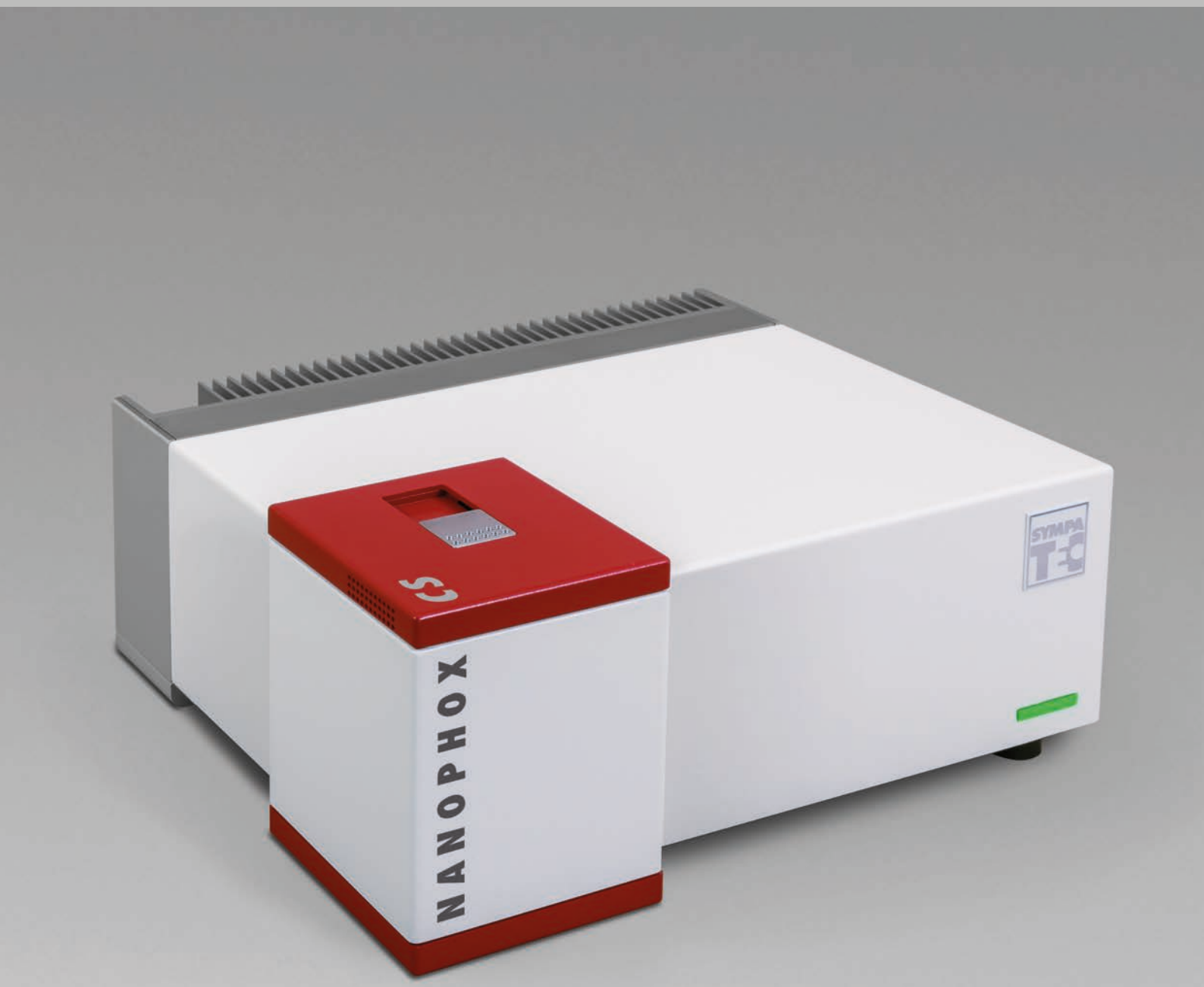
NANOPHOX CS | Dynamic Light Scattering

Particle Measurement | Laboratory

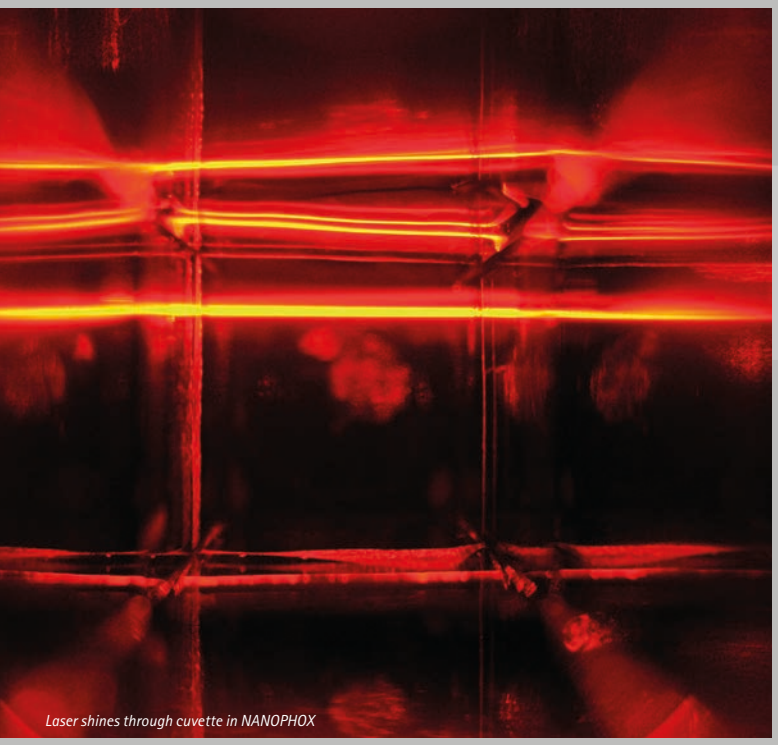
Size and Stability | 0.5 nm to 10,000 nm



Sympatec develops, manufactures, sells, services and supports a range of best instruments for particle size and shape analysis in laboratory and process applications for customers worldwide. With continuous innovations Sympatec makes a prominent contribution to ❱ laser diffraction, ❱ dynamic image analysis, ❱ ultrasonic extinction and ❱ dynamic light scattering.

Photon Cross Correlation Spectroscopy

Measuring Nanoparticles in Liquids



Laser shines through cuvette in NANOPHOX

Trend Nanotechnology

The size reduction of materials plays a central role for the development of innovative products. Applications based on ➔ nanoparticles can lead to advances in the improvement of properties and functionalities of disperse systems.

In pharmacy and biochemistry nanoparticles serve as API carriers or can be charged with molecules for targeted drug delivery. Polymers applied for steric stabilisation or ions serving as electrostatic stabilisation modify the size of the nanoparticles. For fine tuning of their properties and to study the functionality in its natural environment, reliable and efficient technologies and appropriate measuring instruments are required.

Dynamic Light Scattering (DLS)

DLS is a robust, simple and non-contact method for the measurement of particle size and particle size distributions from the nanometre to the submicron range. With high sensitivity it is ideally suited for detection of size changes as a function of time. Process modifications occurring in seconds may be traced in real time.

Within a measuring time of just a few minutes a very high number of particles is captured guaranteeing representative results. Its independence of particle properties, which are difficult to assess or measure (e.g. refractive index, absorption coefficient and material density), constitutes additional advantages for DLS. In the nanometre range these properties largely depend on size. In particular for coated, porous or alloyed particles they are difficult to determine.

For DLS analysis the hydrodynamic diameter is measured through optical detection of the ➔ Brownian molecular motion of particles in a liquid. The thermally agitated liquid molecules collide with the particles causing a random movement or diffusion.

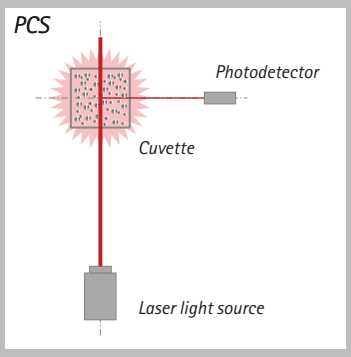
According to the ➔ Stokes-Einstein equation the diffusion velocities are inversely proportional to the size of the particles.

$$D(x) = \frac{k_B \cdot T}{3 \cdot \pi \cdot \eta \cdot x}$$

PCS as conventional technology

The principle of DLS is traditionally realised with Photon Correlation Spectroscopy (PCS). A laser is transmitted through the sample. With the help of a photodetector the scattered light intensity is monitored over time and then ➔ autocorrelated. The particle size distribution can be calculated with the correlation function.

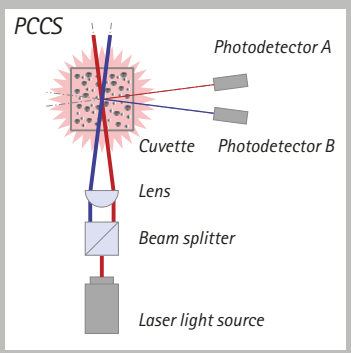
The size analysis with PCS is only valid for single scattered light. Samples of high solids concentration show a large proportion of ➔ multiply scattered light and the method reaches its limitations. To avoid incorrect data on particle distributions and to generate reliable measuring results, samples have to be diluted to a high ratio. In this way significant modifications of the particle properties are likely to occur.



PCCS as key technology

By applying an innovative light scattering technique using Photon Cross-Correlation Spectroscopy (PCCS) we are able to provide concurrent measurements of particle size and stability in opaque suspensions and emulsions.

The outstanding technical features of 3D cross-correlation are the acquisition of two separately generated scattered light intensities and its cross-correlation. The single scattered light proportion is thus separated from the multiply scattered part. A single laser beam is split into two separate beams of identical intensity and superimposed in one sample. Two independent scattering waves are then recorded with one detector for each wave, thus ensuring the exact signal interpretation.



➔ **Nanoparticles** *according to ISO definition are objects with 3 dimensions in the nano range between approx. 1 nm to 100 nm.*

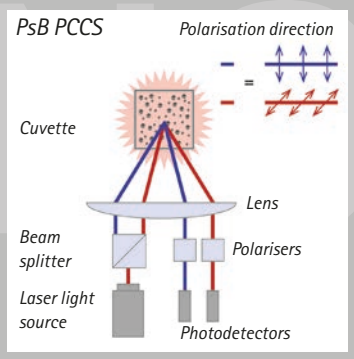
➔ **Brownian molecular motion** *is the thermal motion of particles in liquids. It is measured for determination of the particle size.*

➔ **Stokes-Einstein equation** *establishes the correlation between viscosity η and temperature T of the liquid and the size x of the assumed spherical particles and its velocity. This defines the diffusion coefficient D(x) which serves for calculation of the hydrodynamic particle diameter x. k_B is the Boltzmann constant. If*

Nanoparticle Measurement in Primary Condition

Polarisation-separated Backscatter PCCS

The latest evolution of PCCS brings the Polarisation-separated Backscatter PCCS (PsB PCCS) in the NANOPHOX CS to fruition for the first time. The decoupled intensity patterns of the vertically and parallel polarised laser beams are separately recorded, cross-correlated and evaluated. The combination of backscattering and polarisation separation delivers a signal quality on a new level. Reliably repeatable measurement results with improved accuracy are achieved at up to a hundred times higher sample concentrations and up to ten times faster measurements.



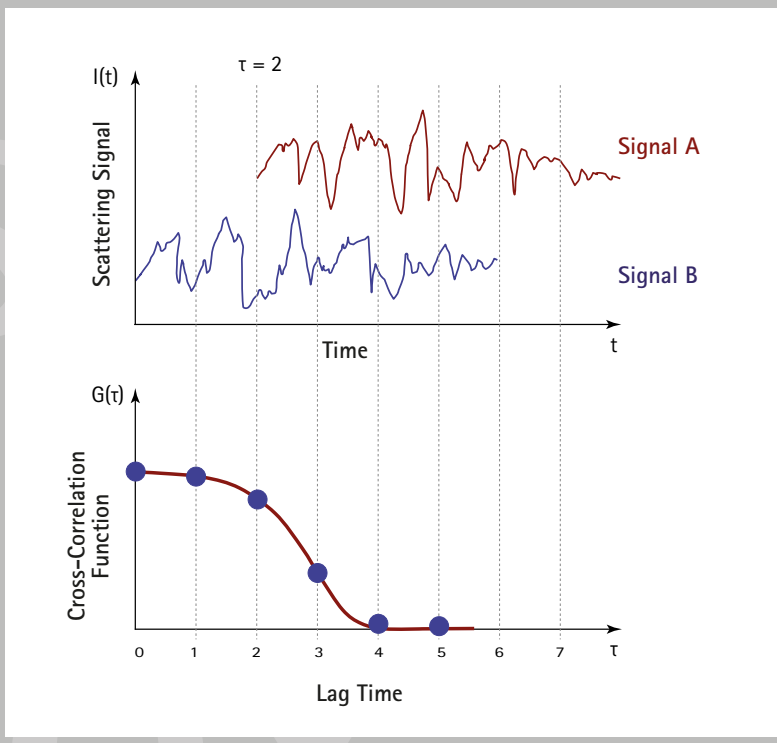
Wide range of applications
NANOPHOX CS covers a wide particle size range from 0.5 nm to 10,000 nm. The size analysis is independent of the concentration and even possible up to approx. 40 vol.%. The wide concentration range is made possible by combining PCS and PCCS in one instrument.

In a precisely adjustable temperature range from 0°C to 90°C, the behaviour and change of nanodispersions can be observed.

NANOPHOX CS proves its special performance in the direct stability analysis of sterically resp. electrostatically stabilising, highly concentrated nanodispersions. Regardless of whether the particles are stabilised by electrical charges, by polymers or surfactants, NANOPHOX CS reliably analyses the aggregation or agglomeration behaviour. This results in new possibilities are opened up for characterising areas of high turbidity dispersions that have been little explored so far, such as particle-particle interactions and changes in dynamic viscosity.

Simply reliable results
NANOPHOX CS guarantees reliable results without time-consuming sample preparation. Errors in manual preparation, which often lead to changes in particle size distribution, stability or functionality of the sample, are avoided. Since the NANOPHOX CS measures even highly concentrated samples, usually undiluted, it contributes significantly to minimising errors and increasing the quality of the measurements. The automatic positioning of the cuvette and the precise adjustment of the laser intensity to optimise the measurement signal are particularly user-friendly. This supports quick and easy measurements.

Sample handling | Cuvettes
The proven acrylic and glass cuvettes, solvent-resistant disposable cuvettes (up to 4 ml) and micro cuvettes for small sample volumes (50 µl to 2.7 ml) are available.



Solvent-resistant cuvettes

[3] *viscosity and temperature remain unchanged, fine particles move faster than coarse particles.*

➔ **Autocorrelation:** *The scattered signal is correlated with itself at different points in time (a comparison of the time lagged and the original function).*

➔ **Multiply scattered light** *occurs if the incident laser light is scattered more than once by particles and disturbs the scattering wave on its way to the detector.*

Applications | Nanoparticles in a Unique Range of Concentration

Reliable Determination of Stability and Polydispersity

Inks

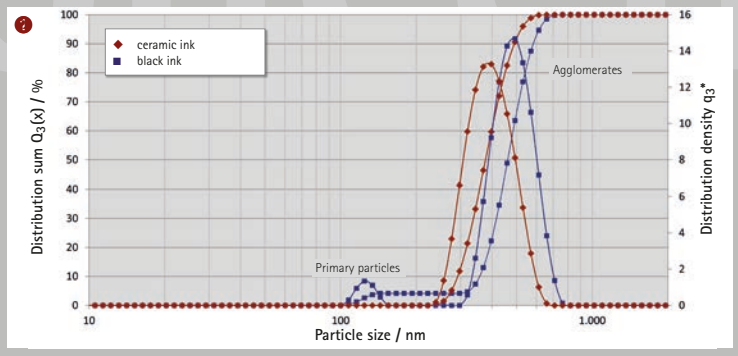
Inks and paints, as used in printers, consist of several components to achieve properties such as colour strength, light fastness or document fastness. One essential component is the pigment that gives the suspension its colour.

Important quality characteristics are the particle size and the distribution width of the pigment particles. They influence the colour strength, surface quality, UV resistance and the durability or stability of the end product.

With inks, there is a risk that coarse pigments, e.g. agglomerates, will block the nozzles of the printing mechanism.

❶ The ceramic ink has a monomodal distribution and is in the size range from 256 nm to 613 nm. The black ink shows a bimodal distribution. The primary pigments are in the size range of 100 nm to 150 nm. A large part of the volume is agglomerates from 300 nm to 750 nm. This ink is of poor quality and could clog the cartridge.

To achieve optimal primary particle sizes, the suspensions are finely ground. The milling progress and the final fineness of the pigments can be investigated by high-resolution particle size analyses with PsB PCCS in the original state. Particle size, distribution width and polydispersity are determined just as reliably with this variant of dynamic light scattering as the stability of the dispersion.



Casein proteins in milk

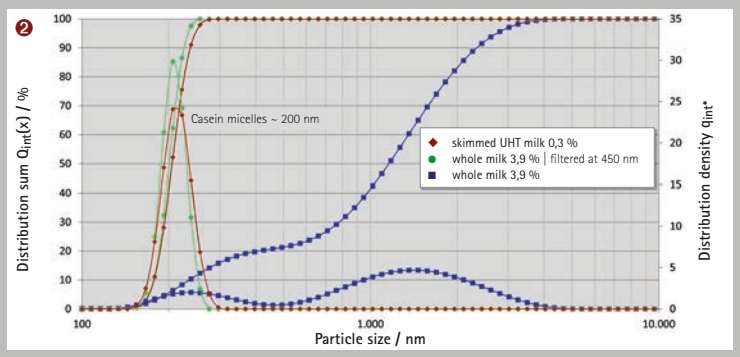
Milk is a colloidal dispersion consisting of partially dissolved minerals, vitamins, lactose, proteins and fat droplets.

The analysis of bimodal particle size distributions of casein micelles and fat droplets in milk provides valuable information about the quality of dairy products.

❷ Two different types of milk with low and high fat content (skimmed milk with 0.3 % and whole milk with 3.9 % fat content) illustrate the existence of two particle populations in the analysis – that of the coarser fat droplets and that of the finer casein micelles.

The skimmed, high-heat UHT milk with a fat content of only 0.3 % shows a mean particle size of 205 nm. The fat content is negligibly small, predominantly casein micelles are detected (red curve). The whole milk with a fat content of 3.9 % shows two modes, one at 250 nm and one at 1,300 nm (blue curve). The fine peak represents the population of casein micelles and the coarse one that of fat globules. After filtration at 450 nm, the concentration of fat globules is negligible. Only the casein micelles with an average particle size of 200 nm are visible in the particle size distribution (green curve).

The advantage of NANOPHOX CS is the possibility to analyse undiluted samples with excellent repeatability and comparability.



➔ *The* **intelligent incoming inspection** *of the NANOPHOX CS, the measurability of a sample can be determined quickly. Turbid suspensions such as inks or emulsions such as milk are easily analysed in their original states.*

Applications | Measurements in Original Concentration

Precise Results without Dilution | High Sensitivity to Size Change

Ophthalmic emulsion

In pharmaceutical applications particulate systems are applied as drug carriers. Its precise characterisation is a basic condition for the approval of the product. Pharmaceutical emulsions such as eye drops are subject to numerous quality characteristics, one of which is the droplet size of the disperse phase within the emulsion.

Droplets being too fine or too coarse may reduce the efficiency or may become a risk for the eye. The ophthalmic emulsion analysed in this example is a white, very turbid homogeneous liquid. With the PCCS technology the sample can be measured without dilution. ❶ The result shows the expected mono-modal size distribution with a mean diameter of 120 nm. When measuring this sample in PCS mode the size distribution shows a mean diameter of just 68 nm and clearly remains below the expected specifications. The strong deviations can be explained by the influence of multiple scattering. Only after dilution to a ratio of 1:100 in pure water the size distribution of the droplets is determined correctly.

NANOPHOX measures the sample in its original condition, minimising the effect of sample contamination. Dilutions which might change the chemical properties can be discounted. Based on the robust and easy to use measuring method, the instrument is ideally suited for production and quality control of pharmaceutical emulsions.

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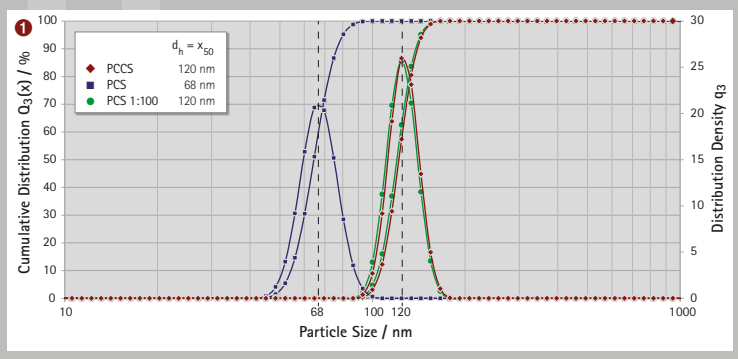

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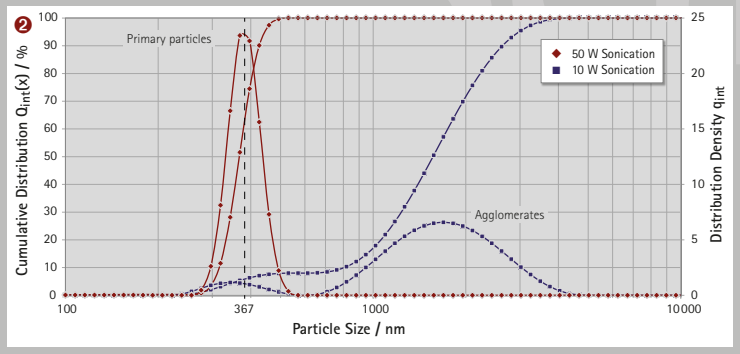


Alumina powder

A variety of nano and micro particles are synthesized by dry production processes. For numerous industrial applications of alumina powders the particles have to be prepared as suspensions before introducing them for efficient product improvements. This introduces the challenge of preparing stable dispersions. Agglomerates may affect the functionality of the product.

❷ For measurement of the dispersion quality a suspension was prepared of 0.05 % by weight alumina powder in water and treated with an ultrasonic probe. The energy input of 50 W yields a monomodal distribution with a diameter of 367 nm. Reducing the ultrasonic power to 10 W provides a significantly worse dispersion quality. A remarkable portion of coarse agglomerates can be observed in the measured particle size distribution together with a reduced fraction of primary particles.

The strengths of NANOPHOX can be found in the broad concentration range and the sensitive resolution of bimodal particle size distributions. It also very reliably indicates size changes.

With PCCS the result is independent of particle concentration and permits measurements of dispersion quality even in suspensions of high concentrations.

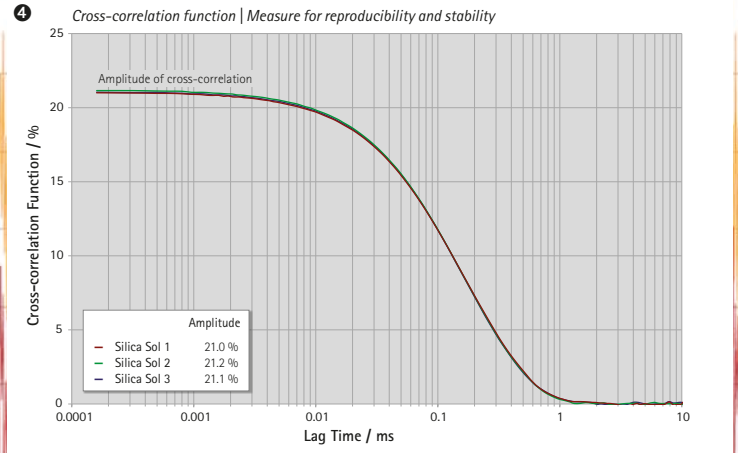
→ *Further typical applications for NANOPHOX CS are polymer emulsions, colloidal silica, pigments and the research of nanomaterials in general, specifically for the automotive industry, electronics, pharma, mechanical engineering, environmental protection and energy.*

PAQXOS Software

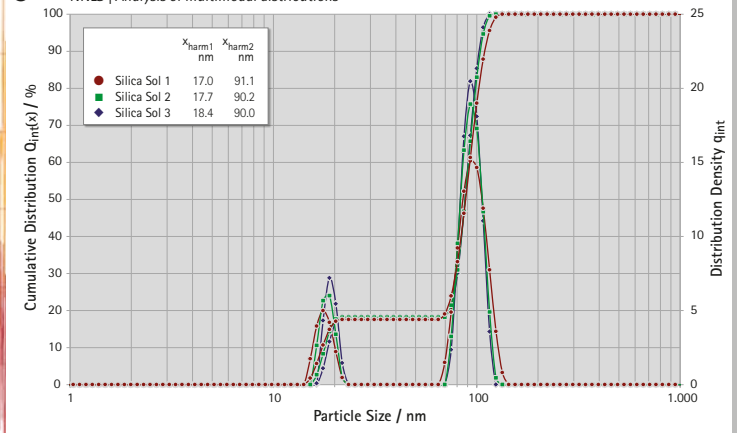
Intelligent Measurement Process and Meaningful Results

Sample Feeding | Measurement | Evaluation

❹ *Cross-correlation function | Measure for reproducibility and stability*



❺ *NNLS | Analysis of multimodal distributions*



PAQXOS combines our collective particle sizing expertise in a user-friendly application software. A step-by-step wizard with built-in expert knowledge also leads the inexperienced user to successful measurements.

After ❶ filling the cuvette, the sample is ❷ loaded into the NANOPHOX CS single-handedly. A simple mouse click starts the measurement. PAQXOS optimally sets all measurement parameters of the particle size analysis and checks the measurability of the sample before starting the measurement. To guarantee a desired level of statistical accuracy and repeatability PAQXOS will make an educated decision for the measurement time, depending on the complexity of the sample. As the measuring signals are ❸ displayed in real time, changes in quality of the measurement may be observed directly. After each run the raw data is stored in the database automatically. This allows for a later change of evaluation parameters and the re-evaluation of the sample without measuring again. For measurements in the range limit, suggestions are made for sample preparation.

PAQXOS provides graphical presentations, tables or paper reports that can be chosen from a variety of predefined and fully customisable formats. Fast and easy graphical display of results out of the measuring database as well as import of measurement parameters from existing measurements are realised by drag and drop. Data browser and filter functions allow the retrieval and presentation of extent analysis data. The intuitive graphical user interface supports the execution of reliable, meaningful measurements.

❹ The cross-correlation function, representing reproducibility and stability, is used to calculate the particle size distribution. In addition to the classic ➔ 2nd Cumulant evaluation PAQXOS offers a distinctly more efficient ❺ Non Negative Least Square (NNLS) algorithm. The NNLS evaluation mode reliably presents polydisperse or ➔ bimodal samples as size distribution. Characteristic values such as mean values of single modes are directly available.
Our ➔ versatile PAQXOS software is ideally suited for demanding production and research applications. It also meets the requirements of FDA-regulated pharmaceutical industries and provides all safety functions, access controls and authentication methods stipulated in regulation 21 CFR Part 11 regarding electronic records and electronic signatures to ensure data integrity and prevent manipulation of records.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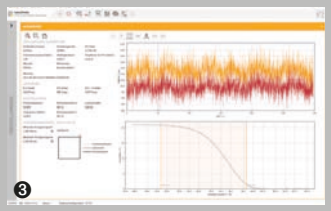
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➔ **2nd Cumulant evaluation** *provides a mean diameter and a polydispersity index (ref ISO 22412). It cannot represent a particle size distribution.*

➔ *A* **bimodal sample** *comprises two particle fractions (modes) of different size distributions.*

➔ *An integrated* **scripting environment** *allows for the programming of elaborate routines to design repeat measurements in an efficient and reproducible manner.*

Furthermore, predefined measurement parameter settings may be implemented as binding **Standard Operating Procedures | SOPs.**

Pulverhaus

Brand Home of Sympatec's Premium Instruments

Development of Innovative Methods for Particulate Systems Characterization

Laser Diffraction | Dynamic Image Analysis | Ultrasonic Extinction | Dynamic Light Scattering

Perspective

"A classic is timeless and at the same time ahead of its time."

The variety of disperse products requires innovative and sustainable technologies to master the challenges in today's research, development, quality and production control.

With dry dispersion we have introduced product orientation and adaptation to laser diffraction.

The HELOS sensor family and a great range of dispersing units – spearheaded by RODOS – offer you premium performance. Our laser diffraction instruments allow for a significant extension of your particle knowledge concerning size and size distributions.

New questions and desires inevitably arise with unbowed progress. Power of innovation consequently remains key to future developments.

Today, if we encounter application limits of laser diffraction e.g., in suspensions of high optical concentration, we offer efficient solutions with ultrasonic extinction (OPUS).

If particle shape becomes of interest, we provide a great spectrum of powerful solutions with high-speed dynamic image analysis (QICPIC family). Now even sophisticated fibre analysis is amongst the range of multifaceted particle shape aspects.

And in case particles predominantly belong to the nanometre range, we have launched photon cross-correlation spectroscopy as a powerful dynamic light scattering technique. The NANOPHOX CS, with its unique polarisation-separated back-scattering, opens up a previously unattained concentration range.

By nature, we also keep an eye on the production of disperse systems when developing methods of particle characterisation. Hence, you may also trustfully address us in case process control becomes an issue. Laser diffraction with MYTOS, ultrasonic extinction with OPUS and dynamic image analysis with PICTOS are hundredfold approved process applications from Sympatec. Designed with a consistent technological basis, our in-, on-, at-line systems reliably deliver results that are perfectly comparable to those of our laboratory instruments – most accurate, reproducible and at the shortest measuring times.

As "Particle People" we originate from the powder technology field. This is why we have a natural approach to process engineering and the production of disperse systems. The collective particle expertise of our physicists, mathematicians, computer scientists, engineers, electronic and mechanic technicians is built into our instruments.

Your particles in the best of hands with us.

Particle Measurement and Know-how from Pulverhaus

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