

# 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 clean-rooms 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: <sup>2</sup>

- E 1216 Practice for Sampling for Particulate Contamination by Tape Lift
- F 24 Test Method for Measuring and Counting Particulate Contamination on Surfaces
- F 312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters
- 2.2 ISO Standard:
- ISO 14644-1 Cleanrooms and Associated Controlled Environments—Part 1: Classification of Air Cleanliness<sup>3</sup>
- 2.3 Government Standards:

Fed-Std-209 Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones<sup>4</sup>

IEST-STD-CC1246 Product Cleanliness Levels and Contamination Control Program<sup>5</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.

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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 American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

<sup>&</sup>lt;sup>4</sup> Although Fed-Std-209 has been cancelled, it still may be used and designations in Fed-Std-209 may be used in addition to the ISO designations.

<sup>&</sup>lt;sup>5</sup> Available from the Institute of Environmental Sciences and Technology, 5005 Newport Dr., Suite 506, Rolling Meadows, IL 60008.

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 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  $\mu$ 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* ( $\mu$ m)—a unit of measurement equal to one millionth of a metre, or approximately 39 millionths of an inch, for example, 25  $\mu$ 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 that uses black glass witness surfaces and measures particle scatter in parts per million.<sup>6</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 (µm), 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

<sup>&</sup>lt;sup>6</sup> The Euramark Model 255 PFO photometer has been found to be satisfactory. The sole source of supply of the apparatus known to the committee at this time is Euramark, 834 East Rand Rd., Unit 6, Box 823, Mt. Prospect, IL 60056. 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.

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 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*, 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 pressuresensitive 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 IEST-STD-CC1246 Level 100 or clean before installing the witness surface. It is recommended that cleaning and packaging be performed in an ISO 14644 Class M3.5 (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 post exposure 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