# Solving problems one particle at a time . . .













# Solving customers' problems one particle at a time...

Particle Sizing Systems is dedicated to providing innovative solutions to our customer's most difficult particle sizing problems.

Particle Sizing Systems' engineers are focused on inventing unique instruments that offer powerful capabilities in the laboratory and process environments. We have been a major force in the manufacture and patenting of automated single particle sizing instruments for both wet and dry applications. Currently, we are the only company to offer an automated, high resolution, single particle optical sizing system in the market.

Our SPOS instrument fits a unique position offering a complimentary technique for many other laser light scattering instruments on the market today. We provide information about the tails of distributions that other instruments lack the sensitivity to detect.

Our approach is simple; we first listen to a client's problems and then focus our more than 30 years of particle sizing experience to solve them.

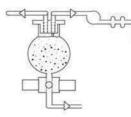
From research and development to quality control environments our particle size analyzers offer unique solutions to our customers' problems with colloidal stability, particle characterization, and by focusing on the de"tails" of their distributions.





The AccuSizer 780 uses the method of single-particle optical sizing (SPOS) to quickly count and size a large number of particles, one at a time, thus constructing the true particle size distribution (PSD). This approach is in sharp contrast to "ensemble" methods, such as laser diffraction and sedimentation, which must process information produced simultaneously by many particles. These alternative techniques require the use of complex mathematical algorithms to invert the data and yield only rough approximations of the PSD, with limited accuracy and very poor resolution. The AccuSizer determines PSDs with resolution and accuracy comparable to those produced by the classical resistive-pore, or electrozone, method. The 780 does not miss the "details", which are often critically important in determining the quality of many products. A wider size range, higher count rate, compatibility with any liquid or gas, and relative immunity to clogging make it an instrument in a class all its own.

The SPOS particle sizing method requires that concentrated suspensions be diluted, in order to eliminate particle coincidences in the sensing photozone. Excessive dilution results in too low a count rate and poor statistical accuracy of the PSD, while overly concentrated suspensions produce distortions in the distribution. The AccuSizer 780 uses Autodilution<sup>Pat</sup>, which quickly and automatically dilutes the starting sample to the optimum concentration, resulting in efficient and accurate analysis. Trial-and-error manual dilution is eliminated, paving the way for single-keystroke operation; a complete analysis and flush cycle can be completed in less than 3 minutes.





The 780's single particle sensitivity detects the important features in the size distribution which other techniques usually miss. Single particle optical sensing makes the measurement accurate, while Autodilution<sup>Pat</sup> and leading-edge electronics make it fast and easy to use. Trading off accuracy and resolution for speed and ease of use no longer have to be made with the AccuSizer 780.







# The AccuSizer 780 delivers reliable size and count data for even the largest particles

A few outliers can spell the difference between product success and failure; however traditional laser diffraction particle size analyzers cannot detect large particle outliers in a sea of smaller particles.

The AccuSizer 780 reveals particle size differences that previously went unnoticed.

The 780 is a stable, sensitive instrument that uses Single Particle Optical Sizing (SPOS) to count and size particles one at a time, eliminating missed particles while allowing:

- o Particle sensitivity of 10 PPT
- o Particle size accuracy of 2%
- o Particle count accuracy of 10%

Recently, an independent study proved that SPOS analyzers were 1,500 to 25,000 times more sensitive in detecting outliers than light scattering and accoustical sizing instruments.

The AccuSizer 780 can also resolve fine particle distributions that are directly related to the materials' properties. It is a proven and valuable tool that laboratories around the world are already using to reformulate their product lines from R&D to production.



# A modular solution to meet your most challenging applications

The AccuSizer 780 AD Autodiluter is the cornerstone of the modular family of instruments. The AccuSizer 780 AD incorporates a series of wide dynamic range sensors with a single stage exponential diluter to provide a flexible high resolution, accurate, sensitive particle sizer. It provides particle size information with unprecedented resolution and accuracy. It is especially suited to providing information on particle size distributions (PSDs) that are on scale but is not limited in the statistical information that it can provide on tails of mostly submicron distributions. This patented module eliminates the need for manual dilution of concentrated samples. Autodilution makes particle size analysis quick and easy, with no training required. Results are highly reproducible.

### AccuSizer 780/APS Automatic Particle Sizer

The APS has been called the bolder detector by many of our customers because of its ability to detect low levels of aggregates several standard deviations away from the mean of the distribution. Often these aggregates are the difference between a good and bad sample. One independent study showed the APS was 1,500 to 25,000 times more sensitive in seeing outliers in the CMP slurry than commonly used laser light scattering and acoustical sizing instruments. The same sensitivity will apply to other applications such as inks, pigments, drug emulsions where a few large particles spell the difference between success and disaster.

# AccuSizer FX Focused Extinction

The AccuSizer FX is a new breed of particle size monitoring system designed to go from the R&D lab to the process line with minimum system reconfiguration. The FX utilizes a patented focused beam technology that allows the sensor to focus on a specific area of the flow channel to count and size individual particles at concentration levels that are hundreds of times higher than traditional sensors. Although this type of approach has been tried by other techniques it has been plagued by low resolution and accuracy. The new FX sensor technology utilizes a patented electro-optical configuration that maintains the same levels of resolution and accuracy found in traditional SPOS sensors.



## The AccuSizer 780/LVS Large Volume Sizer

The AccuSizer 780/LVS Large Volume Sizer is specifically designed to analyze large volumes of liquid with particles as large as 2000 microns. Often it is imperative to pass large amounts of diluted dispersion through an SPOS sensor. The key to the AccuSizer LVS system is its large volume dilution chamber that combines the ease of use of autodilution with the speed of a quick flushing fluidics system.

The AccuSizer 780/LVS allows the user to introduce a spoon full of material into the large volume dilution chamber and immediately start taking data. Ease of use of a fully automated dilution system is combined with SPOS to provide fast, high resolution, high sensitivity particle size distributions.



# AccuSizer 780/SIS Syringe Injection Sampler

When volumetric sampling is paramount, the SIS sampler fits the bill. With syringes ranging from 0.5 to 25 ml and a precision stepper motor with over 25000 steps per pull the SIS sampler is a perfect addition to the family of modular instruments. It is specifically suited to applications in the pharmaceutical industry such as USP <788> small and large volume injectables. Now available for use with the AccuSizer 780/SIS is 21 CFR Part 11 compliant software, Calibration and Validation SOPs.



### AccuSizer 780/MPS Mobile Particle Sizer

The AccuSizer 780/MPS was designed for sequential multi-point process monitoring at line where a multiplexer system or multiple system monitors are not viable options. Now one can have the benefits of at-line process monitoring without the expense of a fully automated on-line system. Just another way PSS allows users to bring laboratory measurements to the process QC area.

### AccuSizer 780/OL Online

Our patented Autodilution technology has allowed us to take our laboratory instrumentation known for high precision, accuracy, resolution and reproducibility, and carry it to the

process line. Simple customizable computer controlled fluid-sampling devices are used to grab a precise aliquot of concentrated in-line dispersion and automatically inject it into the Autodilution system. Then, just as in our laboratory systems, the sample is diluted to the optimal concentration for an SPOS measurement, without regard to its initial concentration or particle size distribution (PSD). The resulting PSDs, with unique single particle resolution are identical to those that historically could only be obtained in the research laboratory. No longer must correlation algorithms and complex statistical tools be developed to compare laboratory findings with online results.

The Autodilution module is an integral part of both of our laboratory instruments-- the AccuSizer 780 Single Particle Optical Sizer and the Nicomp 380 Submicron Particle Sizer. These laboratory instruments have been modified to meet the rigors imposed by online process monitoring. They have been redesigned utilizing ruggedized com-ponents and are housed in different classes of NEMA enclosures, depending on the application. These vary from dust and waterproof enclosures to explosion-proof modules that can handle even the most aggressive environments, including high pressures and sub-zero temperatures.

Multi-port systems are available with specialized software to monitor multiple points in an online process. These reduce costs while maximizing coverage to ensure that quality control requirements are met. Users have a choice of using either an embedded mode Windows based controller or a Programmed Logic Controller (PLC) which are often found in process control systems. In either case the resulting data can be displayed on screen or passed to a Laboratory Information Management System (LIMS) as an ASCII file. It provides for limitless post-capture data analysis that can easily be customized by the user with common programs found on all modern computer systems.

The AccuSizer 780/OL system is able to detect minute amounts of oversized particles in the tail of the process dispersion. It has produced results where the difference between a good and bad sample is determined by a few hundredths percent in volume fraction above a particular critical particle diameter, several standard deviations above the main peak of the distribution. It is also able to detect very small percentages of "fines" in the presence of larger particles. These fines often play an important role in determining the viscosity, flowability and other characteristics of both the process dispersion and the final product.





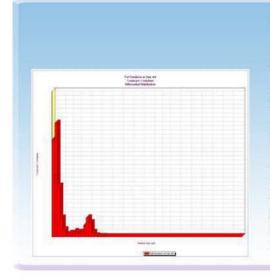








# Applications



#### Problem

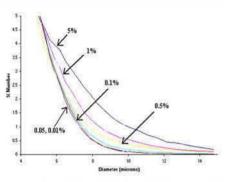
Many times an instrument will display the whole particle size distribution, but the user will be experiencing poor stability with their product. The problem may be caused by a few aggregates in the tail, but the instrument does not have the sensitivity to detect them.

#### Solution

This is a fat emulsion that was spiked with a low concentration of a 1 micron latex standard. As the result shows, the AccuSizer was able to detect the main peak of the distribution but more importantly it was able to find the aggregates that existed in the tail of the distribution. Count and size information for the entire distribution was also provided. It is these aggregates in the tail that can spell disaster.

#### Problem

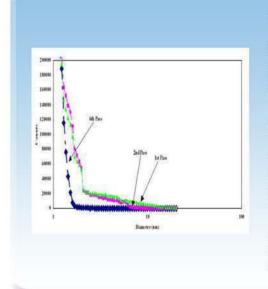
Kaolin Clays have many varied applications, because they change the properties of the materials they are combined with. For example, fruit can be coated with a fine layer of kaolin clay as a safeguard against pests. Most light scattering ensemble systems, however, do not have the sensitivity and resolution to see small differences in the large particle tail of kaolin materials; this can compromise the clay's integrity and cause disastrous product defects. As a result, some manufacturer's have had to implement time-consuming (and inconsistent) manual microscopy methods to identify and count large particles.



#### Solution

During Kaolin processing, several methods - including grinding, centrifugation, and filtration - may be employed to reduce particle size. The effectiveness of these processing methods can easily be determined by the AccuSizer 780, which has the sensitivity, resolution, and statistical accuracy to sample a large segment of the tail population and provide definitive product quality data.

Here, a kaolin clay product that failed due to the pressence of large particles was blended into a product that performed well. The 780 successfully tracked the addition of the "bad" material from 0.01% to 5% by weight. Other light scattering devices did not detect the large particles until the amounts exceeded 3%; even worse, the competitive systems did not pick up the presence of the large particle tail until the volume reached 5%.



#### Problem

In high pressure homogenization, which is used to make beverage emulsions, even a few oil globules can cause the emulsion to destabilize and separate before it reaches the marketplace. Furthermore, overhomogenizing the emulsion can cause it to destabilize even faster.

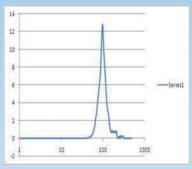
#### Solution

The AccuSizer APS with DLS module accurately monitors and quantifies the large particle tail at the exact point at which the emulsion exits the homogenizer. In doing so, it can count and size any large oil globules that have not been properly processed.

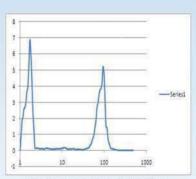
A major beverage emulsion manufacturer has successfully used the 780 to increase shelf life stability and save the time - and money - of product re-processing.



There are five major steps in converting Bauxite to Alumina. In each of the steps, from the crushing and grinding of the bauxite to the calcination of the particle size, the material plays a major role in the process and the profitability of the process. In the settling process, where gravity and filtration are used to remove impurities from the slurry, the presence of fines and the ability to monitor and quantify them plays a critical role. Classical ensemble light scattering techniques do not have the sensitivity and resolution to identify and quantify these fine particle distributions in the presence of a large particle peak that is several standard deviations higher than the area of interest. The problem is in physics of light scattering methods since particles scatter light as a multiple power of their diameter ranging from D<sup>6</sup> to D<sup>3</sup>. The larger particles in the distribution which are there in larger numbers scatter most of the light and obscure the signal from the smaller size peak. The industry has turned to instruments that derive their distributions from particle number based sizing principles rather than particle volume based sizing principles. The main instrument used by the Aluminum industry employ electro-zone sizing principles and microscopy methods to monitor the fines in these processes. The problem with both of these techniques is that they are labor intensive and require an experienced operator to perform the analysis and to interrupt the results.



Volume-weighted Distribution



Number-weighted Distribution

#### Solution

The AccuSizer 780 instruments have revolutionized the measurement for this application. These instruments have the resolution and sensitivity to monitor the fines of these processes in real time whether in a concentrated slurry form or during the alumina cake slurry separation process. Along with autosampler capabilities the throughput in the QC labs has dramatically increased. They can even be used to monitor the low levels of fine impurities in the calcination process. From the AccuSizer Online to the Automatic Particle Sizer (APS) these instruments have given the process control engineers in Alumina companies the tools to reduce their overall costs by multiple factors of the total instrument cost in a matter of months.

To the left displays a volume-weighted distribution which shows no contribution to the overall distribution by the fines that are present. Yet, the number-weighted distribution shows a bimodal where contribution to the overall distribution is almost 60:40. While it is easy to convert the number-weighted distribution to a volume-weighted answer by taking the number of particles in each size bin and multiplying them by their respective volume it is impossible to convert a volume-weighted answer that does not contain any information on the fines into a number-weighted distribution.

Many in the Alumina industry have boasted that the AccuSizer provides the resolution and sensitivity of electrozone sensing with the ease of use and flexibility of laser based on- and at- line ensemble methods. The best of both worlds combined into one flexible user configurable instrument is available with the AccuSizer 780 family of instruments.

# Single particle sizing proven to identify critical differences



The following photographs show two intravenous fat emulsions: one that

passes USP <729> and one that fails. The emulsion that passes has approximately 5 large particles in the micrograph, and can safely be infused into a patient. The emulsion that fails has 9 large particles, and could be lethal if infused.

All of these particles were found in the "tail" of the distribution. As a result of the AccuSizer's ability to count and size particles one at a time, information about the number and size of all of the particles was provided.

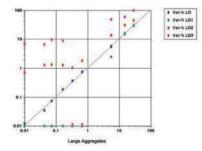
#### Measurement of oversize globules above 5-microns ("PFAT-5")



These particles make up less than 0.01%of the total number of particles that are 5 microns or larger in the emulsion. PFAT-5 < 0.01%This fat emulsion PASSES.



PFAT < 0.025% This fat emulsion PASSES.





PFAT-5 < 0.10% This fat emulsion FAILS.

SPOS properly tracks the destabilization of a Fat IV emulsion.



Over time it can be seen that the fat emulsion becomes very unstable and the number of particles in the tail of the distribution exceeds 20%. PFAT-5 > 0.20% This fat emulsion FAILS.

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