Designation: F 24 – 00

Standard Method for Measuring and Counting Particulate Contamination on Surfaces[1]

This standard is issued under the fixed designation F 24; 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 method covers the size distribution analysis of particulate contamination, 5 µm or greater in size, either on, or washed from, the surface of small electron-device components. A maximum variation of two to one (±33 % of the average of two runs) should be expected for replicate counts on the same sample.

NOTE 1—For satisfactory results on clean parts, it is recommended that all procedures involved in sample preparation be conducted under a dust shield.

2. Terminology

2.1 *Definitions:*

2.1.1 *particulate contaminant*—a discrete quantity of matter that is either foreign to the surface on which it rests or may be washed from the surface on which it rests by the ultrasonic energy procedure herein described.

2.1.2 *particle size*—the maximum dimension of the particle.

2.1.3 *fibers*—particles longer than 100 µm with a length to width ratio of greater than 10:1.

2.1.4 *planar surface*—a surface that does not move out of the depth of field of the microscope when the area to be observed is traversed under the highest magnification to be used.

3. Summary of Method

3.1 The method comprises two procedures for preparing specimens for microscopical analysis: one for adhered particles on planar surfaces and the second for particulate contamination removed from irregular surfaces. A single optical analysis procedure is presented for particle enumeration in stated size ranges. For planar surfaces, the component is mounted on a suitable flat support and mounted on the microscope stage. For irregular surface components, the contamination is removed by subjecting the component to an ultrasonic cavitation field while immersed in water containing a detergent. The contamination is subsequently transferred to a membrane filter disk by filtration and then examined microscopically. Microscopical analysis of the contaminant is conducted at two magnifications using a gating measurement technique with oblique incident lighting. Particles are counted in three size ranges: >100 µm, 25 to 100 µm, 5 to 25 µm, and fibers. For low-contamination levels on irregularly shaped components, a procedure for running a blank is described. The method required strict adherence to the procedures for cleaning apparatus.

4. Apparatus

4.1 *Microscope*, with mechanical stage, approximately 45 and 100×. For 100× magnification, the recommended objective is 10 to 12× (but a minimum of 6×) with a numerical aperture of 0.15 minimum. The optimum equipment is a binocular microscope with a micrometer stage. A stereomicroscope should not be used in this procedure.

4.2 *Ocular Micrometer*, B & L 31-16-10, or equivalent.

4.3 *Stage Micrometer*, B & L 31-16-99, or equivalent, having 0.1- to 0.01-mm calibration.

4.4 *Light Source*—An external incandescent high-intensity, 6-V, 5-A source with transformer.

4.5 *Microscope Slides*—Glass slides 50 by 75 mm.

4.6 *Plastic Film*[2]—Wash with membrane-filtered isopropyl alcohol.

4.7 *Solvent Filtering Dispenser.*[3]

4.8 *Membrane Filter Holder*, having 47-mm diameter and heat-resistant glass base.[4]

4.9 *Filter Flask*, 1 L.

4.10 *Membrane Filters*,[5] having 47-mm diameter, 0.45-µm pore size, black, grid marked.

4.11 *Vacuum Source*—Pump or aspirator (tap recommended).

4.12 *Flat Forceps*, with unserrated tips.

4.13 *Plastic Petri Dishes.*[6]

[1] This method is under the jurisdiction of ASTM Committee E-21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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[2] Saran plastic film or equivalent has been found satisfactory for this purpose.

[3] Millipore Type XX 66 02500, manufactured by the Millipore Filter Corp., or equivalent, has been found satisfactory for this purpose.

[4] Millipore XX 1004700 or equivalent has been found satisfactory for this purpose.

[5] Millipore Type HABG 04700 manufactured by the Millipore Filter Corp., or equivalent, has been found satisfactory for this purpose.

[6] Millipore PD 10 04700 or equivalent has been found satisfactory for this purpose.

NOTE 2—Plastic petri dishes should not be reused for conducting these tests.

4.14 *Ultrasonic Energy Cleaning Apparatus*, having 2-L minimum capacity (see Appendix X1).

4.15 *Beaker*, 500-mL, chemical-resistant glass.

4.16 *Double-Faced Pressure-Sensitive Tape*.

5. Reagents

5.1 *Isopropyl Alcohol*, ACS reagent grade, membrane-filtered.

5.2 *Nonionic Liquid Wetting Agent*, membrane-filtered.

5.3 *Water*—Deionized or distilled water, membrane-filtered.

NOTE 3—Membrane-filtered reagents shall be stored in bottles pre-cleaned as described in 7.2.1 or by use of Swinney hypodermic filters in a Guth bottle. A procedure for control analysis of reagent cleanness is described in Appendix X2.

6. Determination of Background Counts

6.1 Prepare a blank by following steps 7.2.1-7.2.10, without introduction of the part, for the purpose of determining background counts (which are to be subtracted from the final counts when parts are used). If excessively high background counts are obtained, cleaning procedures and handling should be reexamined before proceeding.

7. Preparation of Test Specimens

7.1 *For Planar Surfaces*:

7.1.1 Prepare a 50- by 75-mm microscope slide by adhering to it a 25- by 50-mm strip of double-faced masking tape. With clean forceps, carefully remove the component to be tested from its container and place it on the tape.

7.1.2 Perform a particle count in accordance with Section 8.

7.2 *For Irregular Surfaces*:

7.2.1 Ultrasonically clean all glassware, storage containers, and filter holders in hot water containing a detergent. After washing, rinse the equipment with membrane-filtered water and membrane-filtered isopropyl alcohol (Note 4) and drain dry. For use at low-contamination levels, check the cleanness of the equipment by conducting successive blank analyses (Note 5).

NOTE 4—Isopropyl alcohol final rinse is optional.

NOTE 5—Wash bottles for providing membrane-filtered water and solvents may be constructed by attaching a Swinney adapter containing a 0.8-µm membrane filter to the base of the outlet tube of a Guth wash bottle.

7.2.2 Carefully remove the component to be analyzed from its container with clean forceps and place it in a clean 500-mL beaker containing 200 mL of membrane-filtered distilled water to which 0.1 % by volume of a nonionic wetting agent has been added. Cover the beaker with clean plastic film. Place the beaker in the ultrasonic tank filled to the proper level with water. Apply ultrasonic energy to the system for 5 min.

7.2.3 Preclean a 0.45-µm black grid filter 47 mm in diameter by holding it with forceps and gently rinsing the filter surface with a stream of prefiltered distilled water from the wash bottle.

7.2.4 Place the filter on the fritted base of the filter holder and clamp the funnel portion in place.

7.2.5 Remove the extracted part from the beaker and rinse thoroughly with membrane-filtered water allowing the water to run into the beaker.

7.2.6 Transfer the fluid from the beaker into the funnel of the filter holder. Rinse the beaker with 50 mL of filtered water, or solvent and add this rinse to the funnel.

7.2.7 Cover the funnel with a piece of clean aluminum foil or a cleaned 150-mm glass petri dish.

7.2.8 Apply a vacuum to the filter flask until the liquid has completely passed through the filter. Do not add additional fluid to the funnel after the filter surface has become clear of liquid as this will upset the particle distribution on the filter.

7.2.9 Turn off the vacuum, remove the filter from the holder base with a forceps, and place the filter in a plastic petri dish with the cover ajar. Label the dish and allow the filter to dry for at least 30 min.

NOTE 6—If the filter curls on the slide, apply a thin coat of silicone grease to the slide under the filter. Alternatively, the filter dish may be sandwiched between ultrasonically cleaned glass slides.

7.2.10 When ready for the microscopical analysis, transfer the filter with a forceps to the surface of a 50- by 75-mm glass microscope slide.

NOTE 7—Storage of filters in a glass petri dish permits forced drying at temperatures of 60 to 70°C and allows more rapid sample handling.

7.2.11 Repeat 7.2.1-7.2.10 with the same part (stored in clean container) for the purpose of determining the percentage of removable particles removed during the first run.

NOTE 8—Parts shall be stored in a clean, tight, ultrasonically cleaned container until test preparations have been completed.

8. Procedure

8.1 Calibrate the micrometer eyepiece scale with a stage micrometer for each magnification.

8.2 Count and tabulate particles in the following order of size ranges: fibers, greater than 100 µm, 25 to 100 µm, and 5 to 25 µm.

8.3 Conduct the count within a clean environmentally controlled area having limited access.

8.4 Adjust the microscope focus and lamp intensity to obtain maximum particle definition.

8.5 Use a 100× magnification for counting particles in the 5- to 25-µm range and a 45× magnification for particles greater than 25 µm.

8.6 Scan the entire component surface or the effective area of the filters at each magnification and count the particles. Use the ocular micrometer linear scale as a gate, counting the appropriate size particles as they pass the gate while scanning by means of the mechanical stage. After each lateral scan, move the stage vertically a distance equal to the length of the micrometer scale, using the filter grids or component surface (or grids on overlay cover glass) as a guide for positioning. If the number of particles is in excess of 50 in any size range, the statistical counting technique, outlined in Appendix X2, may be used.

9. Number of Tests

9.1 For both types of surfaces, the number of test specimens measured is governed by the dimensions of the component or

surface being analyzed.

10. Interpretation of Results

10.1 Read the number of particles of each size range as particles per component or as particles per square centimetre of component surface.

11. Keywords

11.1 optical particle counting; particulate contamination; size distribution analysis; surfaces

APPENDIXES

(Nonmandatory Information)

X1. SELECTION OF ULTRASONIC EQUIPMENT

X1.1 To provide uniform and reliable ultrasonic energy, the following factors must be considered in selecting the equipment:

X1.1.1 The type of transducer and method of bonding must be selected so that frequency, inductance, and coupling coefficient are unchanged by heat, vibration, and age.

X1.1.2 The type of transducer and frequency should be selected to minimize focusing of energy in specific areas.

X1.1.3 The power of the transducer should be chosen to prevent standing waves (which do not allow vaporous cavitation) and to prevent physical damage and cavitation erosion.

X2. METHOD OF COUNTING AND MEASURING PARTICLES

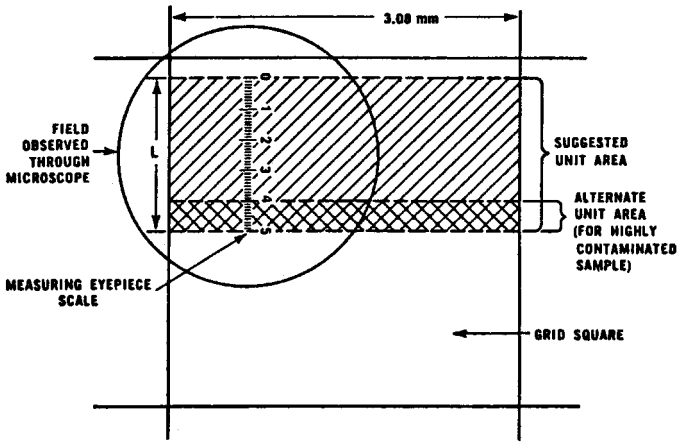
X2.1 In obtaining the number of particles of a certain size range, the number of particles on a representative number of grid squares on the membrane filter paper is counted. From this count, the total number of particles, which would be present statistically on the total filtered area of 100 imprinted grid squares, is calculated.

X2.2 If the total number of particles of a certain size range is estimated to be between 1 and 50, count the number of particles on all 100 grid squares.

X2.3 If the total number of particles of a certain size range is estimated to be between 50 and 1000, count the number of particles in 20 randomly chosen grid squares and multiply this number by 5 to obtain the statistical total particle count.

X2.4 If the total number of particles of a certain size range is estimated to be between 1000 and 5000, count the number of particles on 10 randomly chosen grid squares and multiply this number by 10 to obtain the statistical total particle count.

X2.5 If the estimated total number of particles of a given size range exceeds 5000, particles are counted in standard fractions of grid areas (Fig. X2.1). Count the particles within at least 10 of these fractional areas. The average count per fractional area is multiplied by the ratio of the effective filtration area, to the area counted. Select fractional area so that there will be no more than about 50 particles of a size range.



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

FIG. X2.1 Alternative Unit Areas