



# Standard Practice for Selecting, Preparing, Exposing, and Analyzing Witness Surfaces for Measuring Particle Deposition in Cleanrooms and Associated Controlled Environments<sup>1</sup>

This standard is issued under the fixed designation E 2088; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice is intended to assist in the selection, preparation, exposure, and analysis of witness surfaces for the purpose of characterizing particle deposition rates in cleanrooms and associated controlled environments, particularly for aerospace applications.

1.2 Requirements may be defined in terms of particle size distribution and count, percent area coverage, or product performance criteria such as optical transmission or scatter. Several choices for witness surfaces are provided.

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.*

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

## 2. Referenced Documents (Note 1)

### 2.1 ASTM Standards:

E 1216 Practice for Sampling for Surface Particulate Contamination by Tape Lift<sup>2</sup>

F 24 Method for Measuring and Counting Particulate Contamination on Surfaces<sup>3</sup>

F 50 Practice for Continuous Sizing and Counting of Airborne Particles in Dust-Controlled Areas and Clean Rooms Using Instruments Capable of Detecting Single Sub-Micrometer and Larger Particles<sup>2</sup>

F 312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters<sup>4</sup>

### 2.2 ISO Standard:

ISO 14644-1 Cleanrooms and Associated Controlled Environments—Part 1: Classification of Air Cleanliness<sup>5</sup>

### 2.3 Government Standards:

Fed-Std-209 Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones<sup>6</sup>

MIL-STD-1246 Product Cleanliness Levels and Contamination Control Program<sup>6</sup>

NOTE 1—The Institute of Environmental Sciences and Technology has several Recommended Practices which may also be useful.

## 3. Terminology

### 3.1 Definitions:

3.1.1 *bidirectional reflectance distribution function (BRDF)*—the scattering properties of light reflected off surfaces, expressed as the ratio of differential outputs of radiance divided by differential inputs of radiance. Surface contaminants scatter the incident radiation in all directions and with variable intensities. The BRDF is a method to quantify the spatial distribution of the scattered energy.

3.1.2 *cleanliness level*—an established maximum allowable amount of contamination in a given area or volume, or on a component.

3.1.3 *cleanroom*—an environmentally conditioned area in which temperature, humidity, and airborne contaminants are controlled by design and operation. High-efficiency particulate air (HEPA) filters or better are usually required to achieve the air cleanliness level. Air particulate cleanliness is classified in accordance with FED-STD-209 or ISO 14644-1.

3.1.4 *contaminant*—unwanted molecular and particulate matter that could affect or degrade the performance of the components upon which they reside.

3.1.5 *contamination*—a process of contaminating.

3.1.6 *contamination control*—organized action to control the level of contamination.

3.1.7 *controlled area*—an environmentally controlled area, operated as a cleanroom, but without the final stage of HEPA (or better) filters used in cleanrooms.

3.1.8 *critical surface*—any surface of an item or product which is required to meet established cleanliness level requirements.

3.1.9 *demonstrated equivalence*—the condition in which a

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.03.

<sup>3</sup> Discontinued; see *1993 Annual Book of ASTM Standards*, Vol 15.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

<sup>6</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

method of measurement has passed a series of tests to show that it gives equivalent results to those of a standard measurement.

3.1.10 *environmentally controlled area*—cleanrooms, controlled areas, good housekeeping areas, and other enclosures that are designed to protect hardware from contamination. Cleanliness is achieved by controlling air purity, temperature, humidity, materials, garments, and personnel activities.

3.1.11 *fiber*—a particle >100  $\mu\text{m}$  in length with a length to diameter ratio of ten or more.

3.1.12 *image analysis*—the measurement of size, shape, number, position, orientation, brightness, and other parameters of small objects using the combination of a microscope, an imaging sensor, and a dedicated computer system. Image analysis can be used to perform particle counts or measure particle dimensions automatically, with far greater accuracy than manual techniques.

3.1.13 *micrometre* ( $\mu\text{m}$ )—a unit of measurement equal to one millionth of a metre, or approximately 39 millionths of an inch, for example, 25  $\mu\text{m}$  is approximately 0.001 in. The term “micron” has been used but is not a recommended SI unit.

3.1.14 *nonvolatile residue* (NVR)—soluble material remaining after evaporation of a filtered volatile fluid or precipitate from a gas phase, usually reported in milligrams per unit area (or volume).

3.1.15 *particle deposition*—the settling of airborne particles onto surfaces resulting from electrostatic or dynamic conditions, or both, in cleanrooms or other controlled environments.

3.1.16 *particle fallout* (PFO)—a standard particle deposition method used by the European aerospace community which uses black glass witness surfaces and measures particle scatter in parts per million.<sup>7</sup>

3.1.17 *particle size*—(1) the apparent maximum linear dimension of a particle in the plane of observation, as observed with an optical microscope; (2) the equivalent diameter of a particle detected by automatic instrumentation. The equivalent diameter is the diameter of a reference sphere having known properties and producing the same response in the sensing instrument as the particle being measured; (3) the diameter of a circle having the same area as the projected area of a particle, in the plane of observation, observed by image analysis; (4) the size defined by the measurement technique and calibration procedure.

3.1.18 *particulate contamination*—discrete mass of solid matter, size often measured in micrometres ( $\mu\text{m}$ ), which adversely affects critical surfaces of component and hence system performance.

3.1.19 *percent area coverage* (PAC)—fraction of the surface that is covered by particles, reported in percent as total particle projected area divided by total area of the surface.

3.1.20 *precision cleaning*—cleaning of hardware surfaces approved by established facility methods or methods specified or provided by the customer with verification to a specified cleanliness level.

3.1.21 *visibly clean*—absence of particulate or molecular contaminants when viewed from a specified distance with

normal (or corrected to normal) vision with a specified illumination level.

3.1.22 *witness surface* (WS)—a contamination-sensitive material used instead of direct evaluation of a specific surface when that surface is either inaccessible or is too sensitive to be handled.

3.1.22.1 *optical witness surface* (OWS)—witness surface from which contaminants may be analyzed by optical methods.

3.1.22.2 *particle witness surface* (PWS)—witness surface from which particulate contaminants may be analyzed by standard optical or electron microscopic methods.

## 4. Summary of Practice

4.1 Particle deposition in controlled environments is determined by collecting particles on a clean witness surface for a specified period of time or operational activity, then retrieving the witness surface and quantifying the particle population collected.

4.2 Witness surfaces (WS) are typically surfaces that lend themselves to traditional microscopic or image analysis techniques for sizing and counting particles on the surface, but may be an optical surface that is evaluated on the basis of the change in its optical properties or may be a witness surface that best represents the surface material of interest which is subsequently evaluated by extracting a sample from the surface and sizing and counting particles removed from the witness surface.

4.3 This practice does not address real time particle deposition measurements involving particle counters on site with continuous recording over a specified period of time.

## 5. Significance and Use

5.1 This practice provides a standard approach to measuring particle deposition, or fallout, in cleanrooms and other controlled environments. It is based on the use of a witness surface to collect particles that deposit from the surrounding environment and subsequently sizing and counting the particles by conventional methods. Several options are introduced, with limitations and guidelines for selecting the best choice for the intended application.

5.2 This practice is applicable across numerous industries including aerospace, microelectronics, and pharmaceuticals.

## 6. Selecting Witness Surfaces

6.1 Considerations for selecting WS include available methods of analysis, precision and accuracy required, size of particles of concern, actual material of critical surfaces of concern, and cost. Preferably, the WS should be a surface material which best represents the actual critical surface and should be analyzed using the method which best represents the actual performance characteristics of interest. Additionally, certain surfaces may become charged, especially in dry environments, and this charging can effect the particle deposition. If WS are to monitor a vacuum environment they must be made of low-outgassing, vacuum-compatible materials and held securely in vacuum-compatible, low-particle shedding holders.

6.2 *Microscopic Evaluation*—When microscopic sizing and counting of particles is the planned method of analysis, select one of the following PWS, each of which is easily evaluated

<sup>7</sup> The Euramark Model 255 PFO photometer has been found to be satisfactory.

directly after exposure. Microscopic sizing and counting shall be performed in accordance with Method F 24 or Test Methods F 312.

6.2.1 *Membrane Filters*<sup>8</sup>, should be gridded for ease in microscopic particle counting and precleaned before exposure. A membrane filter can be prepared as either a tacky or tack-free surface. The membrane filter is cleaned and then either (1) immediately placed in a cleaned petri dish, (2) dipped into trichloroethylene or methyl chloroform first so it will fuse to the plastic petri dish, or (3) dipped into a prefiltered tacky adhesive and dried in a cleaned petri dish. The petri dish is then covered and transported to the area being tested.

6.2.2 *Gridded Counting Slides*, such as those used in Practice E 1216 may be used as WS. After exposure, a pressure-sensitive tape is applied to the slide to encapsulate the deposited particles before moving them to a microscope for analysis.

6.2.3 *Stainless or Other Surfaces*, other materials may be selected as WS based on specific needs for durability or to best represent the actual surface materials of interest. For these PWS, particles are subsequently extracted from the surface with a fluid, filtered to collect the particles on a gridded membrane, and subsequently analyzed microscopically. Note, the efficiency of the extraction method must be known or estimated.

6.3 *Other Particle Sizing and Counting Methods*—Particle characterization can also be performed using optical measurements other than manual microscopic methods. Highly polished surfaces serve as WS and are selected based on the analysis method chosen.

6.3.1 The PFO instrument uses a smooth black glass plate 40 by 45 mm protected from unintentional sedimentation by a plate holder. The effective sampling surface is circular with a diameter of 25 mm.

6.3.2 Silicon wafers or disks shall be selected for image analysis or other surface scanning methods.

6.4 *Optical Witness Surfaces*, (that is, mirrors or lenses) shall be selected to best represent the critical surface of interest in the environment being evaluated. Reflectance or transmission measurements shall be made in the wavelengths of interest, and the OWS must be the correct size and shape for the instrumentation planned for use.

6.5 *Gravimetric Methods*—A gravimetric method can also be used, whereby a large witness surface is rinsed with solvent to extract the particles, filtered onto a dry, preweighed membrane filter, and then dried and reweighed on a laboratory balance with a resolution of 0.01 mg. The difference in weight can be a relative quantitative analysis of deposition based on weight. Note, the efficiency of the extraction method must be known or estimated. A preweighed membrane filter could also be used as the witness surface thus eliminating the extraction step. Additionally, a quartz crystal microbalance with adhesive surfaces can measure accumulated mass in situ.

## 7. Preparation of Witness Surfaces

7.1 *Witness Surface Holders*—Holders should be designed

to retain the witness surface securely and maximize the surface exposure. They should be made from smooth, cleanable materials such as plastic, anodized aluminum, or stainless steel. A noncontact, easily removable, protective cover is required which prevents the collection of particulate contamination during transport of the surfaces between the test laboratory and the controlled environment being evaluated. Holders should have captive fasteners and tethers to prevent the holder or associated hardware from impacting critical surfaces if dropped. Holders should also be designed to be secured in the facility being evaluated in either a vertical or horizontal orientation.

7.2 *Cleaning of Holders*—Holders should be precision cleaned in accordance with MIL-STD-1246 Level 100 or clean before installing the witness surface. It is recommended that cleaning and packaging be performed in a FED-STD-209 Class 100 or better clean bench.

7.3 *Cleaning of WS*—Membrane filters should be blanked or recleaned with filtered fluid before exposure. Tapes should be inspected before use or a control of the tape must be taken to compare the actual surfaces. Glass or polyester film-gridded slides should be flushed with a filtered solvent. Silicon wafers and disks may be new, repolished, or recleaned with solvent and individually baselined. The PFO black glass is wiped with methanol-soaked lint-free lens tissue in a unidirectional manner.

7.4 *Baselining of OWS*—The OWS must be baselined by the selected reflectance, transmittance, or scatter measurement before exposure. With this type of analysis, the baseline value is subtracted from the postexposure measurement to determine the net optical degradation as a result of particle deposition on the WS.

7.5 *Protective Packaging*—All precision cleaned holders containing witness surfaces shall be provided with cleanliness protection before leaving the controlled environment. Clean room approved, low-particulate packaging shall be used in a double-wrap sealed configuration. The outer wrap should be a moisture-resistant material.

7.6 *Control Surfaces*—One or two control surfaces shall be prepared in the same manner as the others and be subjected to all conditions of the actual surfaces (that is, cleaning, mounting, packaging, and so forth) except that the cover will be removed, then immediately replaced in the environment being evaluated.

## 8. Exposure of Witness Surfaces

8.1 Transport packaged, covered witness surfaces to and from the controlled environment being measured in a horizontal orientation. Minimize the distance traveled whenever possible and protect the WS from the elements. After exposure, the witness surfaces are covered, repackaged, and promptly taken to the laboratory for evaluation.

8.2 Cleanroom garment requirements are dictated by the controlled environment being measured except that head and facial hair covering is required. Use of low-particle shedding cleanroom gloves<sup>9</sup> is required whenever handling, unpackaging, or exposing witness surfaces.

<sup>8</sup> Millipore Corp. 47-mm membrane filter #RAWG04700 has been found to be satisfactory. Other similar filters, including larger sizes, are acceptable.

<sup>9</sup> Cleanroom latex gloves have been found to be effective.

8.3 Securing or tethering the WS holder during the exposure period is recommended, but dictated by the requirements of the controlled environment being measured. Label WS holders in a discrete manner; if permanent engraving is not used, the label material shall be cleanroom compatible and not a source of particulate contamination.

8.4 The number of WS placed in the environment being evaluated shall be large enough to ensure a representative sampling of the critical area, for example, three or more. The WS should be placed as close as possible to the critical surface(s) within the environment, preferably in the same orientation, and if possible between the critical surface(s) and main contamination sources. The duration of exposure should be equivalent to the exposure period of the critical surface(s), but may alternatively be exposed for specific events or for fixed time periods, that is, day, week, month, and so forth.

8.5 Removal and replacement of the witness surface covers, thus exposing the PWS or OWS is a critical step in the exposure process. The cleanliness of covers and containers must be maintained during the exposure period so that they can be reused without affecting the WS results. Utmost care shall be taken to ensure that the inner surface of the cover remains clean and that particle generation from the handling process is kept to a minimum. This includes standing downstream of the clean air flow during uncovering of the WS.

8.6 The location of surface sites within the controlled environment and their orientation (that is, vertical versus horizontal), along with the date and time of cover removal and replacement, shall be documented. It is recommended that additional information such as airborne particle counts and a log of operations (that is, activity levels, crane operations, large door opening, and so forth) be collected during the witness surface exposure period to supplement the particle deposition data.

8.7 Control surfaces should be exposed in the environment being evaluated only for as long as it takes to remove the cover and replace it immediately. They should remain in the facility and not be exposed to any extra transportation steps.

8.8 Signs and instructions are needed to inform all personnel allowed to enter the environment being evaluated that the WS are not to be disturbed or touched.

## 9. Analysis of PWS and OWS

9.1 *Sizing and Counting by Optical Microscopy*—Microscopic sizing and counting of particles shall be performed in accordance with Method F 24 or Test Methods F 312. The optical microscope shall be capable of measurements as small as 5 μm in size.

9.2 *Sizing and Counting by Image Analysis*—Image analysis is the process of digitizing an image for the purpose of gaining quantitative information about it (diameter, area, length, and so forth). Programs may be written to have the image analyzer measure each particle's longest dimension and count it in appropriate size bins much like the manual microscope method allows, or it may be programmed to measure particle areas and based on the WS area evaluated report the results as a percent area coverage.

9.3 *BRDF Measurements*—The BRDF measurements on OWS are performed using a suitable scatterometer. The wave-

lengths used for the scatter measurements and the light source angles from specular shall be specified. Measurements shall be compared to baseline measurements made before WS exposure and to unexposed control samples.

9.4 *Reflectance or Transmission Loss*—Reflectance or transmission loss on OWS is caused by surface contamination and is reported as a percent change. These measurements are made in the wavelengths of interest.

9.5 *Surface Scanners*—Surface scanners made by Estek, Tencor, VLSI, Q3, and so forth, are designed to detect defects on silicon wafers or disks. They can also be used to detect surface contamination, reported as area defects, usually in square micrometres. Measurements shall be compared to baseline measurements made before WS exposure and to unexposed control samples.

## 10. Calculation

10.1 Particle deposition results shall be calculated per unit area and per unit time. Units of time should be converted to per day.

10.2 Particle distributions from microscopic evaluation can be translated to cleanliness level in accordance with MIL-STD-1246. Control surface background values shall be used as a baseline from which to compare the actual results. A baseline blank of each WS before exposure will be subtracted from the sample results. A control average may also be subtracted if more than one control is taken for a batch of WS and the results are believed to be truly representative of the sample minus the exposure. However, the control for a batch is better used as verification of the handling and cleaning procedure to ensure that they are negligible.

10.3 An alternative method of specifying particle levels on a surface is expressed as percent area coverage (PAC). Particle area may be directly measured using image analysis or other techniques. Otherwise, particle sizing and counting must be performed and the values converted to a PAC value. Table 1 provides the conversion to be used if the shapes of particles are not known. The coefficients in Table 1 are based on statistical studies of particle shapes. The probability that particles are fibers increases with increasing size. Sometimes fibers are counted separately from other particles, and the projected areas can be estimated. Table 1 is based on a sample size of 0.1 m<sup>2</sup>. Other possible methods for PAC include obscuration or light scattering, after having demonstrated equivalence with actual measured projected areas.

**TABLE 1 Formula to Calculate Particle Percent Area Coverage**

Particle Size Range	Particles per 0.1 m <sup>2</sup>	X	Coefficient	Percent Area Coverage <sup>A</sup>
>1 – 10 μm	<sup>B</sup>	X	1.737 × 10 <sup>-8</sup> =	
>10 – 25 μm		X	1.528 × 10 <sup>-7</sup> =	
>25 – 50 μm		X	7.078 × 10 <sup>-7</sup> =	
>50 – 100 μm		X	2.435 × 10 <sup>-6</sup> =	
>100 – 150 μm		X	5.186 × 10 <sup>-6</sup> =	
>150 – 250 μm		X	7.484 × 10 <sup>-6</sup> =	
>250 – 500 μm		X	6.522 × 10 <sup>-6</sup> =	
>500 – 750 μm		X	1.048 × 10 <sup>-5</sup> =	
>750 μm		X	1.922 × 10 <sup>-5</sup> =	

<sup>A</sup>Sum all values to obtain total percent area coverage.

<sup>B</sup>Value may be estimated by multiplying counts within the 10- to 25-μm range for count in the 1- to 10-μm range by 3.25.

## 11. Report

11.1 Report particle deposition results per unit area and per unit time. Units of time should be converted to per day.

11.2 Identify the WS type and the test method used to measure the particle deposition results in the report.

11.3 Identify the location, orientation, and exposure duration of the WS in the report. Report any additional information, as noted in 8.6.

11.4 Notation of the visible physical properties of particles such as morphology, color, and so forth, is optional and may be

useful for trouble shooting cleanroom contamination problems.

## 12. Precision and Bias

12.1 Precision and bias have not yet been determined.

12.2 Some measuring instruments are calibrated with particles of known size (for example, polystyrene latex spheres) and the reporting data are then expressed as an equivalent.

## 13. Keywords

13.1 cleanroom; contamination control; controlled environment

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