 0.80-µm mean pore size, 47-mm diameter with imprinted grid squares having sides 3.10 ± 0.08 mm. Pressure drop across the filter used shall be no greater than 50 torr for an air flow rate of 1 L/min·cm^2.

4.5 *Glass Microscope Slides*, 50 by 75 mm, or 47-mm plastic disposable petri dishes.

4.6 *Forceps*,[2] with unserrated tips.

4.7 *Binocular Microscope*,[3] (Fig. 1) with ocular-objective combinations to obtain 40 to 45× and 90 to 150× magnifications. Latter objective shall have numerical aperture of 0.15 min.

[1] This test method 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] The following apparatus, or equivalent, is satisfactory for this test method; except where mentioned otherwise, the part numbers refer to equipment available from Millipore Filter Corporation, Bedford, MA.

(*1*) Filter holder, Millipore XX50 047 10; or Gelman 1200 A with 1207 Adapter available from Gelman Instrument Co., Chelsea, MI.

(*2*) Limiting Orifice, XX50 000 00.

(*3*) Filter, AA Black Grid, 0.80 µm.

(*4*) Forceps, XX62 000 06,

(*5*) Check Slide Photographic, XX50 000 50 or equivalent,

(*6*) Aerosol Monitors, Type MABG037A0, and

(*7*) Adapter, XX62 000 04.

[3] Microscopes such as Bausch & Lomb No. TBV-5, Series C; American Optical Co. X2BUHBW, Leitz SM 0.4.4S 25/81; and Zeiss: Model KF 124–212 (with accessories): or equivalent, have been found satisfactory for this purpose.

FIG. 1 Suitable Microscope: Inclined Binocular Body; Mechanical Stage; Triple Nosepiece; Ocular-Objective Combination to Obtain 40 to 45× and 90 to 150× Magnification

4.8 *Normal Counter,*[4] (2 gang) or equivalent.

4.9 *Microscope Lamp,*[5] 6 V, 5 A, high-intensity.

4.10 *Ocular Micrometer Scale,*[6] 5-mm linear scale with 100 divisions.

4.11 *Stage Micrometer,*[7] standard 0.01- to 0.1-mm scale.

4.12 *Standard Counting Specimens.* [2]

5. Sampling (Fig. 2)

5.1 The airborne particles shall be collected with the aid of a vacuum source on a membrane filter of 960-mm^2 effective filtering area. The filter surface must be vertical with respect to the floor. For an inplant method of sampling using aerosol monitors, see Appendix X4.

5.2 The standard sample for this test method shall be 10 ft^3 (283 L). For inplant procedure, the sample size may be adjusted for specific conditions.

5.3 The sample shall be taken at waist level (36 to 40 in. (0.9 to 1.0 m)) from the floor) or at bench level unless the area is limited. Sampling points shall be as designated on the sampling plan in Appendix X2. The number of samples for averaging is a function of the floor area of the space being sampled (see 5.4). These sampling locations give a statistical average for the entire room. It is recommended that areas of critical operations also be monitored for closer control of these specific areas.

5.4 The sample shall be taken at the respective locations illustrated on the sampling plan in Appendix X2. Sample at 1 for areas of cabinet size. Sample at 1′ and 2′ for areas less than 150 ft^2 (13.9 m^2). Sample at 1, 2, 3, 4, and 5 for areas up to 1000 ft^2 (92.9 m^2). For areas larger than 1000 ft^2, increase sampling by four locations per 1000 ft^2. If desired, for an average room dust count, a single sample may be taken for 5½ min at each of the five designated sampling points.

5.5 Locations are approximate. Location 1 is the area center, 1′ and 2′ are centers of triangles on respective bases. Locations 2, 3, 4, and 5 are half distances from center to respective corners on area diagonals, as shown in the sampling plan.

6. Preparation of Apparatus

6.1 Before sampling, remove dirt and dust from the filter holder by washing in a free-rinsing detergent, ketone-free isopropyl alcohol and submicron-filtered reagent grade petroleum ether (boiling range 30 to 60°C) or trichloromonofluoromethane or trichlorotrifluoroethane.

6.2 Maintain the laboratory equipment and area used for counting and sizing the airborne particulate in a condition of cleanliness paralleling or superior to the area sampled. Plastic microscope hoods have proven satisfactory as covering in the absence of a laboratory.

6.3 Personnel performing sizing and counting operations shall be equipped with garments consistent with good practice.

6.4 Clean and prepare microscope slides and petri dishes for preserving the membrane filter and specimen. Lens tissue properly used is satisfactory for this operation.

6.5 Handle hazardous chemicals used in the method with recognized precautions.

6.6 Establish a background count on membrane filters by examining each filter used for referee purposes. Examination at 40 to 50× magnifications through the microscope will reveal low or high background count.

6.7 Make a background count (Note 2), following microscopical methods outlined in this method, upon any filter with a contamination level approximating 10% or greater of the estimated test sample (Note 3). This count will be subtracted from the total count (P_t) obtained in 8.1 for each size range.

6.8 Place acceptable filters in clean petri dishes and cover. Identify dishes for test use.

NOTE 2—For routine work a background count on two filters per box of 100 is adequate under present rigid production methods.

NOTE 3—If the background count is estimated to be greater than 10 % of the total count from a 10-ft^3 (0.3-m^3) specimen, a larger sample (15 or 20-ft^3 (0.4 or 0.6 m^3) volume) may be used to eliminate background count procedure.

7. Procedure

7.1 With the aid of laboratory pressure tubing of rubber or plastic, connect the filter holder to the vacuum train which includes the filter holder, and either or both a limiting orifice of 10 L/min (Fig. 3) or a flowmeter having a capacity of 10 L/min, and a source of vacuum (vented outside sampling area or filtered to prevent contamination of the area samples) (Fig. 2).

7.2 With clean unserrated forceps, carefully remove the membrane filter from the petri dish and place, with grid side up, on the screen support of the filter holder (Fig. 4). Twist the locking ring in place to secure the filter.

7.3 When in the sampling area, place the filter holder in a horizontal position (filter surface vertical) 36 to 40 in. (91 to

[4] The Veeder Root counter has been found satisfactory for this purpose.

[5] The AO Spencer Universal, or equivalent, lamp has been found satisfactory for this purpose.

[6] Bausch & Lomb No. 31-16-01, or equivalent, scale has been found satisfactory of this purpose.

[7] Bausch & Lomb No. 31-16-99, or equivalent, micrometer has been found satisfactory for this purpose.

FIG. 3 Inserting a Typical Orifice

FIG. 4 Placing the Filter on a Typical Screen Support

FIG. 2 Typical Air Sampling-Filtration Apparatus

102 mm) from the floor level for purposes of sampling. Apply the vacuum and adjust to a flow of 10 L/min. When using the orifice, no adjustment is necessary. However, the pump should be checked with the manometer to ensure its ability to maintain a vacuum of 500 torr (500 mm Hg) or better while sampling.

7.4 The filter should be removed from the holder with forceps and placed between clean microscope slides or in a clean petri dish for transport to the microscope counting area.

7.5 *Microscopical Analysis*:

7.5.1 Place the ocular micrometer in one eyepiece. Using a stage micrometer, calibrate the measuring eyepiece (ocular micrometer) for each magnification (Fig. 5). (A whipple disk similarly calibrated is satisfactory for many inplant investigations.)

7.5.2 Knowing the subdivisions of the stage micrometer (top), the divisions of the measuring eyepiece (bottom) may be sized from it (Fig. 5).

NOTE 4—*Example*—Stage micrometer 100 µm per major division, 10 µm per minor division; 100 divisions of the measuring eyepiece subtend 1050 µm, one division of the measuring eyepiece = 10.5 µm.

7.5.3 Place the microscope slide or petri dish containing the specimen under the microscope. The petri dish cover must be removed.

7.5.4 Adjust the microscope lamp intensity and direct it on the specimen from an oblique position to obtain the maximum definition for sizing and counting. High intensity illumination is a critical requirement.

7.5.5 Use a magnification of approximately 45× for counting particles 50 µm or larger and approximately 100× for particles smaller than 50 µm. (Greater magnification may be advantageous for examination to identify particles.)

NOTE 5—Analysis for particles in the 0.5- to 5.0-µm size range may be achieved by using transmitted light techniques, after rendering the white filter transparent by placing the filter on immersion oil of refractive index 1.515. A magnification of at least 500× is required. For transmitted light microscopy, a white filter must be used (instead of black filter) since only the white filter can be rendered transparent with immersion oil. If a smaller pore size filter is used, the flowmeter and limiting orifice will require calibration with filter holder and filter in place.

7.5.6 Particles should be counted and tabulated in two size ranges: particles greater than 50 µm and particles 5 to 50 µm. Particles smaller than 5 µm are not to be counted by this method. The size of a particle is determined by its greatest projected dimension. Fibers are counted as particles.

7.6 *Method of Counting Particles*:

7.6.1 Adjust the microscopic focus and lamp position so that maximum clarity of filter surface and particle definition is obtained.

7.6.2 With the lower magnification (approximately 45×) count the entire effective filter area for particles larger than 50 µm. Use a manual counter[4] for this purpose.

7.6.3 At the higher magnification, estimate the number of particles in the 5- to 50-µm range over the effective filtering area by scanning one unit area. If the total number of particles in this range is estimated to be less than 500, count the number of particles in this range also over the entire effective filtering area. If the number is greater, the counting procedure in 7.7 applies.

7.6.4 The largest projected dimension of the particle determines the size category of the particle.

7.7 *Statistical Particle Counting*:

7.7.1 When the estimated number of particles over the effective filtering area in the 5- to 50-µm range exceeds 500, the method entails the selection of a unit area for statistical counting, counting all particles in the unit area which are in the 5- to 50-µm range, and then similarly counting additional unit areas in accordance with the counting plan of Fig. 6 until the following statistical requirement is met:

$$F_n \times N_t = >500 \quad (1)$$

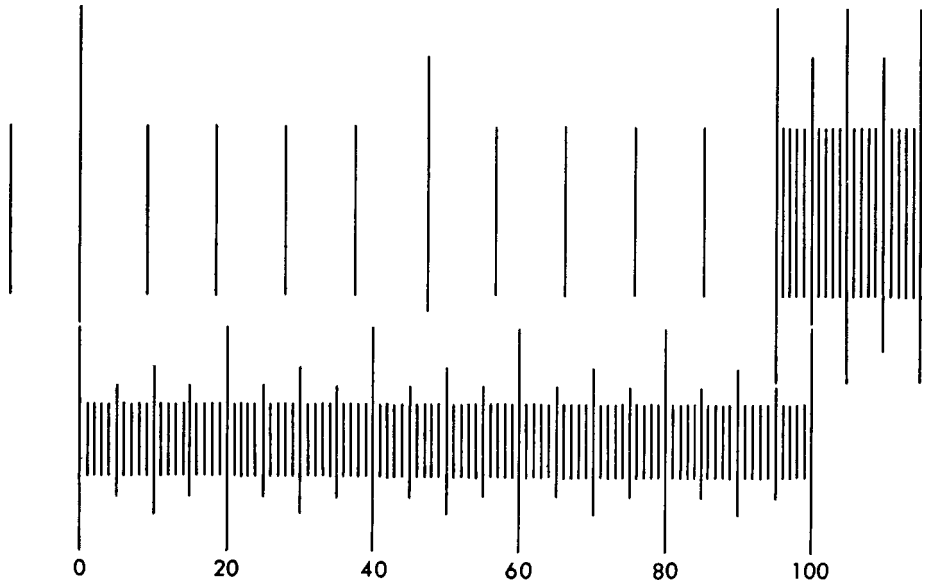


FIG. 5 Calibrating the Measuring Eyepiece

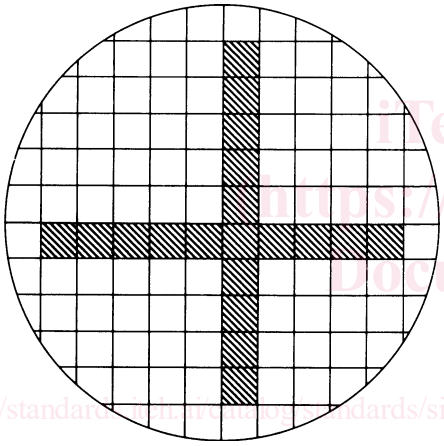


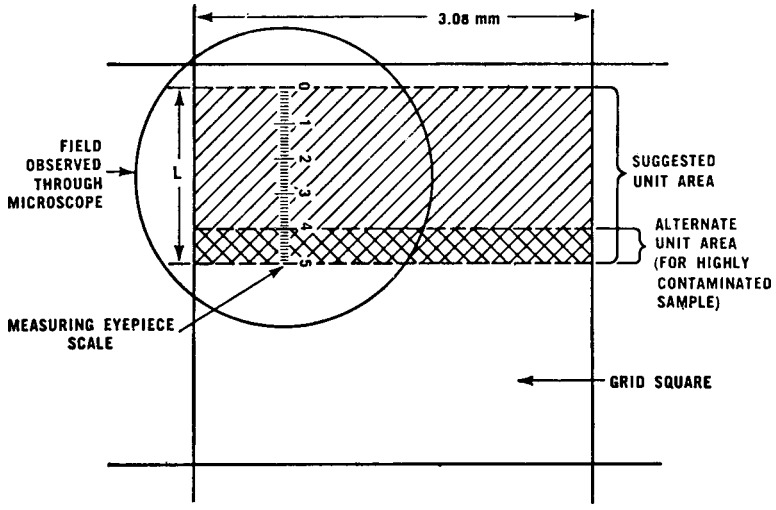
FIG. 6 Double-Diameter Counting Plan (Shaded Area Used)

where:
F_n = number of grid squares or unit areas counted and
N_t = total number of particles counted in F_n areas.

7.7.2 After establishing with low-magnification examination that particle distribution on the filter is uniform, for the referee method, use the counting plan as shown in Fig. 6. Count a number of grid squares or unit areas within different grid squares as indicated in the counting plan of Fig. 7 until the statistical requirements of 7.7.1 are met.

NOTE 6—An alternative test method for statistical particle counting is presented as Appendix X3.

7.7.3 Select unit areas for counting so that the average total number of particles in a unit area does not exceed 50 particles. (See Fig. 7 for alternative unit areas.)



NOTE 1—With membrane filter on stage, movement of the stage makes particles appear to pass the divisions on the measuring eyepiece

FIG. 7 Alternative Unit Areas