ULTRASIZER SV –
PARTICLE SIZING OF CONCENTRATES WITHOUT DILUTION

ULTRASIZER

MALVERN
INSTRUMENTS

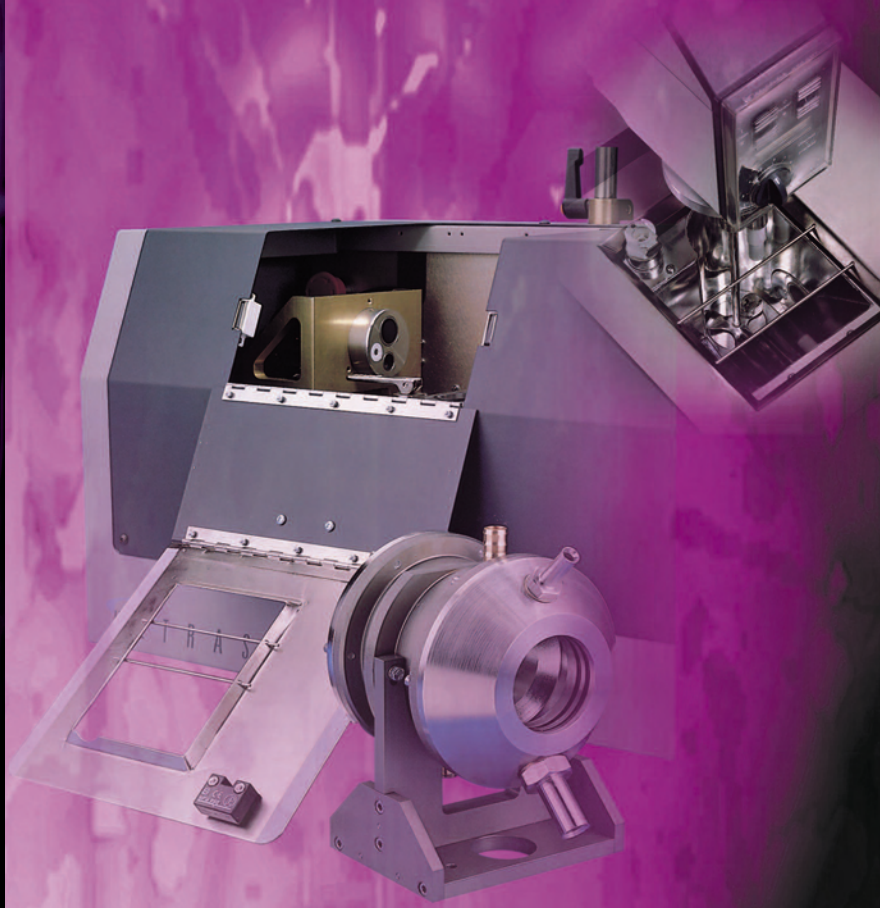
THEORY

Particle size analysis of concentrated dispersions without dilution

Particle size measurements in concentrated dispersions have long posed a problem in many industries. Sample dilution and sample preparation can alter particle size results and these issues have hindered the complete understanding of some industrial processes.

Malvern Instruments' Ultrasizer SV is a patented acoustic spectrometer that can measure sound attenuation and sound velocity as a function of frequency giving particle size distributions in emulsions and suspensions.

Ultrasizer SV offers new insights into processes that may previously have been impossible to study in detail. Understanding brings new levels of control, for improved efficiency in all areas of product and process development and management.



Concentrated systems – the measurement challenge

The size distribution of fine particle dispersions is an important and critical parameter in a multitude of industries and applications. Size measurement and control may be required through many or all stages of a product's life – from the R&D phase, through process control to quality tests on the final product.

Particle size analysis of concentrated systems using light scattering requires severe dilution – typically down to levels of less than 0.1 volume percent. Not only do many of these finely balanced systems respond poorly to dilution, there is also the concern of how representative the sample is.

Particle technologists, chemists and engineers would prefer to measure these systems at process concentrations. Working this way assures the analysis of naturally occurring aggregation, agglomeration and flocculation processes without changing the system. Furthermore, operator and sampling errors associated with complex sample handling protocols are eliminated.

Acoustic based particle size measurements offers the following advantages:

- Measures opaque, high concentration samples
- Measures particle size in suspensions in concentration from 0.5 to 50 volume percent, and up to 80 volume percent for emulsions

For size ranges from 0.01µm to 1000µm in emulsions and suspensions.

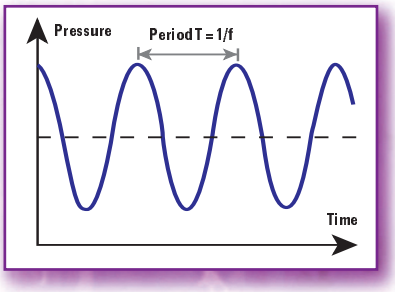


figure 1. Pressure change experienced by a particle

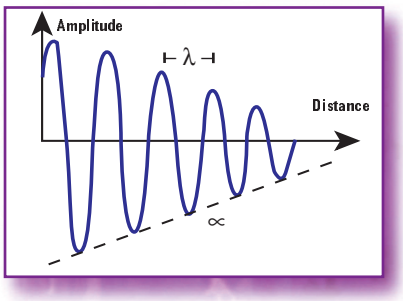


figure 2. Sound wave attenuation

- Measures particles over a wide size range from 0.01µm to 1000µm
- Attenuation and velocity data provide early indication of a changing process or an out of specification product
- No complicated sample preparation schemes
- Non destructive technique

Patented technology – a fundamentally sound approach

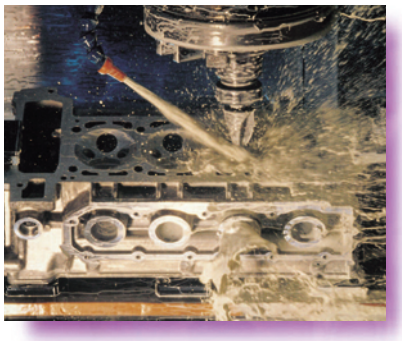
All Malvern's instruments are founded on a first principles physics approach to particle size measurement – and the Ultrasizer SV is no exception – where a fundamental theory links the physical property being measured with particle size. High performance, reliability and a clear understanding of theory are inherent in the instrument design.

Acoustic spectroscopy works by transmitting sound waves through the sample. The attenuation of the sound is measured over a wide range of frequencies. The particle size is then calculated by using software that compares the measured attenuation spectra to the behaviour predicted by the acoustic model.

Sound waves are compressional or longitudinal waves. They travel through a sample as a series of mechanical oscillations in both the macroscopic and microscopic structure. The material through which the sound propagates therefore experiences a constantly changing pressure field. *(figure 1)*.

As the sound wave propagates through a pure liquid, the amplitude of the mechanical oscillation decreases i.e. the wave is attenuated. By measuring how rapidly the amplitude decreases with distance, the attenuation coefficient (∝) can be calculated. *(figure 2)*.

If particles are placed in the liquid phase, the attenuation measured for the sample increases. This increase is normally larger than would be expected based on the intrinsic attenuation of each of the phases present. The visco-inertial, thermo-elastic, diffraction and scattering loss mechanisms cause this excess attenuation.



Metal cutting fluid emulsion in use

THEORY

The interaction of sound with particulates has been well studied. Lord Rayleigh [1] was the first to propose a theory to describe the propagation of sound through materials. This work was extended to allow the changes in sound velocity and attenuation to be predicted for water borne particles in air. Currently, one of the most complete theories is that based on the work of Epstein and Carhart [2] and Allegra and Hawley [3]. This scattering model accurately predicts the magnitude of the attenuation for both suspensions and emulsions. In certain cases compromises can be made in the modelling to allow simpler but less versatile theories to be developed.

The scattering theory implemented in the Ultrasizer software accurately predicts the magnitude of each of the scattering and absorption mechanisms. The movement of the particles relative to the continuous phase causes visco-inertial losses as the sound wave propagates through the sample. Drag between the liquid and the particle causes sound energy to be lost as heat. *(figure 3)*.

Thermo-elastic scattering occurs due to the different compressibilities of the continuous and particle phases. The particles expand and contract relative to the surrounding liquid under the influence of the pressure changes that occur during sound wave propagation. Due to temperature-pressure coupling, heat is lost from the particle during this process. *(figure 4)*.

Scattering and diffraction losses are similar to the processes occurring in light scattering experiments. The presence of the particle in the acoustic field causes the sound wave to be deflected away from the forward direction. *(figure 5)*.

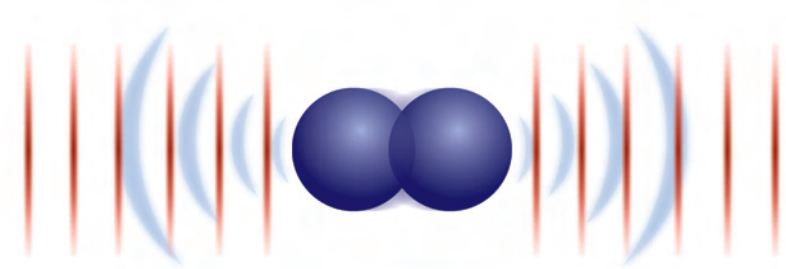


Figure 3. Viscous inertial losses

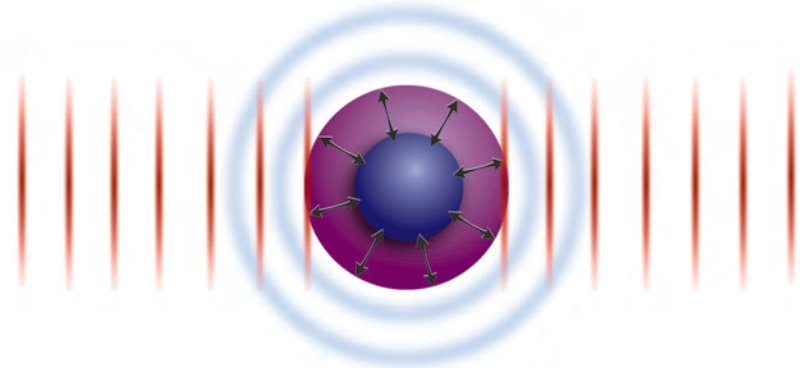


Figure 4. Thermal pulsation losses

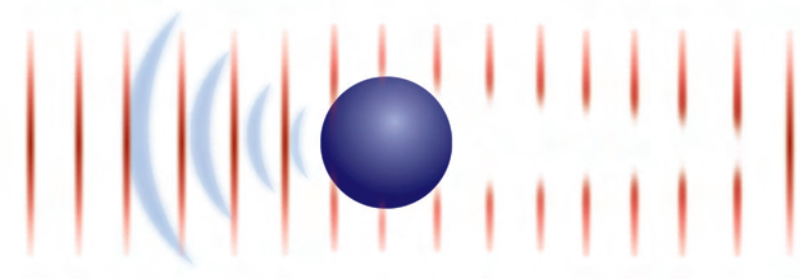


Figure 5. Scattering losses

ULTRASIZER SV

An advanced system ...

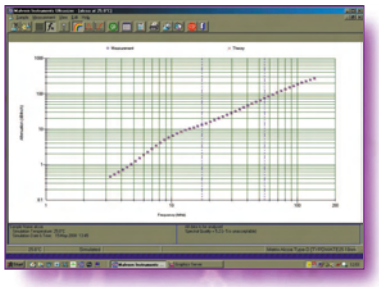
The Ultrasizer SV is an advanced measuring system that uses state-of-the art technology for all stages of the measurement process with novel transducer design, digital signal processing and a fully software controlled operating and data analysis environment.

The Ultrasizer SV has many novel features including a range of interchangeable measurement cells that can be swapped out in seconds. Fixed stirred batch cells are available with volumes from 450mls. Flow cells are available that allow the instrument to be interfaced into particle streams and reactors.

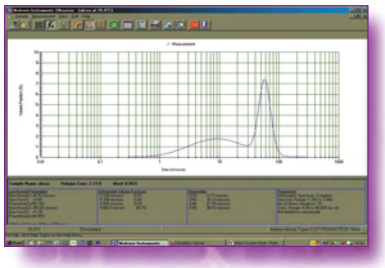
... that anyone can use

Ultrasizer SV is tough and rugged, and remarkably easy to use. The instrument can be placed in the laboratory or in a process environment.

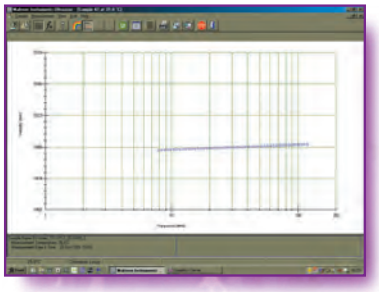
An expert system in the software automatically determines the optimum measurement strategy. The software enables you to run the same sample in the same way every time, ensuring the generation of data you can trust, with full traceability and comparability, whoever operates the system.



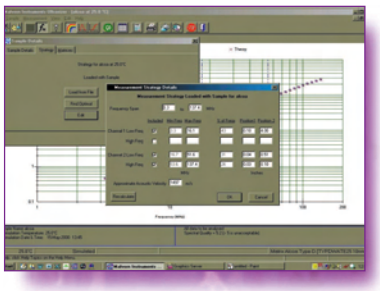
Attenuation Spectrum



Particle Size Distribution



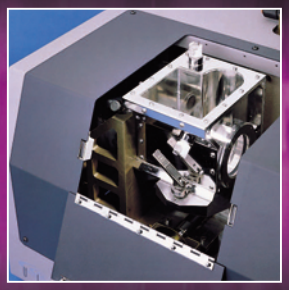
Velocity Spectrum



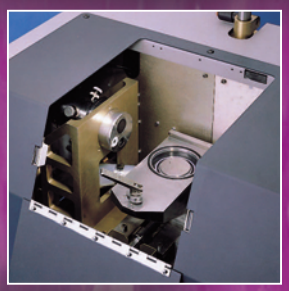
Measurement Strategy

Interchangeable batch and flow measurement cells

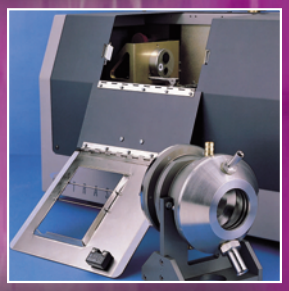
Stirrer and batch measurement cell



Cover open with quick release lever

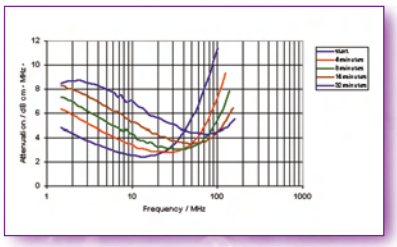


Cell area

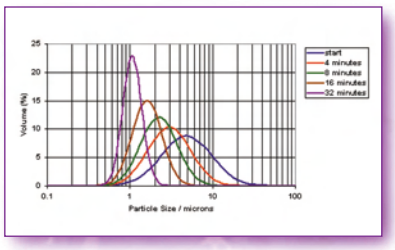


Flow measurement cell

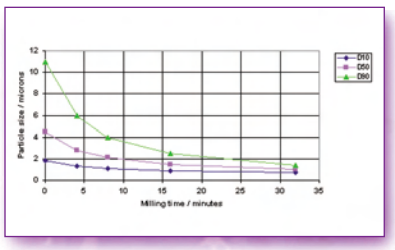
Measurement in practice



Acoustic attenuation spectra showing changes with increase in milling time.



The above acoustic attenuation spectra when converted into size data showing clearly the change in particle size distribution with milling time.



Particle size analysis trend plot – for ease of process control.

Reliable Monitoring

Of particle size reduction or growth in suspensions and emulsions. The Ultrasizer SV lets you track changes through the raw attenuation spectra presented in a variety of ways, or through particle size analysis and trend plots.

Particle reduction

Examine the effects of homogenization time and shear rate - for example food emulsions and lubricant emulsions, and the impact of time of milling, for process optimization and potential energy savings – for example, milling inorganic and organic pigments, ceramics and minerals.

Particle growth

Determine which parameters affect the rate of crystallization, particle nucleation and growth kinetics, as well as the effects and optimization of additives.

Effective formulation

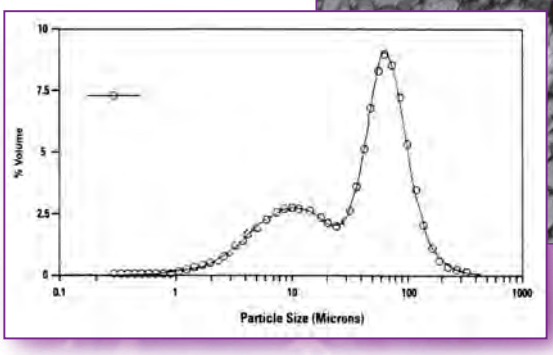
Measure the effects of surfactants and other adjuvantants, optimize process conditions and select raw materials to ensure cost-effective formulation.

Stability determination

Use Ultrasizer SV as part of a range of tests to optimize and assess the stability of disperse systems, suspensions and emulsions, and to study dissolution rates or particle aggregation.

Towards In-line process control

A driving force in today's particle size analysis market is the increasing demand for in-process measurement, to provide real time data and enable direct feedback into the production process. Malvern Instruments is committed to in-process particle size measurement through existing and emerging technology, and in particular the continued development of acoustic spectroscopy.



Lubricant emulsion – working concentration

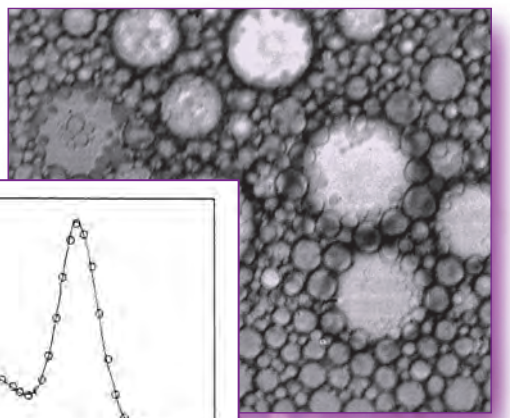


Photo micrograph of lubricant emulsion

SPECIFICATIONS

Technical Specifications	
Particle Size Measurement Range	0.01µm to 1000µm
Measurement Technique	Acoustic Spectroscopy
Patents Granted	US Patent No. 5,121,629
	European Patent No. 0605 409
	German Patent No. 690 25 205.6
	Japan Patent No. 1,901,542
Acoustic Generation and Detection System:	4 broad band transducers with digital signal processing
Sample Requirements:	Volume: ≈ 300ml – 450ml depending on cell
Particle Concentration:	0.5 to 80 volume percent (depending on system)
Measured Parameters:	Attenuation: 0.02 – 2000dB/cm
	Velocity: 900 – 2500m/s
	Frequency: 1 -150 MHz.
Temperature Range:	5°C to 55°C. Accuracy ±0.1°C
Chemical Compatibility:	Samples must be inert in contact with the following materials: 316 Stainless Steel, PTFE, Perlast, Epoxy Resin and fused silica.
Sample Cells:	Custom designed sample cells including flow cells are available as options.
Repeatability:	Variation in results from one sample (multiple tests) on one instrument better than ± 1%.
Software and Data Processing	
Minimum Computer Requirements Software operates on IBM compatible PC running Windows' 98 or NT 4.0 and above operating system. Minimum Hardware requirements: Pentium 150MHz processor, 64Mb RAM, 1Gb hard disc, SVGA monitor and graphics card. 3.5" 1.4Mb Floppy Disc Drive. 3-off RS232 communication ports.	
Theoretical Data Generation A range of proprietary mathematical models is used in the software for the analysis of attenuation data. The program contains a database of physical properties and can be expanded by the user.	
Dimensions:	Ultrasizer SV: 830mm (w) x 422mm (d) x 660-830mm (h) Weight: 75Kg
	Heater Chiller Unit: 225mm (w) x 360mm (d) x 352mm (h) Weight: 18Kg
Power Requirements:	100V to 240V 50/60Hz with standard IEC inlet socket.
Power Consumption:	Ultrasizer SV: 750W max.
	Heater Chiller Unit: 1540W max.

References

1. ***J W Strutt Lord Rayleigh***, "*Theory of Sound*", *Dover, New York, reprint, 1945 originally published 1879.*

2. ***P S Epstein & J S Carhart***, "*The Absorption of Sound in Suspensions and Emulsions – Water Fog in Air*", *Journal of the Acoustic Society of America, 25(3), 553-565, May 1953.*

3. ***J R Allegra & S A Hawley***, "*Attenuation of Sound in Suspensions and Emulsions: Theory and Experiments*", *Journal of the Acoustic Society of America, 51(5), 1545-1564, 1972.*

Setting standards of excellence – our commitment to you

The people at Malvern Instruments are innovators, not just in product design, but in every area of business. Malvern has invested to achieve ISO9001 with TickIt accreditation – we accept nothing short of excellence. As many of our systems are used in the toughest regulatory markets, product validation and development traceability are key commitments to our customers.

Malvern trained specialists are available in more than 50 countries to assist with applications development and to advise on and analyze 'difficult' samples. Our laboratory facilities in North and South America, Europe and Asia routinely run thousands of customer samples every year.

Our innovative approach to customer service is illustrated by the development of after-sales remote diagnostics.

Malvern Instruments' service specialists can access and control systems via standard telephone lines in order to minimize down time and reduce cost.

In many industries particle size analysis has become a key QC parameter. The need to obtain data as close to the line as possible, and to react quickly to that data, is increasingly important. Malvern's Process Systems Group applies its expertise to meet this challenge through the development and production of on-line particle characterization systems for in-process applications.

With laboratory systems and in-process products, Malvern Instruments meets your particle analysis needs from Lab to Line.

For a full description of all

Malvern Instruments' products and services please contact your nearest office.

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