

Designation: F51/F51M – 00 (Reapproved 2014)<sup>ε1</sup>

# Standard Test Method for Sizing and Counting Particulate Contaminant In and On Clean Room Garments<sup>1</sup>

This standard is issued under the fixed designation F51/F51M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

 $\varepsilon^1$  NOTE—Editorial changes were made throughout in March 2013.

# 1. Scope

1.1 This test method covers the determination of detachable particulate contaminant 5  $\mu$ m or larger, in and on the fabric of clean room garments.

1.2 This test method does not apply to nonporous fabrics such as Tyvek (trademarked) or Gortex (trademarked). It only applies to fabrics that are porous such as cotton or polyester.

1.3 This test method provides not only the traditional optical microscopic analysis but also a size distribution and surface obscuration analysis for particles on a fine-textured membrane filter or in a tape lift sample. It utilizes transmitted illumination to render all particles darker than the background for gray level detection. Particles collected on opaque plates must be transferred to a suitable membrane filter.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.5 This standard may involve hazardous materials, operations, and equipment. 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:<sup>2</sup>

# E1216 Practice for Sampling for Particulate Contamination by Tape Lift

F25/F25M Test Method for Sizing and Counting Airborne Particulate Contamination in Cleanrooms and Other Dust-Controlled Areas

2.2 Institute of Environmental Sciences and Technology (IEST) Document:<sup>3</sup>

IEST-RP-CC003.2, Garment System Considerations for Cleanrooms and Other Controlled Environments

# 3. Terminology

3.1 Definitions:

3.1.1 *fiber*, *n*—particle longer than 100  $\mu$ m and with a length-to-width ratio exceeding 10:1.

3.1.2 *micrometre* ( $\mu$ m), *n*—SI unit of length which is 10<sup>-6</sup> of a metre or approximately 0.00004 in.

3.1.3 *particle size* (*L*) ( $\mu$ m)—major projected dimension of a particle.

#### 4. Summary of Test Method

4.1 Filtered air is drawn through five designated  $0.01 \text{-m}^2$  [1.5-in.<sup>2</sup> or approximately  $0.01\text{-ft}^2$ ] areas of a single thickness of the garment fabric at a rate of 14 L/min [0.5 cfm] for a period of 1 min for each area.

4.2 The air drawn through the garment subsequently passes through a membrane filter disk, impinging the entrained particles upon the filter surface.

4.3 The filter disk is then examined microscopically for particles removed from the garment.

4.4 For particles larger than 5  $\mu$ m, use optical analysis. For particles smaller than 5  $\mu$ m, use automated image analysis.

4.5 Cleaning and counting techniques are in accordance with those established in Section 10.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from Institute of Environmental Sciences and Technology (IEST), Arlington Place One, 2340 S. Arlington Heights Rd., Suite 100, Arlington Heights, IL 60005-4516, http://www.iest.org.



FIG. 1 Filter Assembly

### 5. Significance and Use

5.1 The test method for particulate sizing and numbers on garments is nondestructive and may be used to evaluate the contamination levels of fibers and particles on and in clean room garments. The test may be used for evaluating the cleanliness levels of new or newly cleaned garments. It also may be used to evaluate the extent of fiber and particulate contamination on garments that have been worn, if necessary. For this application, it is necessary to sample representative areas of the garment fabric.

#### 6. Apparatus

6.1 Filter Assembly and Adapter, see Fig. 1 and Fig. 2.

6.1.1 *Filter Holder*, aerosol open type having an effective filter area of 960  $\pm$  25 mm<sup>2</sup>.

6.2 *Vacuum Pump or Aspirator*, capable of operating at a pressure of 7 kPa [500 torr] with a flow rate of 14 L/min [0.5 cfm].

6.3 *Flowmeter or Orifice*, calibrated and having a capacity in excess of 14 L/min [0.5 cfm], or a limiting orifice calibrated with the pump, filter holder, and filter used for this test method at a flow rate of  $14 \pm 0.5$  L/min [0.50  $\pm 0.02$  cfm]. Ensure, visually, that the orifice is free of obstructing matter before each test.

6.4 Membrane Filters:

6.4.1 *Black*, 0.80-μm pore size, 47-mm diameter with 3.08-mm imprinted grid for fabric particles.

6.4.2 *White*, 0.80-µm pore size, 47-mm diameter without imprinted grid for fabric particles and automated image analyzer.

6.4.3 *White*, 5.0- $\mu$ m pore size, 47-mm diameter (air prefilter used with the filters in 6.4.1 and 6.4.2).

6.4.4 *Plastic Petri Slides with Covers*, <sup>4</sup> plastic petri dishes, 60-mm diameter or glass microscope slides, 50 by 75 mm.



FIG. 2 Adapter

6.5 *Binocular Microscope* with ocular-objective combinations to obtain 40 to  $45 \times$  and 90 to  $150 \times$  magnifications. Latter objective shall have a numerical aperture of 0.15 min.

6.6 Programmable Image Analyzer, a Computer-Driven Microscope Which Counts and Sizes Particles With Automated Stage and Automated Focus Interface:

6.6.1 *Microscope*, with a large glass platform automatic stage and automated focus.

6.6.2 Objectives and Projection Lenses, to generate a pixel dimension of about 5  $\mu$ m or less.

6.7 Forceps, with unserrated tips.

6.8 Normal Counter, (2 gang) or equivalent. See Note 1.

Note 1—The Veeder Root counter has been found satisfactory for this purpose.

6.9 Microscope Lamp, 6 V, 5 A high intensity.

6.10 Stage Micrometer, standard 0.01- to 0.1-mm scale.

6.11 *Ocular Micrometer Scale*, 5-mm linear scale with 100 divisions.

6.12 Standard Counting Specimens.

#### 7. Sampling Requirements

7.1 The sample shall be collected by drawing air filtered to 5  $\mu$ m through the test garment, impinging the garment-borne particles on the membrane filter. The filter surface mounted in the open-type aerosol filter holder shall be placed on the outer surface of the test garment. The garment is firmly clamped to the filter holder by means of the air-filter adapter. During sampling, the garment shall be hung or carefully positioned to minimize extraneous contamination.

7.2 The standard sample of this test method is secured with the passage of  $14 \text{ L} [0.5 \text{ ft}^3]$  of air through the test fabric during a 1-min period at each of five sampling areas as shown in Fig. 3. One sampling area is adequate for caps, helmets, towels, wipers, and booties with plastic soles. Two areas are suggested for all-fabric booties.

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the apparatus (Analyslides) known to the committee at this time is Gelman Sciences, Ann Arbor, MI. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.



7.3 Locations are approximate and may be modified to suit a specific design factor by agreement.

### 8. Preparation of Apparatus

8.1 Before sampling when using only a microscope, remove dirt and dust from the filter holder by washing in a free-rinsing detergent, ketone-free, isopropyl alcohol and submicrometer-filtered reagent grade petroleum ether (boiling range from 30 to  $60^{\circ}$ C).

8.2 Maintain the laboratory equipment and area used for counting and sizing the particles in a condition of cleanliness parallel or superior to the area sampled. Good clean room and contamination control practices should be followed. Plastic microscope hoods have proven satisfactory as covering, in a clean room, in the absence of a laboratory clean hood.

8.3 Personnel performing sizing and counting operations shall wear garments and behave in a manner appropriate to the cleanliness conditions in which they are working.

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

8.5 Handle hazardous chemicals used in the test method with recognized precautions.

8.6 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.

8.7 Make a background count (Note 2) following the microscopic methods outlined in this test 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 10.1 for each size range.

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

8.9 When using an automated image analyzer, preparation is similar to the preceding except that the white, ungridded 0.08-µm filter is used.

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 0.3-m<sup>3</sup> [10-ft<sup>3</sup>] specimen, a larger sample 0.4- or 0.6-m<sup>3</sup> [15- to 20-ft<sup>3</sup>] volume may be used to eliminate background count procedure.

#### 9. Sampling

9.1 With the aid of laboratory pressure tubing, connect the filter holder to a source of vacuum which has been found adequate to produce a flow rate of 14 L/min [0.5 cfm], at vacuum conditions test (pressure of 5 kPa or 350 torr). The holder may be open, may contain a limiting orifice (Fig. 4), or

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FIG. 4 Inserting a Typical Orifice

may be connected to the flowmeter. If a flowmeter is used between the filter holder and vacuum source, correction to the standard temperature and pressure must be made to determine actual standard temperature and pressure flow.

9.2 With clean forceps, carefully remove the appropriate membrane filter from the container and place, with grid side up, when appropriate, on the screen support of the filter holder (Fig. 5). Twist the locking ring in place after placing the tapered adapter in position. Similarly, place the 5.0-µm air filter in the top portion of the adapter by removing the O-ring from the adapter top, placing a 47-mm white filter on the support screen and replacing the O-ring. (This filter may be used for many tests.)

9.3 See IEST-RP-CC003.2 for additional recommendations on the sampling of garments.

9.4 When ready to sample, place the outer surface of the test garment over the tapered (male) adapter. Firmly lock into test position by placing the air-filter tapered (female) adapter over the test portion of fabric.

9.5 Apply vacuum at the predetermined flow rate of 14 L/min [0.5 cfm] for a period of 1 min for each area. Sample required areas (Fig. 3) by repeating 9.2.

9.6 Remove the filter from the holder with forceps and place it between the clean microscope slides, in a clean transport container (see 6.4.4) or in a clean petri dish for transport to the microscope counting area. The membrane must be cleaned before placing it in the transport container.

#### 10. Microscope Analysis Procedure

10.1 Place the ocular micrometer in one eyepiece. Using a stage micrometer, calibrate the measuring eyepiece (ocular micrometer) for each magnification (Fig. 6). A whipple disk similarly calibrated is satisfactory for many inplant investigations.

10.2 Knowing the subdivisions of the stage micrometer (top), the divisions of the measuring eyepiece (bottom) may be sized from it (Fig. 7).



FIG. 5 Placing the Filter on a Typical Screen Support



FIG. 6 Typical Counting and Sizing Microscope and Illuminator (see Test Method F25/F25M)

10.3 Remove the petri dish cover, then remove the filter from the petri dish and place it, with filtering surface up, on a 50- by 76-mm [2- by 3-in.] microscope slide. Greasing the slide lightly with silicone stopcock lubricant before mounting the filter will assist in holding the filter flat in place.

10.4 Adjust the external light source to obtain maximum particle definition with an illumination angle of approximately 45°. High-intensity illumination is a critical requirement.

10.5 Use a magnification of approximately  $45 \times$  for counting particles 50 µm or larger and approximately  $100 \times$  for particles smaller than 50 µm. Greater magnifications may be advantageous for examination to identify particles.

Note 4—*Example:* Stage the micrometer 100  $\mu$ m per major division, 10  $\mu$ m per minor division: 100 divisions of the measuring eyepiece subtend 1050  $\mu$ m, one division of the measuring eyepiece = 10.5  $\mu$ m.

Note 5—Analysis for particles in the 0.5- to  $5.0 \text{-}\mu\text{m}$  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  $500\times$  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 used and the limiting orifice will require calibration with the filter holder and filter in place.



10.6 Particles should be counted and tabulated in two size ranges: particles greater than 50  $\mu$ m and particles 5 to 50  $\mu$ m. Particles smaller than 5  $\mu$ m are not to be counted by the manual counting method. The size of particles is determined by its greater projected dimension. Fibers are counted as particles and are counted separately as fibers.

#### 10.7 Test Method of Counting Particles:

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

10.7.2 With the lower magnification (approximately  $45\times$ ), count the particles in a number of grid squares selected at random until meeting the statistical requirements in 10.9.

10.7.2.1 For particles larger than 50  $\mu$ m, use the manual counter (6.8).

10.7.3 At the higher magnification (100x), count the number of particles in the 5- to 50- $\mu$ m range in a number of grid squares selected at random until meeting the statistical requirements in 10.9. Use the manual counter (6.8).

10.7.4 If the total number of particles in this range is estimated to be less than 500, count the number of particles in this range over those grid squares. If the number is greater, the counting procedure in 10.8 applies.

10.7.5 Particles smaller than 5  $\mu$ m are not to be counted by optical microscopy. Particles smaller than 5  $\mu$ m can be counted by automated image analysis.

10.8 Particle Count:

$$F_N \times N_T > 500 \tag{1}$$

where:

 $F_N$  = number of grid squares counted and

 $N_T$  = total number of particles counted in  $F_N$  squares.

## 10.9 Statistical Particle Counting:

10.9.1 When the estimated number of particles over the number of grid squares in the 5- to 50-µm range exceeds 500, the method entails the selection of a unit area for statistical

counting, counting all of the particles in the 5- to 50-µm range, and then similarly counting additional unit areas in accordance with the counting plan of Fig. 8 until the statistical requirements are met.

10.9.2 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. 8. Count a number of grid squares as indicated in the counting plan of Fig. 8, until the statistical requirements are met.

10.9.3 Select unit areas for counting so that the average total number of particles in a unit area does not exceed 50 particles. (See Fig. 9 for alternative unit areas.)

10.9.4 If a particle lies on the upper or left boundary line of a counting area, count this particle as if it were within the boundaries of the counting area.

10.9.5 Start and finish a selected grid square or unit area by sizing and counting from the left edge of the grid line, scanning exactly one grid square width as the operation continues from left to right. Optional unit areas are: a grid square, a rectangle defined by the width of a grid square and the calibrated length of the ocular micrometer scale, and a rectangle defined by the width of a grid square and a portion of the length of the ocular micrometer scale.

10.9.6 Scan the unit area for particles by manipulating the stage so that particles to be counted pass under the ocular micrometer scale. Only the maximum dimension of the particle is regarded as significant and, for particles improperly oriented relative to the ocular micrometer, should not be rotated to size-specific particles. Using a manual counter, count all the particles in the selected area which are in the 5- to 50-µm range as indicated by the ocular micrometer scale. Record the number of particles in each unit area counted to have a record of the number of unit areas and the particles counted to meet the statistical requirements. This same procedure applies to those special requirements for counting and sizing in closer size ranges between 5 and 50 µm.



FIG. 8 Double Diameter Counting Plan (Shaded Area Used)

10.9.7 In obtaining the total number of particles, count ten or more grid squares or unit areas on the filter disk. From this count, calculate the total number of particles, which would be present on the total effective filtration area of 100 imprinted grid squares.

10.10 *Calibration of Automated Optical Analyzer*, use a certified stage micrometer to calibrate the pixel dimensions in both horizontal and vertical directions.

10.11 Sample Preparation:

10.11.1 Mount the sample using the techniques in 10.3 or 10.11.2.

10.11.2 Place the membrane filter in a petri slide and optically couple the petri slide to the glass platform of the stage using microscope immersion oil.

10.12 Operation of Image Analyzer:

10.12.1 Set the illumination intensity of the background toward the upper range of gray levels (200+ out of 254, 254 = white).

10.12.2 Index the start position and program the step progression as necessary.

10.12.3 Adjust the illumination and set the gray level threshold.

10.12.4 Initiate the program.

10.12.5 Monitor and note large particles spanning multiple fields (omitted by counting program).

10.12.6 Add large particle data to the sample database.

10.12.7 Print results in the desired format.

10.13 Tape Lift Sampling:

10.13.1 Sampling Method:

10.13.1.1 Follow Practice E1216, using 3M Magic Tape®.

10.13.1.2 Mount the tape on a clean microscope slide (see 6.4.4), and immerse the slide in acetone to remove the tape backing.

10.13.1.3 Finally, mount a cover plate over the sample area prior to counting.

10.13.2 Tape lifts may be counted either by optical microscopy or by using optical image analysis.

10.13.2.1 Paragraphs 10.1 - 10.11 describes the method to count particles by optical microscopy.

10.13.2.2 Paragraph 10.12 describes the method to count particles using the image analyzer.

# 11. Calculation

11.1 Optical Microscopy:

11.1.1 To calculate the total number of particles greater than 5  $\mu$ m and fibers on the filter, multiply the count of each category by  $100/F_N$  where  $F_N$  is the number of grid squares counted. (There are 100 grid squares on the effective filtering area of the filter). To compute the number of particles per 0.1 m<sup>2</sup> [square foot] of fabric, multiply the total filter count previously obtained by 20 providing that five areas of the garment were sampled.

Note 6—Each garment sample area is approximately  $10 \text{ cm}^2$  [1.6 in<sup>2</sup> or approximately 0.01 ft<sup>2</sup>] designed into assembly and adapter (Fig. 6). Five sampling areas provide a garment sample of 50 cm<sup>2</sup> [7.8 in.<sup>2</sup> or 0.05 ft<sup>2</sup>].

Note 7—If the effective filter area is 9.6 cm<sup>2</sup> rather than 10 cm<sup>2</sup>, the resultant error is 4 %. If this error is considered significant, the particle count should be increased accordingly when calculating the number of particles per  $0.1 \text{ m}^2$ .

NOTE 8—See Appendix X1 for alternate method for garment sampling.

11.2 Report results in terms of total particles per  $0.1 \text{ m}^2$  in the two categories:

11.2.1 Particles greater than 5 µm and

11.2.2 Fibers.

11.3 These represent the particles and fibers per  $0.1 \text{ m}^2$  of fabric removed from representative areas of the garment.

11.4 The automatic image analyzer should be programmed to report results in the desired format.

# 12. Precision and Bias

12.1 The precision and bias of this test method can be no higher than the sum total of the variables. To minimize the variables attributable to an operator, a trained microscopist is required. Variables of equipment are recognized by an experienced microscopist, thus further reducing possible error.

12.2 The 500-count method has been determined to have merit. Since the possibility of having from two to five specimens per referee investigation exists, the fatigue factor is less than that for more time-consuming methods of counting.

12.3 For training personnel, low- to medium-concentration specimens may be prepared on a grid filter and preserved between microscope slides as standards for a given laboratory. Standard specimens are commercially available.

# 13. Keywords

13.1 clean room; fabric; particulate contaminant



NOTE 1—With membrane filter on stage, movement of the state makes particles appear to pass the divisions on the measuring eyepiece. FIG. 9 Alternative Unit Areas

# **APPENDIXES**

#### (Nonmandatory Information)

# X1. ALTERNATIVE METHOD FOR PARTICULATE SAMPLING OF CLEAN ROOM GARMENTS

X1.1 A 0.31-m [1-ft] square stainless steel frame should be used as a test jig. The sides should be 76 mm [3 in.] deep to allow the frame to be placed in the vertical position. The frame should have a screen stretched over one side of the foot square opening to serve as a support for the garment test surface.

X1.2 Wrap the garment around the frame so that the closed front of the garment is stretched flat against the screen. One or two clamps may be used as necessary to hold the garment test surface in place and in a vertical position.

X1.3 The test probe (Fig. X1.1) should contain the appropriate 47-mm membrane filter with a 0.8-µm pore size.

Regulate airflow to 20 L/min [0.71 cfm], using a flowmeter, and move the probe in eight overlapping passes so that the complete test surface is covered in 1 min. The perimeter of the probe opening should fully contact the test surface.

X1.4 Remove the membrane filter and analyze the surface for particles and fibers by microscopic or automated image analyzer techniques, as described in Sections 10 and 11.

X1.5 See IEST-RP-CC003.2 for additional recommendations on the sampling using this technique.





X2.1 Table X2.1 provides classifications for various levels of contamination.

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#### **TABLE X2.1 Contamination Classification**

Note 1—Obviously broken fibers and lint-bearing seams on outer surfaces of garments, wiping cloths, caps, hoods, booties, and fabrics shall be cause for rework and rejection. Decontamination processed clean room fabrics shall be free from persistent objectionable odors.

Class	Contamination Level
	Maximum Particles/Fibers per 0.1 m <sup>2</sup> of Fabric
A	999 particles > 5 $\mu$ m/10 fibers
В	4999 particles > 5 µm/25 fibers
С	9999 particles > 5 µm/50 fibers
D	14 999 particles > 5 $\mu$ m/125 fibers
E	25 000 particles > 5 $\mu$ m/175 fibers

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