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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

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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 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
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).

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

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

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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.

3.1.9 *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]

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,

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

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 detector, the background distribution 8.3.2 from the signal distribution in 8.3.3. This process is equivalent to what is often termed “zeroing” the detectors. The resulting distribution (the adjusted signal distribution) is used by data processing schemes.

8.4.1.1 *Discussion*—The light received at the detector of a laser diffraction instrument when the particle artifact region of a photomask reticle is in the beam has contributions from the following six sources: (*1*) light scattered by the particle artifacts; (*2*) light scattered by imperfections on or within the photomask reticle substrate in the region where the particle artifacts are deposited; (*3*) laser light scattered by molecules and/or contaminant particles in the laser beam; (*4*) laser light scattered off of optical elements; (*5*) stray light deriving from the environment of the measurement (for example, room lights), and (*6*) light in the laser beam that passes unscattered through the optical system. Ideally only the first contribution (from the particle artifacts) is of interest, and the process of subtracting a background and a signal measurement is designed to obtain an adjusted signal distribution that has only Contribution (*1*) as desired. In the procedure specified in 8.3.2, a background measurement is made with the laser beam passing through a region of the photomask substrate where there are no particle artifacts. The only light received by the detectors in this background measurement will be from Contributions (*3*), (*4*), (*5*), and (*6*). Since in the signal measurement in 8.3.3 these same contributions should be present and unchanged, subtraction of background from signal as specified in 8.4.1 should eliminate Contributions (*3*), (*4*), (*5*), and (*6*) from the adjusted signal distribution.

8.4.1.2 The most difficult task is to eliminate Contribution (*2*). It is not possible to isolate the scattering contribution from the surface or bulk substrate material just under the particle artifacts from the scattering by the artifacts themselves. However, to the extent that the substrate and the contaminants are homogeneous, both the background 8.3.2 and signal 8.3.3 distributions will include a constant Contribution (*2*). Under this assumption, the adjusted signal distribution from 8.4.1 would then represent the desired differential contribution from the particle artifacts alone.

8.4.2 *Size Distribution Model Selection*—Select the mathematical scheme to be used in data processing. The data processing shall utilize a scheme that makes no assumption about the functional form of the particle size distribution. Computational algorithms that make no such assumption are sometimes referred to as “model independent.” If no “model independent” scheme is available, then an alternative may be used but the results shall be so labelled.

8.4.3 *Scattering Model Specification*—Specify the light scattering model used by the instrument software in the inversion process as one that adequately models scattering by the particle artifacts on the photomask reticle. Achieve this using one of the following methods:

8.4.3.1 If the instrument allows selection of a scattering model valid for two-dimensional circular disks (that is, a diffraction model) then specify that model, or

8.4.3.2 If the instrument allows or requires specification of the relative refractive index of the particles, then specify or select a refractive index with high values for both the real and imaginary parts, (that is, greater than 1.5 for the real and greater than 1.0 for the complex components, respectively).

8.4.3.3 If the instrument does not explicitly allow specification of a scattering model corresponding to either 8.4.3.1 or 8.4.3.2, then the desired result may still possibly be obtained by forcing the instrument to utilize a scattering model valid for the largest particle sizing range (that is, for the longest focal length receiving lens) even though the particles fall in a smaller size range corresponding to a shorter focal length lens. This is done by “telling” the instrument (for example, through computer input) that the longest available focal length lens is being used in the optical system, even when a shorter focal length (better matched) lens is actually used in the experiments. After data processing, the size classes and representative mean diameters will be systematically and artificially large, each multiplied by a constant factor equal to the ratio of the specified (that is, what the instrument was “told”) lens focal length to the focal length of the lens actually used in the experiments. For example, if a 300 mm lens was optimal for a particular photomask reticle and was used in the experiments, while the instrument was “told” that a 600 mm focal lens was in use, then the final results so obtained would originally be given in terms of size classes corresponding to the 600 mm lens (that is, using the instrument matrix for the 600 mm lens). The representative diameters would need to be corrected, in this case reduced by the ratio 300/600 or by one-half.

8.4.3.4 *Discussion*—Far-field, near-forward scattering by the particle artifacts (thin metallic discs) on a reticle is accurately modeled by scalar diffraction theory using the Fraunhofer approximation to the Fresnel-Kirchoff integral.[3] For that reason, if the instrument invokes an anomalous diffraction approximation or uses the full Lorenz-Mie theory valid for spheres, then a bias between the true value and the test results may be introduced. Specification of large refractive index components will make any anomalous diffraction correction insignificant and cause Lorenz-Mie calculations to approach the diffraction approximation.

8.4.3.5 Forcing the instrument to internally utilize data processing schemes valid for long focal length lenses is one method to ensure that the instrument data processing computations are consistent with Fraunhofer diffraction theory. For a given detector geometry, long focal length lenses correspond to small scattering angles, that in turn correspond to large particles where the Fraunhofer diffraction approximation is valid.

8.4.4 *Test Determination and Test Result*—Perform data processing on the adjusted signal distribution (that is, the test observation) to obtain a test determination. Use further data processing to obtain a test result from the test determination according to Section 7.

8.5 *Calibration Verification:*

8.5.1 Compare the test results with corresponding accepted reference values for the photomask reticle to check conformance of the instrument to tolerance limit specifications following Practice D3244.

8.5.1.1 *Acceptance*—If the test results fall within the acceptance range according to Practice D3244, the instrument calibration is verified and no further action is required.

8.5.1.2 *Rejection*—If the test results fall within the rejection range according to Practice D3244 then the instrument calibration state is not verified and corrective service or calibration adjustment shall be performed by a qualified person. If the user attempts this repair or adjustment task then the manufacturer's recommended procedure shall be carefully followed.

9. Report

9.1 Report data obtained by this test method in such manner as to clearly distinguish between observed data, including the use of conversion factors customarily employed, and interpretive results such as are obtained by curve-fitting procedures requiring judgment. Report the following information:

9.1.1 The instrument manufacturer, model, and serial number,

9.1.2 The receiving lens focal length used in acquiring the scattering signal measurements,

9.1.3 The software version number used in data acquisition and processing,

9.1.4 The particle size distribution model assumed in data processing (for example, model independent, Rosin-Rammler, lognormal, see 8.4.2),

9.1.5 The scattering model used in data processing. Depending on the specific method from 8.4.3 used to select the scattering model, specify the following:

9.1.5.1 If 8.4.3.1 was used to select the scattering model, specify the manufacturer's name for the applicable scattering model (for example, Fraunhofer diffraction theory), or

9.1.5.2 If 8.4.3.2 was used to select the scattering model, specify the refractive index used, including both real and imaginary parts, or

9.1.5.3 If 8.4.3.3 was used to select the scattering model, specify: the particle or experiment type used in data processing (for example liquid droplet spray, solid particles), and the receiving lens focal length used in data processing (that is, the "software" focal length that in this case is different from the "hardware" focal length from 9.1.2),

9.1.6 The photomask reticle model and serial number, and

9.1.7 The measured and accepted reference values for the test results, including specification of (or bibliographic reference to) the methods used to determine the accepted reference values (see 5.3.2).

10. Precision and Bias[13]

10.1 *Precision of This Test Method:*

10.1.1 *Interlaboratory Study*—An interlaboratory study (ILS) of this test method was carried out over a two year period beginning in March, 1988. Data from twenty-one different laser diffraction instruments of nine different models in thirteen different laboratories were included. Practice E691 was followed and analysis of the data is given in the research.[12] The laboratories were requested to obtain four test results on two nominally-identical photomask reticles for three receiving lens focal lengths. Table 1 summarizes the results of the ILS in terms of precision statistics as defined in Practice E691.

10.1.2 *95 % Repeatability Limit (Within-Laboratory)*—The repeatability limits, *r*, from Table 1 expressed as a percentage of the test result values are shown in Table 2.

10.1.3 *95 % Reproducibility Limit (Between-Laboratory)*—The reproducibility limits, *R*, from Table 1 expressed as a percentage of the measurement are shown in Table 3.

10.1.4 *Assignable Causes*—The likely assignable causes for single-operator imprecision in tests on laser diffraction particle sizing instruments using a photomask reticle include the following:

TABLE 1 Precision Statistics (Statistical Quantities Defined in Practice E691)

Test Result	*f* (mm)	Reticle Serial Number	$\bar{X}$	S_r	S_R	*r*	*R*
$D_{V0.1}$ (μm)	63	246	24.26	0.57	2.37	1.59	6.64
$D_{V0.1}$ (μm)	63	247	24.11	0.40	2.44	1.12	6.83
$D_{V0.1}$ (μm)	100	246	24.56	0.35	1.29	0.98	3.60
$D_{V0.1}$ (μm)	100	247	24.61	0.19	1.57	0.52	4.39
$D_{V0.1}$ (μm)	300	246	26.13	0.42	1.29	1.18	3.62
$D_{V0.1}$ (μm)	300	247	26.06	0.36	1.21	1.01	3.41
$D_{V0.5}$ (μm)	63	246	46.20	0.20	1.44	0.57	4.02
$D_{V0.5}$ (μm)	63	247	46.44	0.19	1.56	0.54	4.38
$D_{V0.5}$ (μm)	100	246	45.96	0.15	1.63	0.42	4.57
$D_{V0.5}$ (μm)	100	247	45.71	0.21	1.61	0.58	4.50
$D_{V0.5}$ (μm)	300	246	45.92	0.23	1.54	0.65	4.32
$D_{V0.5}$ (μm)	300	247	45.95	0.22	1.74	0.62	4.88
$D_{V0.9}$ (μm)	63	246	81.99	0.79	12.86	2.22	36.01
$D_{V0.9}$ (μm)	63	247	83.41	0.79	14.30	2.20	40.05
$D_{V0.9}$ (μm)	100	246	74.18	0.90	6.01	2.51	16.84
$D_{V0.9}$ (μm)	100	247	72.65	0.36	6.39	1.01	17.89
$D_{V0.9}$ (μm)	300	246	76.72	2.04	7.82	5.71	21.88
$D_{V0.9}$ (μm)	300	247	76.55	3.89	7.53	10.90	21.09
Span (V)	63	246	1.25	0.02	0.25	0.05	0.70
Span (V)	63	247	1.27	0.01	0.28	0.03	0.79
Span (V)	100	246	1.08	0.02	0.12	0.05	0.34
Span (V)	100	247	1.05	0.01	0.12	0.02	0.34
Span (V)	300	246	1.10	0.04	0.14	0.12	0.38
Span (V)	300	247	1.10	0.08	0.13	0.24	0.37

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[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E29-1000. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Repeatability Limits (95 % Confidence)

Focal Length (mm)	$D_{V0.1}$ (µm), %	$D_{V0.5}$ (µm), %	$D_{V0.9}$ (µm), %	Span (V), %
63	5.6	1.2	2.7	3.2
100	3.1	1.1	2.4	3.2
300	4.2	1.4	10.8	16.0

TABLE 3 Reproducibility Limits (95 % Confidence)

Focal Length (mm)	$D_{V0.1}$ (µm), %	$D_{V0.5}$ (µm), %	$D_{V0.9}$ (µm), %	Span (V), %
63	27.9	9.1	46.0	59.1
100	16.3	9.9	23.7	32.3
300	13.5	10.0	28.0	34.0

10.1.4.1 Time-dependent contamination of the photomask reticle by dust, oil, or other foreign material,

10.1.4.2 Variations in the positioning and orientation (that is, relative to the laser beam) at which the reticle is presented to the instrument for background and signal measurements between successive tests,

10.1.4.3 Time-dependent (random) electronic noise,

10.1.4.4 Time-dependent (random) optical noise (background light),

10.1.4.5 Time-dependent speckle noise resulting from changes in the laser oscillation mode, and

10.1.4.6 Time-dependent variations in the laser beam intensity profile.

10.2 *Bias of This Test Method:*

10.2.1 *Discussion*—The bias of a test method is the difference between the mean of a very large set of independent test results obtained with the test method and the true value. Since the true value is unknown in this case, the bias considered here is based on an accepted reference value determined using image-analysis, and an estimate of this apparent bias was obtained through the ILS. Three contributions to this apparent bias can be identified and these are discussed in 10.2.2 – 10.2.4.

10.2.2 *Laser Diffraction Instrument Bias*—The bias between a test result and the true value due to imperfect instrument performance. It is this bias term that a calibration verification procedure hopes to isolate. Bias errors for a single instrument can derive from the assignable causes listed below. Different instruments would, in general, have independent levels of bias from these causes, and for that reason an ILS with a very large number of independent instruments should produce a mean bias of zero from these contributions. The bias sources include the following:

10.2.2.1 Optical misalignment of the instrument,

10.2.2.2 Vignetting,

10.2.2.3 Variations in the responsivities of individual elements in multi-element detector arrays,

10.2.2.4 Variations in the gains of amplification stages on systems where each detector has individual signal conditioning,

10.2.2.5 Differences in the scattering model used by the instrument and the actual light scattering process. This could be significant in the cases of unknown or incorrectly assumed particle refractive index, nonspherical particles, multiple scattering, numerical approximation and/or numerical solution errors in the instrument software, and various sources of noise in the measurements.

10.2.3 *Test Procedure or Test Material Bias, or Both*—Introduced by the test procedure or test material, or both, this bias between the true value and the mean of a large number of test results would still exist even if a "perfect" instrument was used with a "perfectly-characterized" photomask reticle, and is therefore not the "fault" of the instrument. This bias contribution results, in part, from the inability of a two-dimensional simulation (a reticle) to capture all the information contained in a three-dimensional particle field, and from the addition of measurement errors due solely to presence of a photomask reticle in the system. (Note that the magnitude of these bias terms will be very instrument-dependent). Assignable causes of this bias component include:

10.2.3.1 Variations in the substrate properties between the sample region (that is, the substrate under the particle image field) and the background clear area designated for background reading. This causes the actual diffraction pattern (signal minus background) produced by the reticle to systematically differ from that corresponding to the actual size distribution on the reticle.

10.2.3.2 *Bias Due to Nonuniform Intensity Distribution of the Laser Beam*—A radial (transverse to the beam propagation direction) intensity variation will result in nonuniform weighting of reticle particle artifacts by a laser diffraction instrument, with weighting factors proportional to the local incident irradiance on each artifact. An image-analysis system would generally weight all particles in the field-of-view equally. This effect would not normally be present when analyzing a dynamic particle sample more characteristic of normal applications (for example, a spray or flow cell) where particles of all sizes would spend (on average) equal amounts of time in all regions of the laser beam.

10.2.3.3 *Bias Associated With Simulating Spherical Particles Falling in the Same Line-of-Sight*—There are an infinite number of possible arrangements for any specified particle population, but a two-dimensional projection loses all information on particle positions along the line-of-sight. In other words, the mapping from a two-dimensional projection (on which an image analysis is based) to a three-dimensional field is nonunique or multivalued. In contrast, a laser diffraction system will be weakly sensitive to particle position along the line-of-sight insofar as dependent or interactive scattering, that occurs when the interparticle spacing is only a few diameters, is present. Thus a laser diffraction instrument may be sensitive to second order effects which are completely indeterminate from the orthogonal projection stored on the reticle.

10.2.3.4 Even if the line-of-sight coordinate information was accessible, scattering by noncircular particle artifacts (or by nonspherical particles, or by interacting spherical particles) is a complex, nonlinear process that cannot be modeled in the general case. The laser diffraction instrument must respond to a scattering signature of overlapped particles that, in general, will not correspond to the scattering pattern of a circular particle artifact of any one (equivalent) size, or even to a linear combination of scattering signatures from several circular disks. While image analysis can unambiguously assign a size to a noncircular particle artifact, the near-forward scattering pattern will not, in general, be consistent with the scattering pattern of a circular disk of that size. This is not a fault of either image analysis or laser diffraction systems; they are responding to different properties of the projection of multiple particles.

10.2.4 Reflections off the photomask reticle reaching the photodetector of the instrument.

10.2.5 *Speckle*—Spatial fluctuations in the measured near-forward scattering pattern caused by the coherence of the laser light interacting with the stationary particle artifacts on the reticle.

10.3 *Accepted (Image-Analysis) Reference Value Bias*—Difference between the accepted reference value and the true value, that is, a bias resulting from an inability to accurately characterize the photomask reticle or the size distribution simulated by the photomask reticle, or both. The following factors contribute to uncertainty in the reference values:

10.3.1 Bias and precision errors in an image analysis procedure used to determine the size of individual particle artifacts, and the resulting uncertainty in calculating reference values using these measurements,

10.3.2 Bias errors introduced by estimating the sizes of any particle artifacts which were not measured directly,

10.3.3 Errors associated with analyzing and accounting for overlapped particle artifacts. A two dimensional projection of a three dimensional particle arrangement necessarily will lose some information (that is, the information is inaccessible to image analysis) if more than one particle falls on an orthogonal projection line. There is some associated uncertainty in the inverse problem of estimating the properties of the three dimensional population from the two dimensional composite projection. This resulting bias is an artifact of using photomask reticles to simulate a particle field, and is not a shortcoming of laser diffraction instruments, and

10.3.4 Bias between the size of the image of a particle artifact and the light-scattering-equivalent size. This could be due to the fact that the particle artifacts are not infinitesimally thin but are of finite thickness and therefore have an associated uncertainty in defining the edge. For example, assume that a particle artifact (chrome disc) actually had the shape of a frustum of a right circular cone. If a particular image system focussed on the top of the frustum for sizing, but the light scattering process was determined primarily by the projected area consistent with the larger base of the frustum, then a bias would exist between the size determined from image analysis and a size representative of the light scattering signature. In this hypothetical case then, a reference value obtained from a perfect image analysis and one obtained using a perfect laser diffraction instrument would not agree. Anti-reflection coatings on a photomask reticle could also generate a bias between the image-analysis and light-scattering-equivalent sizes.

10.4 *Accepted (Image-Analysis) Reference Values*—The reference values for the samples used in the ILS are listed in Table 4. Details on the method used to determine these image-analysis reference values are presented in the research report.[12] The limits are based on an uncertainty analysis of the error contributions presented in 10.3 and include both bias and precision error (95 % confidence interval) components.

10.5 *Bias*—The bias of the test method is zero (95 % confidence) as indicated by Table 4.

10.5.1 *Discussion*—Based on an analysis of the between-lab reproducibility established in the ILS combined with the uncertainties in determining the image-analysis reference values presented in 10.2.2.5, it is not possible at present to rule out a bias of zero for the test method. Considering also the fact that a bias of zero is expected from physical/scientific reasoning, the value of zero is assigned in 10.5.

11. Interpretation of Results

11.1 Use procedures outlined in Practice E799.

TABLE 4 Bias of the Test Method (All Focal Lengths Grouped)

Test Result	Reticle Serial Number	ILS Results		Reference Value		Bias	
		Mean Value (µm)	Reproducibility Limit *R* (µm)	Image-Anal. Value (µm)	Uncertainty Limits[A] (µm)	Measured Bias (µm)	Statistically Significant?
$D_{V0.1}$	246	25.09	±5.18	27.0	±2.3	−1.9	No
$D_{V0.1}$	247	25.04	±5.37	27.0	±2.4	−2.0	No
$D_{V0.5}$	246	46.05	±4.26	49.2	±2.3	−3.1	No
$D_{V0.5}$	247	46.04	±4.57	48.9	±2.7	−2.8	No
$D_{V0.9}$	246	77.47	±26.50	72.5	±5.1	5.0	No
$D_{V0.9}$	247	77.33	±29.18	72.6	±6.3	4.7	No

[A] The uncertainty limits for the image-analysis reference values represent 95 % confidence intervals based on estimates of precision and bias error in assigning the values. The statistical significance indication also is for 95 % confidence.

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