#micro Report



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Why Demand for the

SediGraph Endures

Paul A. Webb

The SediGraph method for particle sizing has been used in laboratories worldwide since 1967. Although more 'modern' alternatives have been developed for determining particle size, the SediGraph method continues to satisfy best many applications. The reasons for



the continued viability of the SediGraph method is the subject of this abbreviated version of an article located on our web site at www.micromeritics.com.

THE SEDIGRAPH METHOD

The SediGraph has been objectively compared to various other particle sizing techniques and found to be a reliable and repeatable method. In a guide to particle size characterization (1) published by the National Institute of Standards and Technology (NIST), the SediGraph is described as being based on a robust technique, providing rapid analyses, being well-suited for industrial environments, relatively inexpensive, not requiring highly skilled operators, and having the capability to be used over a broad size range with minimal changes.

Anyone shopping for an instrument for particle sizing has a wide selection from which to choose. The wisest choice will be made with a thorough understanding of the model upon which the measurement technique is based. Since size is seldom measured directly, one needs to understand what characteristics of the particle the instrument is measuring and how this measurement relates to size (2). This article answers those fundamental questions for the SediGraph method, and helps to explain why the reliance on the SediGraph method has endured the test of time.

SIZE DETERMINATION

The SediGraph method is based on two well-established and well-understood physical phenomena—gravitational sedimentation and low energy X-ray absorption.

Stokes' law describes the gravitational sedimentation of a particle stating simply that the terminal settling velocity of a spherical particle in a fluid medium is a function of the diameter of the particle. The SediGraph actually measures the time required for a particle to fall (settle) a known distance, thus establishing its settling velocity from the well-known 'distance equals velocity times time' relationship. The velocity value and parameters associated with the liquid and solid are substituted into Stokes' law, which then is solved for particle size. Thus, the SediGraph can report measured settling velocities as well as particle sizes.

MASS FRACTION MEASUREMENT

A thin, horizontal beam of X-rays is projected through the sedimentation cell as the means to determine directly the change in mass concentration in the liquid medium as sedimentation proceeds. This is done by first measuring the intensity of a baseline or reference X-ray beam that has been projected through the liquid medium prior to the introduction of the sample, the analvsis sequence being illustrated in the accompanying Figure. As liquid circulation continues, solid sample is added to the liquid reservoir and mixed until a homogeneously dispersed suspension of solid sample and dispersion liquid is being

pumped through the cell. More X-ray energy is absorbed by the solid than the liquid, therefore the transmitted X-ray beam is attenuated compared to the reference beam. Since the mixture of the flowing suspension is homogeneous, attenuation is greater under this condition than it will be when the particles are allowed to settle out of suspension.

Flow of the mixture is stopped and the homogeneous dispersion begins to settle and separate over time, the transmitted X-ray intensity being constantly monitored during the sedimentation process. The vertical position of the X-ray beam is precisely known and at any time the size of particles that have had sufficient time to settle below the beam can be determined even if they began settling at the top of the cell. Of course, largest particles are first to settle below the measuring zone and, finally, all particles settle below this level leaving only clear liquid. During the process, attenuation of the X-ray beam diminishes from maximum to the value established during the baseline measurement. The intensity of the transmitted X-ray beam at any time is directly related to the mass concentration of the remaining suspended particles.

Initially, the position of the X-ray beam is near the bottom of the cell to allow the larger, fast-settling particles to be quantified before falling below the measurement zone. As the larger particles settle out, the position of the X-ray beam is stepped upward in order not to have to wait for the smaller, slower-settling particles to settle as great a distance. This

technique greatly decreases the analysis time, even when working with slow-settling particles.

Of course, the baseline and full scale measurements and the analysis processes as described above are automated.

APPLICATIONS

Particle size distribution can be determined by a number of techniques (2). However, certain techniques are clearly more applicable than others in certain tests. Take, for example, study of marine silts and sediments. Since the transport of solid components and the subsequent deposition are dependent upon sedimentation rate in water, the SediGraph technique is an ideal method since it, too, is based on sedimentation. Rather than determining particle size by some other technique, then calculating settling velocity from the equivalent spherical size, the SediGraph provides settling velocity information directly and the size of the particle need not even be considered.

Another benefit of the SediGraph method is realized most acutely by those who employ other sizing techniques in addition to that of the SediGraph. This has to do with understanding how reported data are affected by analyzing particles that do not fit the theoretical model upon which the instrument is based. Sedimentation is a comparatively simple mechanism. It is much easier to understand, for example, how non-spherical particles settle compared to spherical particles and, thus, understand what shifts in reported size interpretation are to be expected. This is anything but true when it comes to predicting what effects will be observed in the size data obtained from analyzing nonspherical particles with a laser light scattering system.

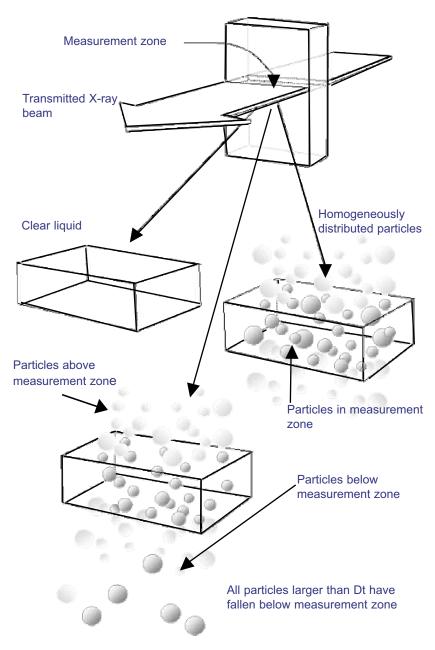
SUMMARY

Particle size is studied in many applications of science and technology. It is easy to understand why particle sizing equipment is available in such a variety of configurations since particle sizes can range from nanometers to millimeters and involve essentially any material, liquid or solid, suspended in a liquid or a gas. The SediGraph is only one of many choices and, because of its time-proven capabilities, continues to hold its place as the most relied-upon method of particle sizing in certain applications.

REFERENCES

1. Particle Size Characterization, Ajit Jillavenkatesa, Stanley J. Dapkunas, Lin-Sien H. Lum, NIST Recommended Practice Guide, Special Publication 960-1, January 2001

2. Interpretation of Particle Size Reported by Different Analytical Techniqus Webb, P.A.



Upper: Diagram of SediGraph sample cell penetrated by a horizontally collimated X-ray beam. The volume of the sample cell through which the X-ray beam passes defines the measurement zone. **Lower:** Measurement zone shown at three stages of an analysis. First, only clear suspension liquid circulates through cell. Next, sufficient sample is introduced to produce a suitable attenuation of the X-ray beam. Finally, circulation is stopped and the suspension is allowed to settle, thus separating the particles by size. Sedimentation continues until all particles have settled below the measurement zone.

For a detailed description of the SediGraph technique refer to ISO 13317-3:2001 Determination of particle size distribution by gravitational liquid sedimentation methods - Part 3: X-ray gravitational technique.