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Standard Test Method for Calibration Verification of Laser Diffraction Particle Sizing Instruments Using Photomask Reticles[1]

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INTRODUCTION

There exists a large variety of techniques and instruments for the sizing of particles and droplets in fluid suspension. These instruments are based on a number of different physical phenomena and interlaboratory comparisons of data on, for example, reference liquid sprays have shown significant variability. This test method evolved in conjunction with efforts to explain the observed variability. The effectiveness of this test method can be traced to the fact it circumvents difficulties associated with producing, replicating, and maintaining a standard sample of liquid particles in a spray. This test method uses a photomask reticle to provide a simulation of some of the optical properties of a reference population of spherical particles. This test method is only applicable to optical particle sizing instruments that are based on measurement and analysis of light scattered in the forward direction by particles illuminated by a light beam. Since modern optical instruments generally use a laser to produce a light beam, and since the light scattered in the forward direction by particles can often be accurately described using diffraction theory approximations, the class of instruments for which this test method applies have become generally known as laser diffraction particle sizing instruments. Because it is specifically Fraunhofer diffraction theory[2], [3] that is used in the approximation, these instruments are also known as Fraunhofer diffraction particle sizing instruments.

The diffraction approximation to the general problem of electromagnetic wave scattering by particles is strictly valid only if three conditions are satisfied. The conditions are: particle sizes must be significantly larger than the optical wavelength, particle refractive indices must be significantly different than the surrounding medium, and only very small (near-forward) scattering angles are considered. For the case of spherical particles with sizes on the order of the wavelength or for large scattering angles, the complete Lorenz-Mie scattering theory[2], [3] rather than the Fraunhofer diffraction approximation must be used. If the size and angle constraints are satisfied but the particle refractive index is very close to that of the medium, the anomalous diffraction approximation[3] may be used.

A complication is introduced by the fact that the optical systems of most laser diffraction particle sizing instruments can be used, with only minor modifications such as changing a lens or translating the sample, for measurement configurations outside the particle size or scattering angle range for which the diffraction approximation is valid. In this situation the scattering inversion software in the instrument would generally incorporate a scattering model other than Fraunhofer diffraction theory, in which case the term "laser diffraction instrument" might be considered a misnomer. However, such an instrument is still in essence a laser diffraction instrument, modified to decrease the lower particle size limit. A calibration verification procedure as described by this test method would be applicable to all instrument configurations (or operational modes) where the photomask reticle accurately simulates the relevant optical properties of the particles.

The ideal calibration test samples for laser diffraction particle sizing instruments would be comprised of the actual particle or droplet material of interest in the actual environment of interest with size distributions closely approximating those encountered in practice. However, the use of such calibration test samples is not currently feasible because multi-phase mixtures may undergo changes during a test and because actual samples (for example, a spray) are not easily collected and stabilized for long periods of time. The subject of this test method is an alternative calibration test sample comprised of a two-dimensional array of thin, opaque circular discs (particle artifacts) deposited on a transparent substrate (the photographic negative, that is, clear apertures in an opaque substrate, may

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be used as well). Each disc or particle artifact represents the orthogonal projection of the cross-section of one member of a population of spherical particles comprising the reference population. The collection of particle artifacts on a reticle represents an orthogonal projection of all the particles in the reference population for one particular three-dimensional arrangement of the population where the member particles are positioned within a finite reference volume. The reference volume is generally defined such that the area covered by particle artifacts on the reticle is roughly equivalent to the cross-section of the instrument light beam. The reference population would generally contain a large number of particles, with a size distribution that approximates distributions of practical interest, randomly distributed over the reference volume. Large numbers and random positions minimize complications that can arise from optical coherence effects (interference).

Of importance here is the fact that the near-forward scattering characteristics of the orthogonal projections of the particle cross-sections onto the reticle plane accurately simulate, in regimes where the diffraction approximation is valid, the near-forward scattering characteristics of the reference population (independent of the chemical composition of the particles in the reference population). In other words the photomask reticle, when illuminated with a laser beam of known properties, generates a reference scattered light signature which can be predicted analytically from a knowledge of the size distribution of the reference population. The properties of the reference population can be inferred from a characterization (using optical microscopy) of the sizes of the particle artifacts on the reticle. As the instrument is operated away from the diffraction regime, the scattering properties of the photomask reticle diverge from that which would be produced by the reference population and interpretation of the measurements becomes more problematic.

The most complete test result for this test method would be a discrete size distribution reported for a very large number of size class intervals, but intercomparisons of such distributions are difficult. For that reason statistical parameters (for example, representative diameters and measures of the dispersion) of the particle size distribution are used. Two examples of statistical parameters are the volume median diameter $D_{V0.5}$ and the relative span $(D_{V0.9} - D_{V0.1})/D_{V0.5}$ as defined in Practice E799 (recall that volume parameters such as D_{Vf} for a photomask reticle are defined in the sense that two-dimensional particle artifacts scatter light like spherical particles of the same diameter). Estimates of the true values of these statistical parameters for a photomask reticle (or more precisely the true values for the reference population simulated by the reticle) can be established using optical or electron microscope measurements of the diameters of the particle artifacts on the reticle. The values so established are termed image-analysis reference values and will be used herein as the accepted reference values. It is the stability of $D_{V0.5}$, the relative span, and all other statistical parameters representative of the particle artifact size distribution for a reticle and the ability to produce nearly identical replicate copies of the reticles that make this test method useful. A comparison of the accepted reference value of $D_{V0.5}$, the relative span, or any other parameter of a reticle with a corresponding test result from the instrument under evaluation can be used to assess the acceptability of the instrument and of the data routinely obtained with the instrument.

[1] This test method is under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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[2] Bohren, C. F. and Huffman, D. R. *Absorption and Scattering of Light by Small Particles*, John Wiley and Sons, New York, 1983.

[3] van de Hulst, H. C. *Light Scattering by Small Particles* , Dover Publications Inc., New York, 1981.

1. Scope

1.1 This test method describes a procedure necessary to permit a user to easily verify that a laser diffraction particle sizing instrument is operating within tolerance limit specifications, for example, such that the instrument accuracy is as stated by the manufacturer. The recommended calibration verification method provides a decisive indication of the overall performance of the instrument at the calibration point or points, but it is specifically not to be inferred that all factors in instrument performance are verified. In effect, use of this test method will verify the instrument performance for applications involving spherical particles of known refractive index where the near-forward light scattering properties are accurately modeled by the instrument data processing and data reduction software. The precision and bias limits presented herein are, therefore, estimates of the instrument performance under ideal conditions. Nonideal factors that could be present in actual applications and that could significantly increase the bias errors of laser diffraction instruments include vignetting[4] (that is, where light scattered at large angles by particles far away from the receiving lens does not pass through the receiving lens and therefore does not reach the detector plane), the presence of

[4] Hirleman, E. D., Oechsle, V., and Chigier, N. A., "Response Characteristics of Laser Diffraction Particle Sizing Systems: Optical Sample Volume and Lens Effects," *Optical Engineering*, Vol 23, 1984, pp. 610–619.

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nonspherical particles, the presence of particles of unknown refractive index, and multiple scattering.

1.2 This test method shall be used as a significant test of the instrument performance. While the procedure is not designed for extensive calibration adjustment of an instrument, it shall be used to verify quantitative performance on an ongoing basis, to compare one instrument performance with that of another, and to provide error limits for instruments tested.

1.3 This test method provides an indirect measurement of some of the important parameters controlling the results in particle sizing by laser diffraction. A determination of all parameters affecting instrument performance would come under a calibration adjustment procedure.

1.4 This test method shall be performed on a periodic and regular basis, the frequency of which depends on the physical environment in which the instrumentation is used. Thus, units handled roughly or used under adverse conditions (for example, exposed to dust, chemical vapors, vibration, or combinations thereof) shall undergo a calibration verification more frequently than those not exposed to such conditions. This procedure shall be performed after any significant repairs are made on an instrument, such as those involving the optics, detector, or electronics.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *This standard does not purport to address all of the safety problems, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[5]

A340 Terminology of Symbols and Definitions Relating to Magnetic Testing

D123 Terminology Relating to Textiles

D3244 Practice for Utilization of Test Data to Determine Conformance with Specifications

E131 Terminology Relating to Molecular Spectroscopy

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E284 Terminology of Appearance

E456 Terminology Relating to Quality and Statistics

E799 Practice for Determining Data Criteria and Processing for Liquid Drop Size Analysis

E1187 Terminology Relating to Conformity Assessment[6]

2.2 *Military Standard:*

MIL-STD-45662 Calibration Systems Requirements[7]

2.3 *NIST Standard:*

NIST SP 676-1 Measurement Assurance Programs[8]

2.4 *ANSI Standard:*[9]

ANSI-ASQC Z-1 Standard for Calibration Systems[9]

2.5 *ISO Standard:*[10]

ISO Guide 2A General Terms and Their Definitions Concerning Standardization Certification, and Testing Lab. Accreditation

3. Terminology

3.1 *Current ASTM Standard Definitions*—Definitions of the terms listed below, as used in this test method are from the *Compilation of ASTM Standard Definitions*,[11]:

3.1.1 *accuracy*—see Terminology D123, (Committee D-13).

3.1.2 *assignable cause*—see Terminology E456, (Committee E-11).

3.1.3 *bias*—see Terminology D123, (Committee D-13).

3.1.4 *calibration*—see Terminology E1187, (Committee E-36).

3.1.5 *Discussion*—This and many other commonly used definitions for calibration are very broad in the sense that they could encompass a wide range of tasks. (See for example MIL-STD-45662, NBS (NIST) SP 676I, and ANSI ASQC Z-1 Draft Standard for Calibration Systems). For example, in some cases *calibration* is only the determination of whether or not an instrument is operating within accuracy specifications (*tolerance testing* in NBS SP 676I). In other cases *calibration* includes reporting of differences between the instrument response and the accepted value of the standard, for example, to produce a "Table of Corrections" to be used with the instrument. Finally, *calibration* can also include any repairs or adjustments required to make the instrument response consistent with the standard within the stated accuracy specifications. To clarify the situation it is proposed that the more specific terms *calibration verification* and *calibration adjustment* (see 3.4) both of which would fall under these broad definitions of calibration.

3.1.6 *coefficient of variation*—see Terminology D123, (Committee D-13). Also known as the *relative standard deviation* (see Terminology E135, Committee E-1).

3.1.7 *reference material*—see Terminology E1187, (Committee E-36) (see ISO Guide 2, *A*).

3.1.8 *scattering*—see Terminology E284, (Committee E-12).

3.1.9 *standard reference material*—see Terminology E131, (Committee E-13).

3.1.10 *test method*, *n*—see Terminology D123, (Committee D-13).

3.1.11 *test method equation*—see Terminology D123, (Committee D-13).

[5] 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.

[6] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

[7] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[8] Available from National Institute for Standards and Technology, Gaithersburg, MD 20899.

[9] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[10] Available from ISO, 1 rue de Varembé, Case Postale 56, Crt 1221, Geneve 20, Switzerland.

[11] *Compilation of ASTM Standard Definitions*, 7 th edition, ASTM, Philadelphia, PA, 19103, 1990.

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3.1.12 *test result*—see Terminology D123, (Committee D-13).

3.1.13 *tolerance limits, specification or calibration*—see Terminology A340, (Committee A-6).

3.1.14 *verification*—see Terminology E135, (Committee E-1).

3.2 *Other ASTM Definitions*—Definitions of the terms given below are either close derivatives of definitions in the *Compilation of ASTM Standard Definitions*,[10] or are given in ASTM Standards approved after that time.

3.2.1 *accepted reference value*—a value that serves as an agreed-upon reference for comparison, and that is derived as: (*1*) a theoretical or established value, based on scientific principles, (*2*) an assigned value, based on experimental work of some national or international organization such as the U.S. National Institute of Standards and Technology (or its predecessor the National Bureau of Standards), or (*3*) a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group. (See Terminology E456.)

3.2.2 D_{vf}— a diameter such that the fraction, *f*, of the total volume of particles contains precisely all of the particles of smaller diameter. (Derivative of that in Practice E799.)

3.2.3 *precision, n, general*—see Terminology D123, (Committee D-13).

3.2.4 *precision, n, single-operator*—the single-operator-laboratory-sample-apparatus-day precision of a method; the precision of a set of statistically independent test results all obtained as directed in the method and obtained over the shortest practical time interval in one laboratory by a single operator using one apparatus. (Derivative of Terminology D123, Committee D-13.)

3.2.5 *precision, between laboratory*—the multi-laboratory, single-sample, single-operator-apparatus-day (within-laboratory) precision of a test method; the precision of a set of statistically independent test results all of which are obtained by testing the same sample of material and each of which is obtained in a different laboratory by one operator using one apparatus to obtain the same number of observations over the shortest practical time interval. (Derivative of Terminology D123, Committee D-13.)

3.2.6 *precision, within-laboratory (multi-operator)*—the multi-operator, single-laboratory-sample, single-apparatusday (within operator) precision of a test method; the precision of a set of statistically independent test results all obtained in one laboratory using a single sample of material and with each test result obtained by a different operator with each operator using one apparatus to obtain the same number of observations over the shortest practical time interval. (Derivative of Terminology D123, Committee D-13.)

3.2.7 *repeatability, repeatability limit*—see Terminology E456, (Committee E-11).

3.2.8 *reproducibility, reproducibility limit*— see Terminology E456, (Committee E-11).

3.3 *Definitions From Other Sources:*

3.3.1 *calibration*—comparison of a measurement standard or instrument of known accuracy with another standard or instrument to detect, correlate, report, or eliminate by adjustment, any variation in the accuracy of the item being compared. (See MIL STD-45662.)

3.4 *Definitions Established in This Test Method:*

3.4.1 *calibration adjustment, for instruments*— the process of adjusting any of the various sensitivity settings or parameters of an instrument to restore the instrument performance to within tolerance limit specifications.

3.4.2 *calibration verification, for instruments*— the process of comparing the response of an instrument or a subsystem of an instrument to the accepted value of a standard of greater accuracy (less uncertainty) for the purpose of evaluating the performance of the instrument with respect to stated precision and bias specifications.

3.4.3 *Discussion*—The failure of an instrument to indicate the value of a standard to within the stated uncertainties of the instrument and standard would suggest corrective action, such as a calibration adjustment.

3.4.4 *image-analysis reference value (for a photomask reticle)*—a reference value for a test result derived from theoretical calculations based on measurements of the sizes of particle artifacts on the reticle.

3.4.5 *reference population (for a photomask reticle)*—a finite population of particles of specified sizes for which a photomask reticle represents an orthogonal projection of one particular three dimensional arrangement of the population.

3.4.6 *Discussion*—Since there are many possible ways to distribute a finite particle population over a finite volume, there are likewise many different photomask reticle configurations that can represent a given reference population.

3.4.7 *reference volume (for a photomask reticle)*— the hypothetical, finite volume within which the reference population of particles represented by the reticle are placed.

3.4.8 *true value (for a photomask reticle)*—a value corresponding to a property of the reference population.

4. Significance and Use

4.1 This test method permits a user to compare the performance of an instrument to the tolerance limit specifications stated by a manufacturer and to verify that an instrument is suitable for continued routine use. It also provides for generation of calibration data on a periodic basis, forming a database from which any changes in the performance of the instrument will be evident.

4.2 This test method for the calibration verification of laser diffraction particle sizing instruments is suitable for acceptance testing of laser diffraction instruments so long as current estimates of the bias (see Section 11) and the between-laboratory precision of the test method (see Section 10) are acceptably small relative to typical laser diffraction instrument accuracy specifications; see Practice D3244.

5. Apparatus

5.1 *Laser Diffraction Instrument*:

5.1.1 *Discussion*—A laser diffraction particle sizing apparatus generally consists of a laser source to produce a beam of light, optical means for producing a suitable beam that passes through a region of the particle field, means for detecting the laser energy scattered by the particles into a multiplicity of collection angles, and means for transforming the measurements

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