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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 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. 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

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[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.

[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.

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 standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E799 Practice for Determining Data Criteria and Processing for Liquid Drop Size Analysis
E1187 Terminology Relating to Conformity Assessment (Withdrawn 2006)[6]

2.2 *Military Standard:*[7]

MIL-STD-45662 Calibration Systems Requirements

2.3 *NIST Standard:*[8]

NIST SP 676-1 Measurement Assurance Programs

2.4 *ANSI Standard:*[9]

ANSI-ASQC Z-1 Standard for Calibration Systems

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 D13).

3.1.2 *assignable cause*—see Terminology E456, (Committee E11).

3.1.3 *bias*—see Terminology D123, (Committee D13).

3.1.4 *calibration*—see Terminology E1187, (Committee E36).

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, NIST SP 676-1, 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 NIST SP 676-1). 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 D13). Also known as the *relative standard deviation* (see Terminology E135, Committee E01).

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

[7] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

[8] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

[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 International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

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

3.1.7 *reference material*—see Terminology E1187, (Committee E36) (see ISO Guide 2A).

3.1.8 *scattering*—see Terminology E284, (Committee E12).

3.1.9 *standard reference material*—see Terminology E131, (Committee E13).

3.1.10 *test method, n*—see Terminology D123, (Committee D13).

3.1.11 *test method equation*—see Terminology D123, (Committee D13).

3.1.12 *test result*—see Terminology D123, (Committee D13).

3.1.13 *tolerance limits, specification or calibration*—see Terminology A340, (Committee A06).

3.1.14 *verification*—see Terminology E135, (Committee E01).

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 D13).

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 D13.)

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 D13.)

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 D13.)

3.2.7 *repeatability, repeatability limit*—see Terminology E456, (Committee E11).

3.2.8 *reproducibility, reproducibility limit*—see Terminology E456, (Committee E11).

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 observations into statistical estimates of particle size distribution characteristics. In obtaining particle size calibration verification data using this test method, the analyst shall select the proper instrument operating conditions to realize satisfactory instrument performance. Operating conditions for individual instruments are best obtained from the operation manuals provided by the manufacturer because of variations in instrument designs.

5.2 *Photomask Reticle:*

5.2.1 *Discussion*—There are typically thousands of particles or droplets in the optical sample volume of a laser diffraction particle sizing instrument during a measurement period. These large numbers are the result of the relatively large (line-of-sight) optical sample volume and are necessary to ensure adequate statistical sampling of the distribution and to minimize coherent scattering effects. A photomask reticle is designed to simulate the near-forward scattering properties of a specified, finite population of spherical particles (the reference population) randomly distributed within a hypothetical finite volume (the reference volume). The photomask reticle represents an orthogonal projection of the cross-sections of all the spherical particles in the reference population onto a plane. The projected area of the reference volume, that is, the area covered by particle artifacts on the reticle, is normally approximately equivalent to the cross-section of the instrument light beam. The reference population generally contains a large number of particles with a size distribution that approximates distributions of practical interest. (Large numbers and random positions are necessary to ensure that the scattering contributions from the individual particle artifacts sum incoherently.)

5.2.1.1 A perfect simulation of a real particle or droplet system would require a continuous distribution of particle sizes, but multiple replications of a limited number of discrete particle sizes (primary particle sizes) may be used to approximate an actual size distribution on photomask reticles. The number of replications of the various primary sizes of the particle artifacts is specified in order to provide a discrete approximation to the desired size distribution of particles or droplets. The photomask reticle used in the ILS for this test method had 23 discrete primary sizes with from one to several thousand replications of these sizes.

5.2.2 *Particle Artifacts*—A photomask reticle shall have a number of thin circular discs (particle artifacts) deposited on a substrate.

5.2.3 *Clear or Background Area*—A photomask reticle shall have an area at least as large as the laser beam used by the instrument that is free from particle artifacts. This region of the photomask reticle is used for the background measurement to zero the detectors.

5.2.4 *Substrate*—The reticle substrate shall be of optical quality since it is used in a transmission mode. Antireflection coatings will minimize the possibility of spurious readings due to reflections from the reticle reaching the detectors.

5.3 *Accepted Reference Values for a Photomask Reticle:*

5.3.1 *Discussion*—In order to verify the performance of an instrument in an absolute sense it is necessary to calculate the bias of the instrument, that is, the difference between a measured value and the true value that for this test method corresponds to the reference population. Although a photomask reticle is only a projection of the reference particle population, image analysis of the array of particle artifacts on a photomask reticle combined with information on the design of the reticle can provide good estimates of the true values for the reference particle population. Reference values so obtained are termed image-analysis reference values. Since it may be impractical to measure the sizes of thousands of particle artifacts, only a representative sample of measurements may be available. However, estimates based on an incomplete sample would have uncertainty resulting from bias and precision errors in measurements of the size of the individual particle artifacts, and also from uncertainties in inferring properties of the entire population of particle artifacts from the sample, that is, statistical sampling errors.

5.3.1.1 Further uncertainty results from the fact that an orthogonal projection of a three-dimensional arrangement of randomly-positioned spherical particles will generally result in overlapping images. The image analysis method used to characterize overlapped (and thus noncircular) images may produce different results than the scattering mechanism.

5.3.2 *Specification of Accepted Reference Value:*

5.3.2.1 The procedure used to determine the accepted reference value for a photomask reticle used in this test method shall be specified. If the accepted reference value is based on image-analysis the following shall be specified:

5.3.2.2 *Size Values for Nonoverlapping (Circular) Artifacts*—The method for assigning a size to nonoverlapping (circular) artifacts shall be specified. One possible measure of size is the maximum chord in some preferred direction (that is, the Ferret diameter).[12]

5.3.2.3 *Size Values for Overlapping (Noncircular) Artifacts*—The method for assigning a size to overlapping (noncircular) artifacts shall be specified. Additional uncertainty is introduced in the process of assigning a size to a nonspherical or noncircular artifact, as there are many possible approaches.[11]

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[12] Allen, T., *Particle Size Measurement*, 4th edition, Chapman and Hall, London, 1990.

5.3.2.4 *Statistical Sampling*—If only a subset of the particle artifacts on the reticle were measured and the sizes of the remaining members of the particle population were inferred statistically, then the procedure shall be specified. For example, sizes for unmeasured particle artifacts might be determined based on the assumption of a Gaussian within-primary-size-class distribution function.

5.3.2.5 *Calculating Representative Diameters*—Calculation of representative diameters D_{vf} using image-analysis data shall be performed according to Practice E799.

6. Reference to This Calibration Procedure

6.1 Reference to this practice in documents relating to a laser diffraction particle sizing instrument shall constitute due notification that the adequacy of instrument performance has been evaluated by means of this test method. Performance is considered to be adequate when test results are in agreement with the accepted reference value of the photomask reticle taking into account, according to Practice D3244, the repeatability and reproducibility limits of this test method given in Section 10.

NOTE 1—A successful calibration verification using this test method will not ensure that all data obtained with the instrument will be meaningful. Data obtained while operating an instrument outside the prescribed operating parameters may be invalid. For example, data obtained from measurements in optically dense aerosols where no correction for multiple scattering has been made will generally be invalid.

7. Test Observations, Test Determinations, and Test Results

7.1 *Discussion*—Specifying a test result for a particle size distribution measurement is more complicated than for many test methods where only a single parameter (for example, mass, length) is desired. A particle size distribution function is, in general, a continuous function but no practical measurement system has infinite resolution as required to measure a complete, continuous distribution. Further, a general size distribution function is particle frequency versus particle diameter, but there are several measures of particle population frequencies of interest depending on the application. For example, while the number distribution is commonly used, the surface area distribution (second moment of the number distribution) is important in catalyst studies, and the volume distribution (third moment) is important in fuel spray combustion.

7.1.1 Laser diffraction instruments sense the angular distribution of scattered optical energy at some finite number of angles (the test observations) and then utilize these observed values in mathematical inversion schemes to estimate the particle size distribution (a test determination). Since many engineering problems (for example, development of correlations) require a relatively small number of parameters and since laser diffraction instruments inherently sample a subset of the particles of interest and are therefore statistical in nature, the use of representative statistical parameters of the size distribution determinations as a test result is common and is used in this test method. The various representative mean diameters discussed in Practice E799 are examples of typical statistical parameters.

7.1.2 The necessarily finite resolution of particle sizing instruments requires that the measured size distribution either be represented as a discrete histogram of frequency for a finite number of size class intervals, or be specified by a small number of parameters (say two to four) of an analytic or parametric size distribution function. For that reason an important aspect of a laser diffraction measurement is the computational procedure used to obtain test results from the actual observations.

7.2 *Test Observations*—The test observations consist of the following two parts:

7.2.1 The set of measured scattered energy levels over a range of discrete scattering angles, and

7.2.2 The measured optical extinction (that is equal to 1-*T* where *T* is transmittance or the fraction of the laser energy beam transmitted directly through the medium).

7.3 *Test Determinations*—The test determinations consist of either of the following (7.3.1 is preferred over 7.3.2):

7.3.1 A discrete histogram of particle quantity (number, area, or volume) in a finite number of discrete size class intervals, or

7.3.2 The parameters specifying an analytic size distribution function (for example, the Rosin-Rammler distribution or other analytic functions discussed in Practice E799).

7.4 *Test Result*—A test result for this test method consists of the following statistical parameters representative of the particle size distribution function:

7.4.1 The volume median diameter $D_{V0.5}$ defined in Practice E799, and

7.4.2 The relative span (volume basis) given by $(D_{v0.9} - D_{V0.1})/D_{0.5}$ as defined in Practice E799.

7.4.3 All test determinations and calculations of all test results must be consistent with Practice E799.

8. Procedure

8.1 *Discussion*—In a test method to verify the state of calibration of an instrument there are only two possible conclusions, either the instrument is operating within specified tolerance limits or it is not. To arrive at the latter conclusion requires that the bias of the instrument (that is, the difference between the true value for the reticle and the average of some number of test results on that reticle) be greater than the specified tolerance limit (with some appropriate level of confidence allowing in part for random measurement errors that affect the test result) plus the total uncertainty of the true value. In this test method the true value is not known, and the comparison of test results is with the accepted reference value that is generally based on some form of image-analysis. In that context there are three possible causes for an apparent instrument bias: the instrument is malfunctioning and requires calibration adjustment or other service, the instrument is operating properly but the accepted (image-analysis) reference value for the test material (reticle) is biased (that is, differs from the true value), or the instrument is operating properly and the accepted reference values are unbiased, but the test method or test material alters or masks, from the instrument's point-of-view, the true value. The second and third possibilities would be shortcomings of the test method; clearly it would be inappropriate to judge an instrument as out of tolerance if the bias in the calibration verification was larger than the apparent instrument bias.

8.1.1 This procedure consists of three basic parts, preparation of the apparatus (see 8.2), the procedure to obtain a test result (see 8.3 and 8.4), and checking the test results for conformance with tolerance limit specifications to make a pass/fail judgment on the instrument calibration (see 8.5).

8.2 *Apparatus and Preparation*—It is necessary to consider the following items before attempting a calibration verification experiment based on this test method:

8.2.1 *Reticle Positioning Apparatus*—The positioning apparatus for the reticle shall have two translational degrees of freedom allowing motion in the plane normal to the optical axis of the instrument. Further, one rotational degree of freedom (about an axis perpendicular to the optical axis) should also be available to help eliminate the effects of reflections as discussed in 8.2.4 below. Position the reticle adjacent to the receiving lens.

8.2.2 *Reticle Cleaning*—Contamination (for example, fingerprints or foreign particles) on the reticle in the areas used for either the background or signal measurements will scatter light and cause a bias in the test result. It is important that the reticle be free of contamination before a test is attempted. Reticles should be cleaned using standard methods for precision transmission optics such as lenses or windows before each test.

8.2.3 *Reticle Care*—Scratches on the reticle in the areas used for either the background or signal measurements will scatter light and cause a bias in the test result. Inspect the reticle for damage in these critical areas prior to each test.

8.2.4 *Reticle Orientation (Tilt Angle)*—Placing a planar substrate in an optical beam will produce reflections, and it is mandatory to ensure that these reflections do not reach the detector of the laser diffraction instrument. Direct the reflections away from the detector by purposefully tilting the reticle. Verify the effectiveness of tilting the reticle in directing the reflections away from the detector by varying the tilt angle slightly and ensuring that the detector outputs do not vary systematically and significantly.

8.2.4.1 Observation of the position where the beam reflected off the front face (laser side) of the reticle strikes the transmission optics (for example, laser housing or collimating lens holder) is a convenient way to set the tilt angle. The amount of tilt required is a balance between the need to move the reflections off the detector, and the need to avoid alteration of the diffraction pattern at large angles where the particle artifacts present themselves as ellipses to the beam. Tilt angles of a few degrees have been found to be a good compromise since reflection effects can be eliminated and experimental data have shown[4] no appreciable effect on the reticle scattering properties. The range of acceptable tilt angles will depend on the specific laser diffraction instrument configuration and shall be determined by the operator for each instrument configuration.

8.3 *Procedure (After Completing 8.2 – 8.2.4):*

8.3.1 *Discussion*—After completing the preparatory items in 8.2, obtain a single test result by obtaining a background reading, a signal reading, and then computing the result. To minimize the effects of random electronic noise, both background and signal data for the various detectors should be averaged over more than 50 observations for each detector signal. A subsequent independent test result would require another reading of both background and signal which would require physically moving the reticle between tests.

NOTE 2—If the reticle is repositioned between runs to give identical readings on a micrometer stage then the runs would not be truly independent, as any bias resulting from a particular reticle position would be repeated in the measurement.

8.3.2 A single test result is obtained by the following:

8.3.2.1 *Background Reading*—A background reading is necessary to cancel the scattering contribution of the substrate on which the particle artifacts are deposited.

8.3.2.2 Move the photomask reticle to a position such that the light beam passes through a clear area thereby striking no particle artifacts. This positioning may be accomplished by translating the reticle a distance dictated by the layout of the reticle pattern. A loss in scattering signal is an indication that the laser beam is passing through a clear area. Do not choose the background area by eye as the scattering from the reticle glass surface may not be reliable due to the low levels of intensity in the edges of the beam.

8.3.2.3 Acquire a background distribution averaging over more than 50 signal readings for each detector.

8.3.3 *Signal Measurement*—Center the particle artifact region of the reticle in the laser beam. Perform the centering by one of the following means, with method 8.3.3.1 preferred:

8.3.3.1 Obtain coarse resolution centering by maximizing the extinction (also called obscuration) reading that indicates the amount of light scattered out of the beam. This will occur when the reticle is very near the center of the beam. Then complete the centering process by maximizing the scattering signal integrated over all detectors. If centering according to preferred method 8.3.3.1 is not possible, then the following methods (8.3.3.2 and 8.3.3.3) may also be used:

8.3.3.2 Visually center the laser beam in the particle sample region of the reticle by observing the apparent intensity of the scattered light from the particles. Published data[4] indicate that visual centering of the reticle is possible with a repeatability of better than 0.5 mm that typically will result in less than 1 % variation in measured representative diameters.

8.3.3.3 Use centering crosshairs on the reticle (for example, by aligning an image of the reticle to a centered pattern).

8.3.3.4 Verify the alignment of the instrument at this stage to ensure that introduction of the reticle has not affected the centering of the transmitted beam that is situated on-axis (centered) in the detector plane.

8.3.3.5 Acquire a signal distribution averaging over more than 50 signal readings for each detector.

8.4 *Data Processing*—The diffraction signal observations consisting of signal less background for each of the detectors are used as input to the data processing algorithm of the instrument. The data processing step shall conform to the following:

8.4.1 *Zeroing*—Adjust the signal measured in 8.3.3 to correct for the background level by subtracting, detector by