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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Calibration of Liquid-Borne Particle Counters for Submicrometer Particle Sizing¹

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^{ε1} NOTE—Keywords were added editorially in September 1994.

1. Scope

1.1 This test method describes a procedure for establishing the sizing accuracy of an automatic, optical liquid-borne single particle counter, using light scattering automatic particle counter (APC). This test method is directed at determining the sizing accuracy of the APC when it is used to measure a challenge suspension of precisely-sized spherical isotropic particles, particularly those sized at and below 1 μ m in diameter.

1.2 The particle size parameter that is reported is the equivalent diameter based on the projected area of an isotropic spherical particle of known composition suspended in a liquid that is optically different from the suspended particle. Particles in the size range of 0.1 μ m and 5 μ m are used for calibration in this procedure.

1.3 This test method does not provide a procedure for APC counting accuracy calibration, since that procedure is available in Practice F 658. However, some knowledge of APC maximum concentration capability is necessary in order to avoid introduction of sizing errors as a result of excessive particle concentration during the sizing calibration procedure.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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. For hazard statement, see Section 9.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 20 Practice for Particle Size Analysis of Particulate

Substances in the Range of 0.2 to 75 μ m by Optical Microscopy³

F 658 Practice for Defining Size Calibration, Resolution, and Counting Accuracy of a Liquid-Borne Particle Counter Using Near-Monodisperse Spherical Particulate Material⁴

2.2 Other Standard:

ANSI B93.20 Procedure for Qualifying and Controlling Cleaning Methods for Hydraulic Fluid Power Fluid Sample Containers⁵

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *agglomerate*—two or more particles in intimate contact that cannot be separated by normal mixing or stirring and the resultant small shear forces generated by such normal sample handling.

3.1.2 *coincidence*—the presence of more than one particle within the viewing volume of the APC at any time, resulting in the production of a single data pulse from the APC indicating the presence of a single particle of projected area equal to the sum of the areas of those particles present in the viewing volume. This is a consistent error that produces a bias that will increase directly with particle concentration and inversely with viewing volume dimensions of the APC.

3.1.3 *equivalent diameter*—the diameter of a sphere of equal projected area or volume to the particle measured and with optical properties similar to those of the calibration material. The particle diameter reported by an APC is based on calibration data for the particular APC.

3.1.4 *saturation level*—the pulse counting rate limit for the electronic components of a particular APC. At a rate greater than that limit, the recovery time between pulses is not adequate for the APC electronics to clear itself in order to accept subsequent pulses. As a general rule of thumb, the rate limit for pulses arriving at random time intervals, as can be expected from particles randomly distributed within a liquid, will be about one-tenth that for uniformly spaced pulses.

¹ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.10 on Contamination Control.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol 10.05.

⁵ Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

4. Summary of Test Method

4.1 *In-line Sensor Calibration*—A series of suspensions of precisely-sized near-monodisperse spherical calibration particles are prepared in liquid of composition nearly identical to that normally flowing through the APC sensor. They are injected into the flowing liquid stream upstream of the sensor and sized by the sensor. The particle size of the calibration particles is determined beforehand, either by microscopic observation as in Practice E 20 for those particles large enough to be so sized, by using NIST standard reference materials (for example, SRM 1690 nominal 1 μm or SRM 1691 nominal 0.3 μm), or by using secondary standard calibration material referenced by the supplier. The response of the APC to the calibration spheres is recorded, in terms of pulse height, along with data that can be used to determine the standard deviation of the APC output pulse-height distribution. The particle concentration for this measurement is maintained sufficiently low so that the APC coincidence error is less than 3 %, based on the manufacturer's specification for sensor coincidence versus particle concentration levels (See Note 1).

NOTE 1—Do not confuse coincidence error with saturation level. The coincidence error is a function of concentration only (as long as the particle size is insignificant in comparison to the sensing zone size), while the counting rate is a function of both concentration and liquid flow rate. Thus, it is possible to exceed the saturation level of the electronic system by using too high a flow rate, even with acceptable concentration in the liquid. In addition to the possibility of too high a counting rate for the electronic system, note that the higher flow rate will result in faster data pulses with short rise times. For electronic systems with limited bandwidth, the short rise time pulses will result in distorted data output, indicating particles smaller than those actually present.

4.2 *Volumetric or Batch-Analyzing Sensor Calibration*—A series of suspensions of precisely-sized near-monodisperse spherical calibration particles is prepared in clean reagent grade water. The particle size of the precision-sized calibration particles is determined beforehand, either by microscopic observation as in Practice E 20 for those particles large enough to be so sized, by using NIST SRM (for example, SRM 1690 nominal 1 μm or SRM 1691 nominal 0.3 μm), or by using secondary standard calibration material referenced by the supplier. Reagent grade water is flowed through the APC at its rated flow rate by pumping through a membrane type filter of pore size less than the minimum size that the APC will define. The stream of clean water is passed into an open container that is connected directly to the APC inlet port. The APC outlet port may be used as the supply point for the pump filter system. The entire assembly is maintained in an ultra clean environment. A suspension of the calibration material is added to the open container and the particles in that material are sized by the APC. The response of the APC to the precision spheres is recorded, in terms of pulse height, along with data that can be used to determine the standard deviation of the APC output pulse-height distribution. The particle concentration for this measurement is maintained sufficiently low so that the APC coincidence error is less than 3 %, based on the manufacturer's specification for sensor coincidence versus particle concentration levels (See Note 1).

5. Significance and Use

5.1 Manufacturing processes for many electronic, pharmaceutical and mechanical devices require extreme particulate cleanliness, both in process liquids and in cleaning operations where flushing liquids are used. If the liquid contains particulate material whose size is so large or in concentration sufficient to interfere with manufacture or subsequent operation of the device, then yield and performance are degraded. Accurate measurement of the number and size of particles in the liquid must be carried out. Acceptance of data from the APC used to characterize the liquid cleanliness includes reliance on the integrity of the APC for defining the size of any particle present in samples of liquid passed through the APC.

5.2 Although other procedures exist for calibration of APCs for liquid-borne particle measurement, the development of APCs capable of submicrometre measurement has made it necessary to prepare a procedure that addresses the special problems of handling these very small particles. In particular, the difficulties of cleaning the sample handling equipment are not adequately solved in other test methods.

5.3 Care is required to make sure that good sampling is accomplished from a well-mixed liquid and that no artifacts are introduced in the sample acquisition, storage or handling processes. These precautions are necessary both for operation of this test method and for normal APC use.

5.4 Note that materials of equal size but with differential refractive index other than that of the calibration particles in water may produce data that differ from that of the calibration particles.

6. Interferences

6.1 Ingestion of miscellaneous debris during this test causes errors. In the course of testing with in-line APCs, when either normal liquid flow lines or sample flow tubing has been in use for long time periods, or has been inadequately cleaned before use, bursts of submicrometre particles may be released during flexing. These may be surface film fragments produced by local stress concentration or they may be previously deposited particles released by disturbance of the boundary layer during flexing. When testing with batch sampling or volumetric APC, it is necessary to ensure that ingestion of airborne particles in size ranges near to those in use does not occur during the test. Operation in an environment controlled by adequate filtration is required. Use either a horizontal or a vertical flow clean bench. Make sure that any liquid containers, closures or tubing used in storing, mixing, diluting, transporting, etc., the fluid samples are clean. Adherence to the requirements of ANSI B93.20 is recommended to state definitively the cleanliness of these items.

6.2 The presence of air or vapor bubbles during this test causes erroneous data. When in-line measurements are being made, make sure that the liquid is under sufficient pressure to cause such bubbles to remain in solution, or make sure that a bubble control trap is used. When the calibration particle suspension has been prepared, it is necessary to mix the material well in order to ensure that the particles are uniformly distributed throughout the sample before removing any aliquots from that sample. If a surface active dispersant has been