PARTICLE SIZE ANALYSIS

micromeritics[®]



PARTICLE SIZE

ccurately determining particle size has become essential in many industries, as it is a fundamental physical characteristic that must be selected, monitored, and controlled from the raw material source to the finished product. There is an optimum particle size, or at least a smallest and largest acceptable size, for most items involving particles. The key to accurate particle size determination is selecting the most appropriate sizing instrument for a particular application. This is not a simple undertaking.

If all particles were spheres, their size would be defined explicitly by their diameter or radius; if cubical, the length along one edge would be characteristic; and, if of other regular shape, another equally appropriate dimension could be chosen. Unfortunately the great majority of particles are quite irregular and an arbitrary definition of size is the only simple solution. Moreover, typical collections of particles include many different sizes and shapes. Thus, a definition is required that accommodates this diversity, and the 'equivalent spherical diameter (or radius)' satisfies this requirement.

Equivalence of size means that a specific, experimentally measured attribute of the particle is the same as that of a sphere of a certain size. In other words, when the particle under test and a sphere of a specific size are exposed to the same conditions, the identical reaction occurs. Examples of reactions that may occur are the light scattering characteristics (scattering equivalency), the attainment of a certain terminal settling velocity (settling equivalency), or the displacement of fluid (volume equivalency).

Therefore, obtaining particle size information about a material may be accomplished by a number of techniques, each of which test for a specific equivalency. Micromeritics offers instruments that use three different techniques. This allows one to select the best technique for a material and application rather than trying to adapt one method to all situations. The sizing techniques used by Micromeritics are light scattering, sedimentation, and electric sensing zone. As is always the design objective with Micromeritics instruments, each instrument provides the user with high data quality (resolution and accuracy) and with a dependable measuring tool (reliability, repeatability, reproducibility).

ANALYTICAL RANGES AND APPLICABILITY

SIZE UNITS	MEASUREMENT RANGE			
m	10-7	10-6	10-5	10-4
mm $(10^{-3} m)$	10-4	10-3	10-2	10-1
mm (10 ⁻⁶ m)	10-1	1	10	10 ²
nm (10-9 m)	10 ²	10 ³	104	10 ⁵
Å (10 ⁻¹⁰ m)	10 ³	104	105	106
DIGISIZER				
SEDIGRAPH				
ELZONE				

INSTRUMENT	SAMPLE REQUIREMENTS	SAMPLE PRESENTATION	
DIGISIZER	Isotropic solid; preferably of known refractive index; refractive index different from that of suspension liquid	Homogeneously dispersed in a liquid preferably of known refractive index	
SEDIGRAPH	Solid; capable of absorbing soft X-ray (inorganic), density greater than that of suspension medium	Homogeneously dispersed in liquid	
ELZONE	Solid, or liquid immiscible in suspension liquid; organic or inorganic	Homogeneously dispersed in electrolyte liquid	

Why does Micromeritics have more than one particle sizing method? -- because no single measurement technique is ideal for all materials and applications. The most appropriate selection of instrumentation depends on many factors including the physical properties of the sample materials, their size ranges, the analytical throughput requirements, resolution and accuracy requirements, and the environment in which the instrument is to be operated. Particle size in itself is seldom the reason for the measurement; rather, it is the effect particle size has on something else—either a process or a material. This may influence the selection of the instrument technique selected. For example, if measuring particle size to better understand the stability of a suspension or the deposition process in a marine environment, the sedimentation technique likely would be more appropriate.

By offering more than one method of particle measurement, it is more probable that Micromeritics can provide the optimum particle sizing solution. Unlike a 'one technique' supplier, Micromeritics has no motivation to try to fit one technique to all particle-sizing applications.



LIGHT SCATTERING

particle illuminated with monochromatic, collimated light scatters the light at specific angles relative to the incident beam and at specific intensities at these angles as described by Mie and Fraunhofer theory. The intensity-angle relationship is a function of particle size, the wavelength of incident light, and the relative refractive index of the suspension fluid and particle. If multiple particles of various sizes are illuminated simultaneously, the resulting pattern is the summation of all the contributions of intensity by each particle at each angle.

There are various embodiments of Mie and Fraunhofer scattering theories in particle sizing instrumentation. These are generally referred to as 'laser particle size analyzers,' but are more appropriately described as 'laser diffraction' or 'low angle light scattering' (LALS) techniques. These instruments generally are applied either to the sizing of liquid or solid particles suspended in a gaseous medium (aerosols), or to solids suspended in liquid. Other applications are also found in laboratories and in production environments such as in sizing freely falling (cascading) particles or particles forcefully carried by fluid streams. By far, the widest application of LALS is to solid particles suspended in liquid media.

Light scattered from the assemblage of particles under test is detected at various angles and the intensity at each of these angles recorded. This set of ordered pairs (angle, intensity) describes the experimentally collected scattering function. Extracting information about the quantity of particles in each size class from this set of data requires that the composite information be deconvoluted into individual scattering patterns from each size class according to Mie or Fraunhofer theory for spherical particles. Deconvolution is based on fitting a summation of 'reference' scattering patterns from spherical particles to the experimentally collected light scattering signature of the particle assemblage. The particle sizes and quantities reported, therefore, represent the collection of spherical particles whose composite (summed) scattering pattern best reproduce the experimental data.

Micromeritics' Saturn DigiSizer 5200 Particle Size Analyzer uses light scattering to determine particle size. The hallmarks of this instrument are the exceptionally high resolution of the scattering angle and the wide dynamic range of light intensity accommodation. These capabilities are made possible by the use of a 1.3 million element charge-coupled device (CCD). Fine-tuning of the optical alignment is accomplished in software by dynamically remapping the array around the central beam (optical axis). Reproducible, pinpoint alignment assures that the instrument will provide the same data for the same sample from analysis to analysis and from laboratory to laboratory. High-resolution capabilities mean that a slight change in size or quantity distribution from sample to sample will be distinguishable.

SATURN DIGISIZER CAPABILITIES

- Range 0.1 to 1000 micrometers
- Resolution: baseline separation (Monosized particles separated by 30% in size) (not applicable from 0.1 to $1\,\mu m)$
- Automatic sampling, diluting, and dispersion
- Optional autosampler



ELECTRIC SENSING ZONE

he Electric Sensing Zone method detects the volume of liquid displaced by a particle as individual particles of a sample suspended in an electrolyte are transported through a sensing zone. The sensing zone is a short capillary that separates two volumes of electrolyte liquid, each volume being maintained at different electrical polarities. When a particle is being swept through the sensing zone, it displaces some volume of the conducting liquid, thus decreasing the conductance of current through the capillary. The momentary interruption of current flow results in an electrical pulse that is detected by sensing circuitry. The amplitude of the pulse is proportional to the volume of electrolyte displaced and, thus, to the volume of the particle. The instrument sums the number of particles (pulses) occurring in each volume (amplitude) class. From these data, a frequency distribution of particle volume versus particle size is obtained. For particles of uniform density, volume is directly proportional to mass. Although volume is measured directly, size is reported as the equivalent spherical size and equals the size of a sphere that displaces the same volume of liquid, as does the particle under test.

Micromeritics' Elzone particle sizing and particle counting analyzers use the electric sensing zone technique.

ELZONE CAPABILITIES

- Range 0.4 to 1200 micrometers
- Immune to almost all physical properties of samples. Samples can be a mix of one/all:
 - Optical properties; including index of refraction, opaque or transparent
 - Different densities
 - Different shapes
 - Organic and/or inorganic
- Can measure true concentration of particles to give number of particles per ml, per gram, PPM, etc.
- Different liquids can be used without knowing viscosity, only that they are conductive
- Particles can be dispersed in a liquid that is different from the electrolyte
- Traceable calibration applies to all materials, not just the reference material



SEDIMENTATION

edimentation is perhaps nature's primary method of size separation as evidenced by the deposition of waterborne and airborne materials. It has been used by man to separate particles by size longer than any other method with the exception of sieving. Sizing by sedimentation is not difficult and the process is described rigorously by Stokes' equation. The difficulty arises in determining the quantity of particles in each size class. This problem was solved by the use of soft X-rays to detect mass. Micromeritics introduced 'X-ray sedimentation' as a measuring tool over 30 years ago. For many industrial applications and scientific studies, Micromeritics' SediGraph remains the de facto standard technique.

The SediGraph employs sedimentation from a homogeneous liquid-solid suspension to separate a sample by size. The absorption of X-rays directly detects the mass concentration in the spatially separated collection of particles. Measuring the rate at which particles of a certain density fall under the influence of gravity through a liquid of known density and viscosity provides all of the necessary parameters to apply Stokes' equation and determine the equivalent spherical sizes of the particles. In this case, the size reported is the size of a sphere that has the same settling rate as the test particle.

SEDIGRAPH 5100 CAPABILITIES

- Range 0.1 to 300 micrometers
- Complete particle accountability; all particles are accounted for, even ones outside range of instrument
- Fully automated operation
- Up to four instruments can be operated from one computer
- Optional autosampler

MICROMERITICS. SETTING THE PACE WORLDWIDE

Micromeritics has more than a half-century of experience in providing innovative products to the particle technology marketplace. Instrumentation developed by Micromeritics has been the recipient of awards for design excellence on numerous occasions. Our instruments have been chosen as the standard of performance by many multi-national corporations. As a result of this,

Micromeritics is pleased to provide products and product support to customers around the globe. The driving force of our Company is to develop and support high-quality, high-performance instrumentation of unmatched accuracy and utility, never losing sight of the primary importance of satisfying the needs of our customers.

Micromeritics maintains a high level of interest in the needs of the many industries it serves and aggressively responds to these needs. It is this response that firmly establishes Micromeritics as one of the world's leading suppliers of particle technology instruments.

Micromeritics has over 50 sales, service, and distribution offices throughout the world.



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