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Standard Test Method for Size-Differentiated Counting of Particles and Fibers Released from Cleanroom Wipers Using Optical and Scanning Electron Microscopy[1]

This standard is issued under the fixed designation E2090; 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.

INTRODUCTION

Techniques for determining the number of particles and fibers that can potentially be released from wiping materials consist of two steps. The first step is to separate the particles and fibers from the wiper and capture them in a suitable medium for counting, and the second step is to quantify the number and size of the released particles and fibers.

The procedure used in this test method to separate particles and fibers from the body of the wiper is designed to simulate conditions that the wiper would experience during typical use. Therefore, the wiper is immersed in a standard low-surface-tension cleaning liquid (such as a surfactant/water solution or isopropyl alcohol/water solution) and then subjected to mechanical agitation in that liquid. The application of moderate mechanical energy to a wiper immersed in a cleaning solution is effective in removing most of the particles that would be released from a wiper during typical cleanroom wiping. This test method assumes the wiper is not damaged by chemical or mechanical activity during the test.

Once the particles have been released from the wiper into the cleaning solution, they can be collected and counted. The collection of the particles is accomplished through filtration of the particle-laden test liquid onto a microporous membrane filter. The filter is then examined using both optical and scanning electron microscopy where particles are analyzed and counted. Microscopy was chosen over automated liquid particle counters for greater accuracy in counting as well as for morphological identification of the particles.

The comprehensive nature of this technique involves the use of a scanning electron microscope (SEM) to count particles distributed on a microporous membrane filter and a stereo-binocular optical microscope to count large fibers. Computer-based image analysis and counting is used for fields where the particle density is too great to be accurately determined by manual counting.

Instead of sampling aliquots, the entire amount of liquid containing the particles and fibers in suspension is filtered through a microporous membrane filter. The filtering technique is crucial to the procedure for counting particles. Because only a small portion of the filter will actually be counted, the filtration must produce a random and uniform distribution of particles on the filter. After filtration, the filter is mounted on an SEM stub and examined using the optical microscope for uniformity of distribution. Large fibers are also counted during this step. Once uniformity is determined and large fibers are counted, the sample stub is transferred to the SEM and examined for particles. A statistically valid procedure for counting is described in this test method. The accuracy and precision of the resultant count can likewise be measured.

This test method offers the advantage of a single sample preparation for the counting of both particles and fibers. It also adds the capability of computerized image analysis, which provides accurate recognition and sizing of particles and fibers. Using different magnifications, particles from 0.5 to 1000 μm or larger can be counted and classified by size. This procedure categorizes three classes of particles and fibers: small particles between 0.5 and 5 μm; large particles greater than 5 μm but smaller than 100 μm; and large particles and fibers equal to or greater than 100 μm. The technique as described in this test method uses optical microscopy to count large particles and fibers greater than 100 μm and SEM to count the other two classes of particles. However, optical microscopy can be employed as a substitute for SEM to count the large particles between 5 and 100 μm^2.

1. Scope

1.1 This test method covers testing all wipers used in cleanrooms and other controlled environments for characteristics related to particulate cleanliness.

1.2 This test method includes the use of computer-based image analysis and counting hardware and software for the counting of densely particle-laden filters (see 7.7 – 7.9). While the use of this equipment is not absolutely necessary, it is strongly recommended to enhance the accuracy, speed, and consistency of counting.

1.3 The values stated in SI units are to be regarded as the standard.

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:*[3]

D1193 Specification for Reagent Water

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

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

2.2 *Other Documents:*

ISO 14644-1 Cleanrooms and Associated Controlled Environments – Classification of Air Cleanliness[4]

ISO 14644-2 Cleanrooms and Associated Controlled Environments – Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1[4]

Fed. Std. 209E Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automatic counting, n*—counting and sizing performed using computerized image analysis software.

3.1.2 *cleanroom wiper, n*—a piece of absorbent knit, woven, nonwoven, or foam material used in a cleanroom for wiping, spill pickup, or applying a liquid to a surface.

3.1.2.1 *Discussion*—Characteristically, these wipers possess very small amounts of particulate and ionic contaminants and are primarily used in cleanrooms in the semiconductor, data storage, pharmaceutical, biotechnology, aerospace, and automotive industries.

3.1.3 *effective filter area, n*—the area of the membrane which entraps the particles to be counted.

3.1.4 *fiber, n*—a particle having a length to diameter ratio of 10 or greater.

3.1.5 *illuminance, n*—luminous flux incident per unit of area.

3.1.6 *particle, n*—a unit of matter with observable length, width, and thickness.

3.1.7 *particle size, n*—the size of a particle as defined by its longest dimension on any axis.

4. Summary of Test Method

4.1 *Summary of Counting Methods*—See the following:

Counting Technique	Particle Size Range		
	>100 µm	5–100 µm	0.5–5 µm
Stereobinocular optical microscope	20× manual	[A]	NA[B]
Scanning electron microscope	NA	200× auto	3000× manual or automatic[B]

[A] See Footnote 2.
[B] NA = not applicable.

5. Significance and Use

5.1 This test method provides for accurate and reproducible enumeration of particles and fibers released from a wiper immersed in a cleaning solution with moderate mechanical stress applied. When performed correctly, this counting test method is sensitive enough to quantify very low levels of total particle and fiber burden. The results are accurate and not influenced by artifact or particle size limitations. A further advantage to this technique is that it allows for morphological as well as X-ray analysis of individual particles.

6. Apparatus

6.1 *Scanning Electron Microscope,* with high-quality imaging and computerized stage/specimen mapping capability.

6.2 *Stereo-Binocular Optical Microscope,* with at least 40×-magnification capability equipped with a two-arm, adjustable-angle variable-intensity light source and a specimen holding plate.

6.3 *Orbital Shaker,* that provides 20-mm (¾-in.) diameter circular motion in a horizontal plane at 150 r/min.

6.4 *Microanalytical Stainless Steel Screen-Supported Membrane Filtration Apparatus,* with stainless steel funnel, TFE-fluorocarbon gasket and spring clamp.

6.5 *Vacuum Pump,* capable of providing a pressure of 6.5 kPa (65 mb) (49 torr) or lower.

6.6 *Cold Sputter/Etch Unit,* with gold or gold/palladium foils.

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[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 counting of particles 5 to 100 µm by optical microscopy is not described in this test method. However, procedures for counting particles in this size range are described in the Test Methods F25 and F312.

[3] 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.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Cancelled Nov. 29, 2001 and replaced with ISO 14644-1 and ISO 14644-2, FED-STD-209E may be used by mutual agreement between buyer and seller. Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

6.7 *Video Camera* (3-CCD preferable), that can be attached to the stereo-binocular microscope and a monitor to provide video microscopy capability.

6.8 *Personal Computer (486-Type Processor or Better) and Monitor.*

6.9 *Frame-Grabbing Hardware and Image Analysis Software,* compatible with the personal computer.[6]

6.10 *Hand-Operated Tally Counter.*

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

6.12 *Horizontal, Unidirectional Flow Workstation,* with ISO Class 5 (Fed. Std. 209 Class 100) or cleaner air.

7. Materials

7.1 *Deionized Water,* in accordance with Specification D1193, Type III, 4.0×10^{-6} $(\Omega\text{-cm})^{-1}$ or better.

7.2 *Cleanroom Gloves* (for example, unpowdered latex gloves).

7.3 *Fine-Point, Duckbill Tweezers.*

7.4 *Forceps,* two pairs, with flat gripping surface tips.

7.5 *Glass Beakers,* 1.5 L, cleaned in accordance with 10.2.1.

7.6 *Polyethylene Photographic Tray,* approximately 250 by 340 by 45 mm cleaned in accordance with 10.2.1.

7.7 *Polycarbonate Membrane Filters* (typically 0.1- to 0.4-µm pore size), white, and 25-mm diameter.

7.8 *Petri Slide,* 47 mm.

7.9 *SEM Aluminum Specimen Stubs,* typically 32-mm diameter by 10-mm height.

7.10 *Polystyrene Latex Microspheres* (sizes 0.5 and 5 µm) for use in calibration (see Section 9).

7.11 *Carbon Paint,* for SEM stub preparation.

7.12 *Low-Surface-Tension Cleaning Liquid*—Any 8- to 10-mole ethoxylated-octyl- or nonyl-phenol-type surfactant[7] prepared as a 0.1 % stock solution in deionized water. This solution will facilitate the release of both nonpolar and polar contaminants and can serve as a general test standard across industries. However, this test method is not limited to a specific cleaning solution and only requires that the cleaning liquid used be relatively free of particles and fibers. It is recommended that the cleaning liquid most relevant to the product end use be considered for this test method.

[6] "Image-Pro Plus," Version 7, available from Media Cybernetics, has been found to be satisfactory for this test method.

The sole source of supply of the apparatus known to the committee at this time is Media Cybernetics. 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,[1] which you may attend.

[7] Triton® X-100 manufactured by Rohm and Haas Co. has been found to be satisfactory for this test method.

The sole source of supply of the apparatus known to the committee at this time is Rohm and Haas Co. 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,[1] which you may attend.

8. Preparation of Apparatus

8.1 *Setting Up Stereo-Binocular Optical Microscope*—See Section 10.

8.2 *Fiber Counting by Optical Microscopy*—See Section 10.

8.3 *Setting Up Scanning Electron Microscope (SEM)*—See Section 10.

8.4 *Particle Counting by SEM*—See Section 10.

9. Calibration and Standardization

9.1 For the fiber counting by optical microscopy, the size calibration at 20× magnification can be done by comparing the fiber sizes, as visualized in the video monitor, with the rulings on the stage micrometer (with 0.1- and 0.01-mm subdivisions). For the equipment described above, a linear dimension of 8 mm in the video screen equaled 100 µm. The conversion factors are equipment-dependent and users of this test method shall establish the relation between screen size and object size.

9.2 In the SEM study, to determine the values of the start and the end areas for the computer-assisted automatic particle counting, it is necessary to perform the size calibration study by experimenting with standard-sized particles such as polystyrene microspheres or actual particles of known dimensions which can be ascertained by using the micrometre bar measurement tool available on most SEMs.

9.3 To prepare a stub with 0.5- and 5-µm spheres, add 10 µL of each of the 0.5- and 5-µm sphere suspensions to a beaker containing 500 mL of deionized water.

9.4 Filter the solution using a new membrane filter.

9.5 Prepare the SEM stub. Save the stub in a clean container as a standard size reference for the automatic particle counting at 200 and at 3000×.

9.6 For the manual procedure at 3000×, avoid counting particles having approximate linear lengths of 25 mm and up, as those will have sizes larger than 5 µm as determined from measurements done against the micrometre bars at various magnifications in the SEM.

10. Procedure

10.1 The procedure consists of two parts: preparing the sample and counting the fibers and particles. Fibers and particles greater than 100 µm are counted using an optical microscope at 20× magnification; large (between 5 and 100 µm) and small (between 0.5 and 5 µm) particles are counted using an SEM at 200 and 3000× magnifications respectively. Both manual and computer-aided automatic counting methods are used in this procedure.

10.1.1 *Sample Preparation*—Sample preparation consists of two steps:

10.1.1.1 Preparation of a background filter stub and

10.1.1.2 Preparation of the sample filter stub containing particles released from a cleanroom wiper.

10.2 *Preparation of a Background Filter Stub*—To measure the background level of particles from the glassware, polyethylene tray, and filtration system, it is necessary to prepare an experimental blank.