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

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

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

1. Scope

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

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