W-63-13

Designation: F 651 – 80

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103
Reprinted from the Annual Book of ASTM Standards, Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

Standard Method for PARTICLE COUNTER SINGLE-POINT CALIBRATION BY THE MEDIAN METHOD[1]

This standard is issued under the fixed designation F 651; 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 describes an instrument that counts particles suspended in fluid, within a specified size range and in random order. This procedure is intended for use by skilled operators.

1.2 A single-point calibration is generated, primarily for field use.

1.3 The purpose of this test is to make a single adjustment of the system scale factor. The adjustment is to compensate or accommodate a multiplicative variable such as transducer gain drift, or other operational factors such as aperture size changes or source lamp changes.

1.3.1 If there are additive effects they must be corrected in separate steps. Normally this is done with a zero or off-set adjustment of the output baseline voltage. When these are two or more significant effects or interactions, the median procedure must be repeated with different sized particles. In general, the procedure should be performed with one test for each degree of freedom of the output.

1.3.2 The series of tests may be repeated with different particle sizes until the calibration curve is entirely reconstructed. For multiple size tests one size is used to fix the nominal gain (preferably near full-scale size), and the remaining procedure is done with a measured, varying threshold voltage instead of a varying gain.

2. Summary of Method

2.1 Standard material with particles of known median size is sampled by the instrument under test. The instrument net gain is adjusted until an average of half the particles of the standard material are counted above a fixed threshold. The threshold is set to the known median size of the particles.

2.2 The instrument must have one gain designated to adjust either the system scale factor (gain) or, equivalently, the particle pulse detecting thresholds. If there is more than one threshold, they must change in proportion.

2.3 The field use of the procedure consists only of three steps, iterated until the desired accuracy is obtained.

2.3.1 All of the particles larger than a fixed minimum size are counted for a given sample quantity of the standard material.

2.3.2 Next, or simultaneously, all of the particles greater than a threshold size (voltage) are counted. The threshold is set at the median size (voltage) of the particle distribution for the distribution region above the minimum size used in 2.3.1.

2.3.3 The second measurement is compared with the first. If the system gain is correct, it will be 50 % of the first count. If the second count differs by more than a calculated or given error, the system gain (or the threshold of 2.2) is adjusted to correct the error. The procedure is then repeated until the error is less than or equal to the allowable maximum.

2.4 This procedure includes cases where the standard material median particle size differs from fixed instrument thresholds. Additional

[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.

Current edition approved March 3, 1980. Published July 1980.

apparatus is used to temporarily count particles with a threshold equal to the particle median size, if a continuously variable or predetermined matching threshold is not available.

2.5 Individual instrument manufacturers should be consulted to identify specific controls and outputs suited to this procedure.

3. Significance

3.1 This procedure applies only to a single parameter of a particle counter, the scale factor. It does not test any change in linearity nor does it test counting accuracy. It is exact only for the material tested, at the size tested. It may be related to other materials with reference to comparative tests with those materials. It may be related to other sizes by interpolation of the calibration curve for the instrument.

3.2 In general, the particle counter may not be operated using the same thresholds as used for this procedure. This requires that there be an accurately known relationship between the calibration threshold and the operating threshold.

3.2.1 Since there is presently no way to manufacture large numbers of small particles with a predetermined median size, instrument calibration must accommodate procedures that involve interpolation, and temporary thresholds that will be transferred to new levels for operation.

3.2.2 For example, a standard material may have a particle median of 5.678 μm, while the instrument is operated with a counter threshold of 5.0 μm. Assume that the instrument is arbitrarily designed to a scale of 5.0 μm equals 1.0 V, and that the calibration curve in the vicinity of 5.0 μm is 0.38 V/μm. Then the threshold voltage for a median of 5.678 μm is $1.0 + 0.678 \times 0.38 = 1.2576$ V. Nonlinear interpolation may be required if the calibrate and operating points differ by large amounts. These values should be either obtained from the instrument manufacturer, determined at least once by the extended procedure of 1.3.2, or determined by any complete primary calibration.

3.3 *Classes of Standard Material*—The significance of the calibration depends on the type of particles used and their original calibration. This procedure allows accurate calibration for a wide variety of particles. The instrument calibration documentation should identify the class of standard material and the source of original calibration because later tests with materials different from the calibration material may result in errors. The class (see 4.2) limits the significance of the calibration for each material.

4. Terminology

4.1 *Descriptions of Terms*:

4.1.1 *channel*—discrete range of sizes to be counted.

4.1.2 *debris*—uncontrolled and undefined material in the standard material.

4.1.3 *discriminator*—electronic device that produces a countable pulse whenever a particle generated pulse exceeds a given threshold.

4.1.4 *equivalent diameter*—diameter assigned to a particle equal to the diameter of a particle of the standard material producing the same pulse at the discriminator.

4.1.5 *gain*—multiplier of the scale factor.

4.1.6 *reference region*—range of particle sizes where the count density is negligible, below the size of the test particles, and above most of the debris.

4.1.7 *scale factor*—volts per unit size as measured at the discriminator for a given size.

4.1.8 *standard material*—total material chosen that includes both the test particles and any debris in the original material, and in subsequent dilution fluids.

4.1.9 *test particles*—portion of the standard material whose size properties are known and which are counted by the instrument.

4.1.10 *threshold*—voltage or corresponding particle size above which all pulses or particles are counted.

4.1.11 *specifier's document*—one that provides criteria for tolerances, materials, etc. When this method is specified, a list of numerical values, materials, and steps should be supplied for incorporation in the user's document. Choices that are the user's option should be listed.

4.1.12 *user's document*—one that requires different specific steps for each type or model of particle counter used. When a user's document is needed, it must identify the options to be selected, for example, parallel or series counts. Specific hardware should be identified: for example, "the 100 % threshold is controlled by R36, the sensor gain is controlled by CAL ADJ, Aerosol Generator S/N 1234 is the laboratory standard." Specific voltages, tolerances.

and other numerical values are to be taken from the specifier's document and the instrument manufacturer's data.

4.2 *Classes of Particles*:

4.2.1 *Class A material*—spherical particles with symmetrical distribution of the primary particles around the median, where the median, mode, and mean are the same, and there are negligible particles in the reference region or at the double size.

4.2.1.1 Class A material has easily characterized particles which are the type usually used for the instrument manufacturer's calibration. Accuracy that may equal or exceed the counter manufacturer's is attainable.

4.2.2 *Class B material*—spherical particles with an asymmetric distribution of the primary particles. The median size is approximated by the mode or mean, or by the median size of a different parameter. An example is: measurement of the area mean vis-a-vis the volume median.

4.2.2.1 Class B materials have limited accuracy due to a change in parameters. This transfer error is a secondary effect of the particle distribution width. When the distribution of the primary particles is narrow, the transfer error will be a small fraction of the width of the distribution. An example of the transfer error would be the error in estimating the median diameter when the mean volume is known.

4.2.3 *Class C materials*—particles of arbitrary shape, chosen to represent the normally measured material. The material may have a significant number of particles in the reference region. Then a unique threshold must be specified for the 100 % count, *N*, to be repeatable. Further, the count of very large particles may be truncated with a specified upper limit to both the 100 % and median, *M*, channels.

4.2.3.1 Class C particles give calibrations that are good only for specified materials; for example, blood cells or powdered minerals. The calibration is not to be applied to other materials without research and documentation. The instrument manufacturer may supply the relationship of Class A materials to specific Class C materials. Accuracy for specific materials may be greater for the Class C materials than for Class A materials.

4.3 *Symbols*:

N = total count above reference region.
M = observed count above median threshold.
m = predicted count above median threshold.
E_a = allowable error, specified by authority, counts.
E = measurable error of counting, counts.
E_d = error in size, %.
i = 1, 2, 3, index of successive measurements or calculations.
$\bar{E}$ = bar symbol is average value.
σ = standard deviation of size, μm.
D = true median diameter, μm.
d = specific diameter in particle size distribution, μm.
n = specific number of particles $\geq d$ in diameter.

5. Apparatus

5.1 *Particle Source*—The method of supplying particles to the instrument under test will incorporate apparatus to supply particles at a given rate, and include any apparatus necessary to keep the particles separate and suspended in the medium.

5.1.1 Aerosols and hydrosols are usually unstable, and specific measures must be taken to assure that the properties of the test particles do not vary during the test. If the particle counts of Section 9 are done simultaneously, the particle concentration need only be controlled between an upper limit where coincidence effects occur, and a lower limit where statistical accuracy is affected.

5.1.2 If sequential counts are used, the rate of change of particle concentration must be limited, or known. If the distribution of the particle sizes is narrow, then variation in the count has a proportionally decreased effect on the size determination. See 11.3.1.

5.2 *Ultrasonic Bath*, 100 cm^3 mixing capacity, 30 W, 20 to 200 kHz.

5.3 *Pulse Height Discriminators and Counters*, is not part of the specimen instrument. If there is only one discriminator or counter, it must be able to switch from 100 % threshold to median threshold rapidly in comparison with the average rate of change of particle concentration.

6. Reagents and Materials

6.1 *Test Particles*—The cumulative size distribution is required to have the following two features—a region of zero or near zero slope,