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Standard Method for DETERMINING THE QUALITY OF CALIBRATION PARTICLES FOR AUTOMATIC PARTICLE COUNTERS[1]

This standard is issued under the fixed designation F 322; 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.

1. Scope

1.1 This method is applicable to the examination of essentially spherical near-monodisperse particles, in sizes ranging from 7.5 to 100 μm, of known density such as polystyrene or glass, to determine whether they are suitable for calibrating automatic particle counters for liquid or gaseous suspensions.

2. Applicable Documents

2.1 *ASTM Standards*:

A 555 Specification for General Requirements for Stainless and Heat-Resisting Steel Wire[2]

D 1193 Specification for Reagent Water[3]

D 2021 Specification for Neutral Detergent, 40 % Alkylbenzene Sulfonate Type[4]

E 319 Testing Single-Arm Balances[5]

F 323 Practice for Precision Classification of Particles[6]

F 490 Microscopical Sizing and Counting Particles on Membrane Filters Using Image Shear[6]

3. Summary of Method

3.1 The weight average diameter of the particles is measured by suspending a known weight in a suitable liquid, removing them onto a membrane filter, and counting them under a microscope.

3.2 The range of diameters, the ellipticity, and the mean visible diameter are measured by a microscopical sizing method.

3.3 The oblateness is calculated from the measured parameters.

3.4 This method contains two procedures, to match two ways particles are shipped:

3.4.1 Procedure A applies to particles shipped dry.

3.4.2 Procedure B applies to particles in aqueous suspension.

4. Significance

4.1 This method enables the user to accept or reject shipments of calibration particles by criteria that are relatable to National Bureau of Standards measures and weights, and that are not dependent on the use of an automatic particle counter.

NOTE 1—For example, this method can be used to verify the product from Practice F 323.

5. Terminology

5.1 *Descriptions of Terms*:

5.1.1 *weight average diameter* D_w—the diameter calculated from the weight and density of a counted number of particles, μm.

5.1.2 *major axis*, *A*—the longest dimension of a particle, μm.

5.1.3 *second axis*, *B*—the shortest dimension of a particle as seen under the microscope, μm.

5.1.4 *minor axis*, *C*—the shortest dimension of a particle, not seen under the microscope because it is vertical, μm.

5.1.5 *range of diameters*, ΔA—the difference

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[1] This method is under the jurisdiction of ASTM Committee F-7 on Aerospace Industry Methods and is the direct responsibility of Subcommittee F07.01 on Contamination.

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[2] *Annual Book of ASTM Standards*, Part 3.

[3] *Annual Book of ASTM Standards*, Part 31.

[4] *Annual Book of ASTM Standards*, Part 30.

[5] *Annual Book of ASTM Standards*, Part 41.

[6] *Annual Book of ASTM Standards*, Part 25.

between the largest and smallest major axes of a field of 100 or more particles, μm.

5.1.6 *ellipticity, E*—the mean percentage difference from circular cross section in a field of 100 or more particles, %.

5.1.7 *mean visible diameter, D_o*—the average of the geometric means ($\sqrt{AB}$) of the visible axes in a field of 100 or more particles, μm.

5.1.8 *oblateness, F*—the mean percentage difference from prolate spheroidal volume, as calculated from D_w and D_o, in a field of 100 or more particles, %.

5.2 *Symbols*:

5.2.1 N = number of particles in filter funnel.

5.2.2 n = number of particles actually counted or measured.

5.2.3 D = diameter of bottom of filter funnel, cm.

5.2.4 N_S = number of squares counted on membrane.

5.2.5 A_S = area of each square, cm^2.

5.2.6 W = weight of particles delivered to funnel, μg.

5.2.7 d = density of material used to prepare particles, g/cm^3.

6. Apparatus

6.1 *Microbalance*, accurate to 0.005 mg. The zero shall not drift more than 0.005 mg during the test period. The rated accuracy shall be obtainable by personnel acutally making the weighings, under actual conditions of use and shall be verified in accordance with Method E 319.

6.2 *Calibration Weights*, for microbalance, 10 and 20 mg accurate to 0.005 mg and made from nonmagnetic material conforming to Type 310 in Specification A 555. The accuracy shall be traceable to the National Bureau of Standards as discussed in their Technical Note 288.

6.3 *Air Ionizer*, alpha emitter, 18.5 MBq of polonium-210, with a useful life of 1.5 years to a final value of 1.1 MBq.[7]

6.4 *Ultrasonic Bath*, with 100-cm^3 mixing vessel, 30 W, 20 to 200 kHz operation.

6.5 *Measuring Pipets*, 1.0, 5.0, 25.0, and 100-cm^3, 100 divisions on each size.

6.6 *Filtration Funnel*—The funnel opening in contact with the membrane shall be approximately 35.0 mm in inside diameter. The effective area shall be calibrated.

6.7 *Membrane Filter Support*—Either a fritted-glass, sintered-metal, or stainless steel screen may be used. The support shall be so designed as to enable attachment to a vacuum flask.

6.8 *Funnel-Holding Device*—A provision should be made for the dissipation of static electricity from the funnel.

6.9 *Funnel Cover*, to prevent extraneous contamination. An inverted watch glass is suitable.

6.10 *Vacuum Source*, minimum vacuum reading of 18 in. Hg (61 kPa).

6.11 *Forceps*, unserrated.

6.12 *Filtered Liquid Dispenser*, with 1.2-μm absolute pore size or finer filter.

6.13 *Membrane Filters*, 47 to 51-mm, 0.45 or 0.2-μm pore size. The filter shall have an imprinted grid on 3.10 ± 0.02-mm centers. The color shall be chosen to provide maximum contrast with the particles.

6.14 *Microscope*, capable of resolving the smallest particles to be counted and producing a flat field of view.

6.14.1 The following optic combinations are recommended:

Magnification	Ocular	Objective	Minimum Numerical Aperture
50×	10×	5×	0.15
100×	10×	10×	0.25
200×	10×	20×	0.50

Similar ocular-objective combinations resulting in magnifications of 50 ± 10×, 100 ± 10×, and 200 ± 20× may be used. The optimum equipment is a compound binocular microscope. Conventional stereomicroscopes will not meet these requirements.

6.15 *Mechanical Stage*, capable of traversing the entire effective filter area.

6.16 *Stage Micrometer*, with 0.1 and 0.01-mm subdivisions. The accuracy shall be traceable to the National Bureau of Standards, as discussed in their Technical Note 288.

6.17 *Illumination*—Provisions for variable high-intensity external oblique incident illumination and for a focusing condenser. A flexible or jointed arm is desirable.

6.18 *Tally Counter*, hand-operated, for recording particle counts.

6.19 *Petri Dishes*, covered, plastic or glass,

[7] May be obtained from any source approved by the U.S. Nuclear Regulatory Commission.