



Standard Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering¹

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1. Scope

1.1 This test method covers the determination of particle size distribution of alumina or quartz using laser light scattering instrumentation in the range from 0.1 to 500 μm .

1.2 The procedure described in this test method may be applied to other nonplastic ceramic powders. It is at the discretion of the user to determine the method's applicability.

1.3 This test method applies to analysis using aqueous dispersions.

1.4 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 Quartz has been classified by IARC as a Group I carcinogen. For specific hazard information in handling this material, see the supplier's Material Safety Data Sheet.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *background*,—extraneous scattering of light by elements other than the particles to be measured. This includes scattering by contamination in the measurement zone.

2.1.2 *Fraunhofer Diffraction*,—the optical theory that describes the low-angle scattering of light by particles that are large compared to the wavelength of the incident light.

2.1.3 *Mie Scattering*,—the complex electromagnetic theory that describes the scattering of light by spherical particles. It is usually applied to particles with diameters that are close to the

wavelength of the incident light. The real and the imaginary indices of light diffraction are needed.²

2.1.4 *multiple scattering*,—the rescattering of light by a particle in the path of light scattered by another particle. This may occur in heavy concentrations of a particle dispersion.

3. Summary of Test Method

3.1 A sample dispersed in an aqueous medium is circulated through the path of a light beam. As the particles pass through the light beam, the particles scatter light at angles inversely proportional to their size and with an intensity directly proportional to their size. Detectors collect the scattered light which is converted to electrical signals and analyzed in a microprocessor. The signal is converted to size distribution using Fraunhofer Diffraction or Mie Scattering, or a combination of both. The scattering information is then processed, assuming the particles to be spherical, using algorithms or models proprietary to the particular instrument manufacturer. Calculated particle size distributions are presented as equivalent spherical diameters.

4. Significance and Use

4.1 It is important to recognize that the results obtained by this method or any other method for particle size distribution utilizing different physical principles may disagree. The results are strongly influenced by the physical principles employed by each method of particle size analysis. The results of any particle sizing method should be used only in a relative sense, and should not be regarded as absolute when comparing results obtained by other methods.

4.2 Light scattering theory that is used for determination of particle size has been available for many years. Several manufacturers of testing equipment have units based on these principles. Although each type of testing equipment utilizes the same basic principles for light scattering as a function of particle size, different assumptions pertinent to applications of

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² Muly, E. C., Frock, H. W., "Industrial Particle Size Measurement Using Light Scattering," *Optical Engineering*, 19[6], pp. 861–69 (1990).

the theory and different models for converting light measurements to particle size may lead to different results for each instrument. Therefore, the use of this test method cannot guarantee directly comparable results from the various manufacturers' instruments.

4.3 Manufacturers and purchasers of alumina and quartz will find the method useful to determine particle size distributions for materials specifications, manufacturing control, and research and development.

5. Interferences

5.1 Air bubbles entrained in the circulating fluid will scatter light and then be reported as particles. Circulating fluids do not require degassing, but should be bubble-free upon visual inspection.

5.2 Reagglomeration or settling of particles during analyses may cause erroneous results. Stable dispersions shall be maintained throughout the analyses. To determine if stability is present, make multiple runs on the same sample and observe if the distribution stays the same throughout the analysis. If the distribution gets coarser, then agglomeration is occurring. If the distribution gets finer, there exists the possibility of material settling. Dispersion properties may be altered by changing dispersants, use of ultrasonic energy prior to or during analyses, and change of pumping speed during analyses.

5.3 Insufficient sample loading may cause electrical noise interference and poor data repeatability. Excessive sample loading may cause excessive light attenuation and multiple scattering, thereby resulting in erroneous particle size distributions. The size distribution will have a tendency to be finer than actually exists.

6. Apparatus

6.1 *Particle Size Analyzer*, based on Fraunhofer Diffraction or Mie Scattering or a combination of both light scattering analysis techniques. Care must be taken to ensure that the analyzer system or subsystem is optimum for the size range being tested.

6.2 *Liquid Handling System*.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade of chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the precision of the determination.

7.2 *Dispersion Media*—Dissolve 1.5 g of sodium metaphosphate in 1 liter of distilled water and use this solution at an appropriate level so that the particles remain suspended in the aqueous system without creating bubbles. Other dispersants may be used for this purpose as well, such as Sodium Pyrophosphate, Tween 80, Triton X100, Photoflow, or others. The optimum dispersant for the analysis is dependent on the

material being analyzed and the amount of mixing and ultrasound available for each particular particle size analyzer system.

8. Calibration and Standardization

8.1 Performance of the instrument is defined by the spacing and position of the optical components. Refer to the manufacturer's instruction manual.

8.2 Diagnostic materials should be available from the instrument manufacturer to ensure consistent instrument functioning.

8.3 Since no absolute standards are available for particle size analysis, it is recommended that one should develop a secondary reference material to assist in evaluating and optimizing instrument performance.

9. Procedure

9.1 Allow the instrument to warm up for the time recommended by the instrument manufacturer.

9.2 If necessary, select applicable instrument range as indicated by the instrument manufacturer's instructions and establish correct optical alignment according to the instructions.

9.3 If required and available, use the index of refraction capability of the instrument. Many of the common compounds have their index of refraction listed in the Handbook of Physical Chemistry. Many compounds can also be found listed in the instrument manufacturer's instruction manual. The index of refraction used should be relative to the aqueous media, which has a refractive index of 1.33. When entering the index of refraction for the material being analyzed therefore, it is necessary to divide the index of refraction of the compound being analyzed by the index of refraction of water.

9.4 Measure the background in the mode in which the analysis will be performed. The dispersion media should be added to the sampling chamber before the background measurement is performed. Be sure that the carrier fluid is flowing through the light path and the sample cell while measuring the background, and make sure that no bubbles are present. Background values shall not exceed the manufacturer's specifications. If the background values exceed the manufacturer's recommendations, perform the necessary procedures as specified by the manufacturer to bring the background values within acceptable limits.

9.5 Before adding the sample, be sure to use the appropriate amount of the dispersion media to the sampling chamber. Then add the test sample. Obtain a test sample using appropriate sampling techniques. Sample-splitting equipment such as chute riflers and rotary riflers are available commercially to assist in these tasks. Refer to the instrument manufacturer's recommendation to insure that the amount of the test sample is acceptable to obtain optimum light scattering conditions. A range of sample size is acceptable depending upon the median particle size and particle density.

9.6 Select the appropriate run time for the sample. This procedure is very specific to the application and is generally gauged by the run-to-run repeatability.

9.7 Select the desired data output parameters according to the requirements set forth by the instrument manufacturer.

9.8 Determine proper dispersion conditions for the test sample. An example is described in Test Method C690 section 6.4.

NOTE 1—Some instruments have built-in ultrasonic baths to aid in dispersion. Others do not, and as a result, dispersions will have to be made externally using ultrasonic baths or probes. Also, food processors such as blenders may be used.

9.9 Perform the analysis according to the manufacturer's instruction.

9.10 Upon completing the analysis, drain and rinse system in preparation for the next analysis. Drain and rinse as many times as necessary to obtain the background values as specified by the manufacturer.

10. Precision and Bias

10.1 *Precision*—Repeatability study varied from 0.18 % above 7 μm to 0.01 % at 1 μm . Reproducibility study varied from 0.5 % above 7 μm to 0.1 % below 1 μm .

10.2 *Bias*—As there are no generally accepted absolute standards, bias cannot be determined.

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