QICPIC | RODOS & Co. | Dynamic Image Analysis

Particle Measurement | Laboratory
Size and Shape | < 1 μm to 34,000 μm















The Universal Shapefinder

The Discovery of Shape - Particle after Particle after Particle



The spherical model of laser diffraction

Laser diffraction has been recognized as a technology for fast and reliable determination of particle size distributions. For decades it has been the dominant method for the analysis of particle collectives in research and industry. The diffraction method assumes the validity of the spherical particle model.

»All particles are spheres? Let us simply check ...«

With the determination of particle size as the equivalent diameter of a sphere characteristics of different particle shapes are assumed as integral values. For many applications this calculation is viable. The assumption of spherically shaped particles is sufficiently precise to characterize the relevant physical properties of a particle collective.

Size and shape – closer to reality with image analysis

For different applications not only particle size but also particle shape determines relevant properties of the collective. Flowability, adhesion and friction, hardness, compressibility, roughness and attrition or solubility of disperse systems can better be characterized with supplementary shape descriptors such as sphericity, aspect ratio or convexity. Differences in the quality of batches, showing identical particle size distributions become clearer with the additional evaluation of particle shape.

Shape recognition of single particles

With laser diffraction the particle size distribution is determined by the characteristic diffraction pattern of a particle collective. In contrast, image analysis captures the physical properties of each single particle. The distribution of a property such as size or shape descriptor can thus be resolved in nearly any class.

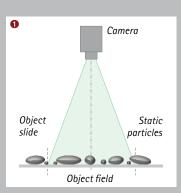
With image analysis even smallest amounts of over or under sized particles may be detected. Even single particles with specific geometric properties such as aggregates, fractures or foreign particles are traceable.

Image analysis operates in a similar way to a modern microscope: a digital camera with special optics captures the particles within the frame. Physical information about particle properties is transmitted to a computer. For each single particle in the image size and shape descriptors are determined by evaluation software.

Static image analysis 0

Particles are prepared on to an object slide with a fixed orientation, which may generate systematic errors. Particles are normally presented with their largest area facing the camera, with little clue as to the real shape. Depending on nature and preparation of the sample, dispersions may be poor with overlaying particles and hidden fine fractions.

The ⊕ statistical significance of static image analysis has the most substantial limitations. The particle number is significantly constrained

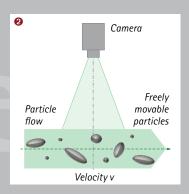


due to the limited size of the object slide leading to poor statistical analysis and larger error for broadly distributed particle collectives.

Dynamic image analysis 2

Considering dynamic image analysis the particles are streaming continuously through the measuring volume controlled by the camera. The free movement leads to random orientation of the particles. From the different perspectives their actual shape and size distribution can be accurately determined.

With a continuous feed of dispersed particles reliable and representative results are achieved based on a statistically significant number of particles. By closely controlling the concentration of the particle flow the overlay of particles is prevented.





Innovations for High Performance Image Analysis

Dynamic image analysis with QICPIC

Applying components of highest performance our modular sensor QICPIC develops the full power of dynamic image analysis.

High speed image analysis
Using a pulsed light source with
illumination times of less than
1 nanosecond the particles are optically frozen while a high-resolution,
high-speed camera captures the
razor-sharp particle projections with
a frequency of up to 500 frames
per second. With powerful algorithms QICPIC is evaluating millions
of particles in shortest time and
guarantees outstanding statistically
relevant results.

Modular system design

With a selection of up to 4 from 7 available precision lenses the total measuring range from below 1 µm to 34 mm is covered seamlessly. The wide dynamic measuring range of the optical modules (1:2,000) also means the characterization of disperse systems with broad distributions is easily achievable. As the images of each single particle is evaluated individually the quality of singling of particles is of prime importance. Applying dosing and dispersing units of modular design for dry and wet products QICPIC is flexible to adapt to powders, granules, fibres, suspensions and emulsions. This ensures a product-specific characterization matching the true nature of the application. The extremely short exposure time of the sensor and the high image frequency are adjusted to the dry disperser RODOS, which has been proven in dry powder

applications many thousands of times. Thus the particles or fibres, perfectly dispersed in an aerosol free jet, are accurately and sharply imaged even with particle velocities of up to 100 metres per second.

Meaningful results

A specially developed optical set-up sets the standards for precision of the measuring results. The particle outlines, generated in a parallel beam, are imaged with telecentric optics practically free of aberrations and with highest contrast – even for transparent particles. The camera precisely captures the particle projections with 256 greyscale intensities and a resolution of up to 4 megapixels. The raw data is transmitted to the computer for storage and evaluation with transfer rates up to 25 gigabit per second.

Applying powerful algorithms the evaluation software provides all relevant size and shape descriptors within seconds after the measurement. Measures for length, width, equivalent circumference and diameter of a circle describe the size of a particle. Sphericity, aspect ratio and convexity provide information about the shape. Fibres can also be properly evaluated with length, diameter, straightness and elongation. All parameters may either be presented as distribution for the whole sample or individually for each single particle.

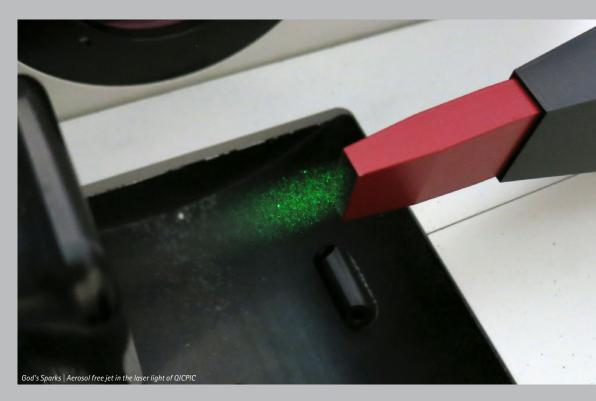
A particle gallery with numerous selection and filter parameters supports the generation of specific and meaningful results. With the

recorded particle video stored in the data-base the complete measurement can be viewed retrospectively.

Image analysis in the process

The powerful and proven system components of QICPIC are also available for process integration. With PICTOS & Co disperser and sensor of QICPIC are integrated into one robust body, developed specifically for dry and wet on-line applications. Elaborate technologies for representative in-line sampling like TWISTER and many possibilities for customer-specific sample feeding make image analysis an excellent option for process control.

The quality of dispersion determines the significance and reliability of results – also for image analysis.











Dry Dispersion

with RODOS | OASIS | VIBRI | ASPIROS | MULTISAMPLER

Powerful Dry Dispersion in a Free Aerosol Jet









Dry dispersion

For a product-adapted characterization powders, fibres and granules need to be analysed dry. The successful interplay of dosing and dispersion is the basis for significant and dependable results. Consistent, reliable dosing generates a constant mass-flow.

Product-adapted dispersion forces consecutively provide an effective singling of particles in the free aerosol jet. This works for tightly bound agglomerates as reliably as the smooth and gentle dispersion of brittle and coarse particles.

RODOS

For a broad spectrum of dry and cohesive products ranging from 5 μ m to 4,000 μ m RODOS is the first choice. In just a few seconds sample quantities from below 1 g to 1,000 g are steadily dispersed and analysed. The application of energy for optimum dispersion is adjusted to the product using precise control of the primary air pressure.

No other dispersing procedure is of comparable performance. Speed, reproducibility, comparability and a high statistic relevance for large sample volumes are specifically significant.

OASIS = RODOS & SUCELL
The OASIS system provides an easy change between dry and wet measurement. A → SUCELL mounted on top of RODOS serves a versatile pump loop for suspensions and emulsions with particles from



 $2 \mu m$ to 2,000 μm . For dry samples the well-known aerosol free jet of RODOS is ready for use.

VIBRI

With the controllable precision vibratory feeder VIBRI the ⊕ product-specific supply of sample to the funnel of RODOS is maintained. A constant, optimized particle flow is precisely adjusted with feed-rate and funnel height.

ASPIROS

Small amounts of precious, active or toxic substances can be safely analysed with the micro-dosing device ASPIROS mounted to the RODOS disperser. The sample tubes are filled with milligrams of sample and capped in a glove box or fume cupboard. After loading the closed tubes into ASPIROS a barcode

reader identifies the samples. The requested sample-specific conditions for dispersion are automatically set, the tube is opened and the sample aspired into the RODOS injector for dispersion and analysis. With a closed measuring zone, product exposure to the environment is inhibited.

MULTISAMPLER dry

A fully automatic sample handling system for high sample throughput is available with MULTISAMPLER. In batch operation up to 70 vials with 39 ml volume each are fed from a tray into the system. Sample identification is achieved with a data matrix code etched to the bottom of the glasses. The complete amount of sample is captured into the airflow of the RODOS injector, dispersed in a free jet and measured with QICPIC.

Application QICPIC & RODOS



High Resolution Shape Analysis of Dry and Cohesive Particle Systems Efficient Dry Dispersion | Statistically Relevant and Quick Results

Quality control of methylcellulose

Methylcellulose is a water soluble, white, cohesive powder with multiple → industrial applications.

Typically, the physical properties of this fibrous product substantially determine its desired characteristics for final use. Correspondingly, the quality control follows shape descriptors such as length, diameter or elongation and straightness of the fibres.

QICPIC & RODOS | M7

The injection disperser RODOS provides reliable particle thinning. No special preparation is required to supply about 20 g of powder with the dosing chute VIBRI for dispersion into a free aerosol jet.

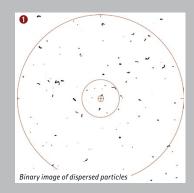
With the optical module M7 QICPIC covers a wide dynamic measuring range from about 4 μ m to 8,500 μ m. Almost 6 million particles are captured within less than 100 seconds. The \odot binary live image of the sensor visualises the complete dispersion of the methylcellulose fibres.

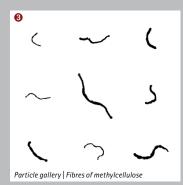
The presentation of the \odot size distribution of the fibres follows the length distribution (O_1) differentiated according to length and diameter. In a first step the sample can be characterized with a mean fibre length of $X_{50,LEFI} = 116.37 \, \mu m$ and a mean fibre diameter of $X_{50,DIFI} = 28.40 \, \mu m$. A clear, qualitative impression of individual fibres is provided with the \odot particle gallery of the PAQXOS evaluation software.

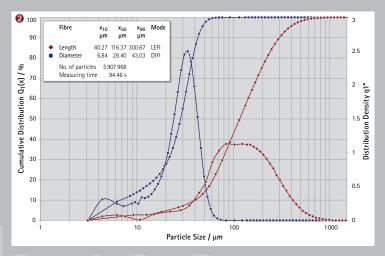
The additional diagrams show the elongation and straightness in relation to length of the fibre (LEFI).

The ratio of diameter to length of a fibre defines the delongation. Its relationship provides values between zero and one. Slim fibres are characterized with smaller values, stubby fibres show larger values. Here, the value for elongation is decreasing continuously. The fibres become slimmer and slimmer while fibre length is increasing.

Longer fibres typically tend to become crooked and curled. This can be expressed by **6** straightness

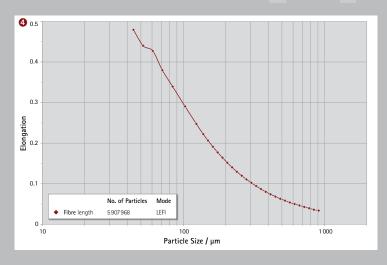


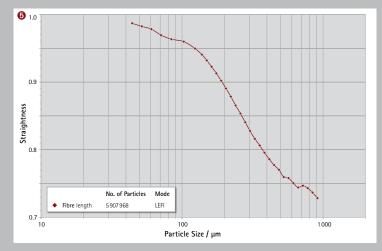




which is defined by the ratio of maximum Feret diameter to the length of a fibre. Values close to one describe

straight fibres. The smaller this value the more crooked the fibres are. The curve shows a typical progression.













Dry Dispersion

with GRADIS | FIBROS

Gently Dispersed in Free Fall

Gravity dispersion

Dispersion in the free fall is an option for dry, free flowing products, which do not tend to agglomerate. Specially coarser or sensitive powders and granules are gently dispersed when accelerated by gravity on their way into the measuring zone.

Constant dosing also in this case has an important impact on a reproducible dispersion. A continuous particle flow, optimized for the product, is guaranteed by the intelligently controlled VIBRI.

GRADIS

Using the free-fall shaft, GRADIS masters the careful dispersion of compact particles and granules in a size range from a few micrometers

up to 10 millimeters. Elongated or curled fibres may even be much longer. For optimum presentation of the individual particles in the measuring zone of QICPIC, a selection of outlet tips and apertures is provided.

If GRADIS is operated with ⊕ extraction unit, an integrated flow control supports the gravity dispersion. In case additional dispersion energy is required impact cascades can be provided. For complete recovery of the sample after analysis an optional collection tray may be supplied.

The dispersion of products, which are easily charged by electrostatics, succeeds with the GRADIS fall-shaft coated with a conductive surface.



Dispersion of fibres with FIBROS

FIBROS provides a smooth separation and dosing of dry and heavily entangled fibre collectives. This pre-disperser and dosing device can be applied mounted on a standard GRADIS or as a stand-alone system with shortened fall-shaft.

With the help of diverse combinations of standard laboratory sieves and specific brushes, FIBROS can be adapted to numerous applications for efficient dispersion of dry fibres in a range of 500 μm to 30,000 μm length and 5 μm to 5,000 μm diameter.

Combining a topside rotating brush and static teasel, a bottom side rotating air jet nozzle with an intermediate sieve, the fibres are completely dispersed within a few minutes. The separated fibres are then carried in an air stream through the vertical GRADIS fall-shaft to the measuring zone and sucked away after measurement.

Setting and control of all dispersing parameters are either managed via software or the operation keypad. The control parameters comprise of the rotational speed of the brush and air jet nozzle, the primary pressure and the vacuum within the system.

Ease of access to the dispersing unit and fall-shaft allow for a simple and thorough cleaning of the parts in contact with the fibres.

Application QICPIC & GRADIS



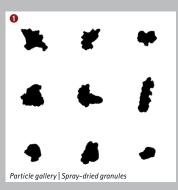
Illustrative Characterization of Free-flowing Particle Systems Diverse Evaluation Modes | Outstanding Repeatability

Quality control of spray granules

During spray-drying liquid media are transformed into compact and homogenous granules with atomisation and drying layer by layer. The aim of spray-drying is the generation of dry, dust-free, medium to coarse dispersed particle systems with well-defined physical properties. Flowability, attrition, strength, solubility, bulk density and also dosage therefore directly depend on particle size and shape. Achievement of optimum qualities is guaranteed with reliable, statistically relevant image analysis.

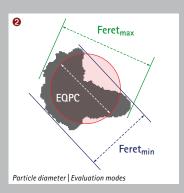
QICPIC & GRADIS | M7

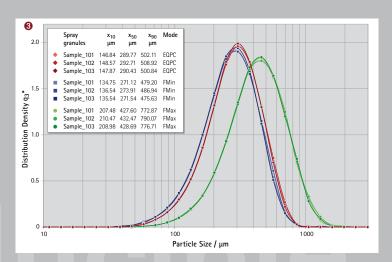
In this application about 12 g of spray-dried, non-riffled milk powder is fed into the GRADIS fall-shaft with the
VIBRI chute. The sample is smoothly dispersed in free fall and then analysed. Within 300 seconds about 1.5 million particles are captured with a frame rate of 175 images per second. The measurements stored as raw data can also be evaluated retrospectively with several modes or reviewed in the particle video. The 1 gallery provides a visual first impression of the particle projections of a selection of typical granules.

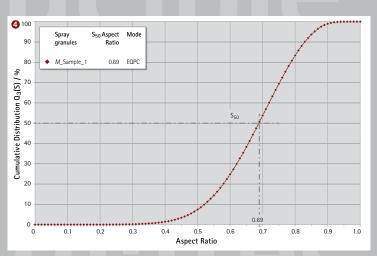


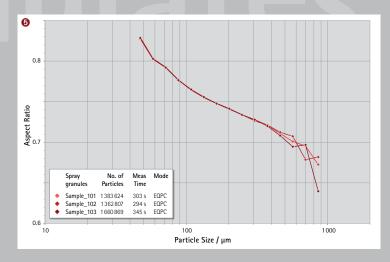
Three sub-samples of the same batch are measured and characterized with three 2 evaluation modes each with respect to their diameter. EQPC (EQuivalent Projection area of a Circle) results from the transformation of the projection area of the real particle into a circle of equal area. Feret diameters describe the dimensions of a particle through the distance of two parallel tangents (calliper gauge model). Minimum and maximum Feret diameters (FMin and FMax) display the shortest and the longest extension of a particle, respectively.

The @ particle size distribution shows the excellent reproducibility of the measurements. Due to the rugged shape of the sprayed granules EQPC and FMin values are close together. FMax values meanwhile indicate slightly elongated particles. This is confirmed by the presentation of the 4 cumulative Q distribution related to the
→ aspect ratio (FMin to FMax). The particles range from 0.4 (elongated) to 0.9 (compact) with a mean aspect ratio of $S_{50} \approx 0.69$. In the presentation of the 3 aspect ratio related to the size it is shown that smaller particles tend to be more compact and round than larger ones.

















Wet Dispersion

with MIXCEL | LIXELL | LIQXI | FLOWCELL | MULTISAMPLER

Product-specific Diversity





Wet Dispersion

For particle systems in liquid form such as suspensions and emulsions the natural way of analysis is wet.

The basis for reliable measuring results is a flexibly adaptable, product compliant dispersion that generates a homogenous flow of single particles. Pumping, stirring and flow through a measuring cuvette already cause a dispersion of particles. For agglomerating products capillary and cavitation forces need to be enhanced appropriately. The optical concentration within the measuring volume is determined through the transmission depth of the flow cuvette. The analysis volume ranges between 20 millilitre to over 20 litres (per minute) depending on the disperser.



MIXCEL

With MIXCEL size, shape and number of particles in suspensions or emulsions can be determined with high precision over a range of 1 µm to 3,000 µm. This applies in the same way to disperse systems of complex mixtures, broad distributions or particles of high density with high viscous or organic dispersion liquids.

Controllable ultrasound and a unique double stirrer unit serve for homogenous mixing and complete dispersion. With a centrifugal pump the sample is then transported through the measuring circuit as a representative particle flow.

A selection of flow-optimized cuvettes with depths of 30 µm to 6 mm provides flexible adaptation to the specific application. The jacketed ⊕ basin holds liquid volumes between 250 ml and 1,000 ml. It also serves as a heat exchanger and provides an effective temperature control between 0°C and 90°C. Sample feed, additive supply and cleaning of the basin is fully automated with the top of range model.

LIXELL & LIQXI

Due to its open and modular design LIXELL represents the most versatile wet dispersion system over a particle size range of 1 µm to 2,000 µm. Numerous applications can be realised with a variety of ⊕ cuvettes and application kits starting at a sample volume of 20 ml. True-to-method measuring applications are set-up in combination with wet dosing system LIQXI. A choice of stirrers and optional flow control baffles guarantee for a reliable homogenization in glass



beakers with 250 ml and 400 ml volume, respectively. Suspensions with particles up to 500 µm are being fed to the measuring cuvette as a representative product flow with a peristaltic pump. Depending on the set-up, LIXELL can be operated in flow-through or circulation mode. Chemically resistant sealing and tubing materials are available for aggressive media.

FLOWCELL

The large volume flow cuvette FLOWCELL is applicable for the image analysis of coarse suspensions with particles from 10 µm to more than 10,000 µm. The particle size for soft disperse materials extends even to 16 mm. Flow diameters of 10 mm and 20 mm enable a sample throughput of more than 10 l/min and 20 l/min, respectively, to achieve representative particle numbers.

MULTISAMPLER wet

The combination of MIXCEL and MULTISAMPLER supports efficient laboratory analysis with high sample throughput. The system can run up to 70 sample glasses with 39 ml each without operator input. The glasses, which are sealed with a silicone septum, carry a data matrix code for sample identification.

Application QICPIC & FLOWCELL



Statistically Relevant and Illustrative Image Analysis of Coarse Particles Wide Dynamic Measuring Range | Shape Descriptor Sphericity

Premium pulpy fruit juice

Coarse, natural fruit components, such as pulp, play a major role in the preparation of precious, high-quality fruit juices. In order to keep fruit cells in best condition for the desired final product a gentle handling during manufacture and filling is necessary. Valuable information about the pulp quality is collected by a reliable size and shape characterization of the solids in the juice. This ensures the adherence to quality standards from incoming goods and processing to the finished premium juice.

QICPIC & FLOWCELL | M8

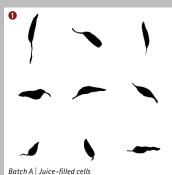
The analysis of coarse fruit cells to a length of more than 10 mm is performed with measuring range M8. From a 20 litre container mounted above the QICPIC the fruit juice sample is led through a FLOWCELL with a 10 mm flow cross-section. A peristaltic pump, which is positioned behind the cuvette, provides a constant sample flow during the measurement. For the ⊕ evaluation of the particle size the mean Feret diameter (FMean) is applied.

To determine the quality of incoming raw fruit juice, two batches were analysed. The first difference in quality can be seen when looking at the particle gallery: • intact, juice filled cells are found in batch A and • disintegrated cells in batch B.

The \odot particle size distribution now quantifies the differences in quality of the supplied raw juices. Batch A shows a mean particle size of $x_{50} \approx 9,785~\mu m$ and a quite narrow distribution indicating many uniform fruit sacs. Batch B features a significantly finer $x_{50} \approx 5,230~\mu m$ together with a wide distribution, due to the destruction and shredding of the cell structures. Also the number of particles in batch B is significantly higher showing approximately 5 million particles compared to 1.2 million in batch A.

Defibration of the cell walls also creates modified, dissolved particle shapes. This is visible in shape descriptors such as ③ sphericity, a measure of closeness to a perfect sphere. The smaller the value for the sphericity the more irregular a

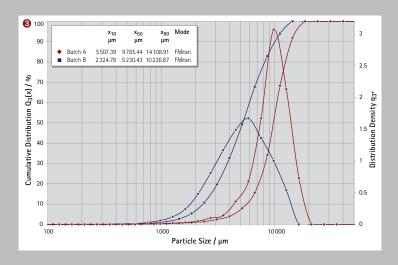




particle is. Thus the 4 cumulative curve of batch B consistently exhibits smaller values for the sphericity of



the particles than the cumulative curve of batch A with its undamaged, naturally shaped, sac-like fruit cells.



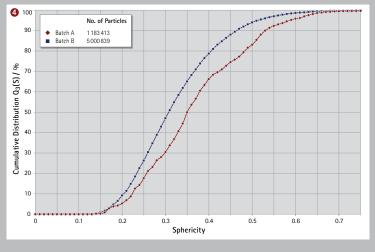










Image Analysis on-line

Application PICTOS & TWISTER

Reliable Detection of Oversized Particles Greatest Sensitivity | Production Control in Real-Time

Production of EPS Raw Beads

The foaming of Expandable Polystyrene (EPS) materials requires high density EPS beads as a raw material. After its initial polymerization the compact and granular EPS is rinsed, dried, sieved and packed. Depending on its final → application, the EPS beads need to fulfil specific requirements regarding particle size distribution.

The raw beads are separated into different product qualities with tight specifications applying large-scale screen decks. While sieving smallest amounts of oversized grain are indicators for a beginning screen damage. In order to avoid production down time and expensive reprocessing of rejected batches screen failures need to be detected reliably at an early stage.

Due to its ability to detect single particles dynamic image analysis provides exceptional sensitivity.

Smallest amounts of coarse material is reliably detected even within narrow size distributions.

PICTOS & TWISTER

The application showcases the monitoring of screen decks of nine product lines, operating in parallel. Sampling is realized with the dynamic probe TWISTER continuously scanning the product flow in each line. The probe tip is travelling the entire pipe cross-section on a spiral path in order to yield a representative sample.

Line by line, the vacuum of the integrated RODOS injector aspirates the sample and creates a perfectly



dispersed free aerosol jet inside the PICTOS measuring zone. The 10 mm RODOS nozzle together with the optical module M8 of PICTOS is mastering a measuring range of about 10 µm up to 3,500 µm.

Two batches of EPS beads have been analysed. The first batch (EPS A) was contaminated with a small amount of coarser material in order to test the sensitivity of the on-line image analysis. Evaluation of particle size is based on diameter-oriented EQPC (EQuivalent Projection area of a Circle).

The • size distributions of both batches display the narrow grain range of about 900 μ m to 1,500 μ m, which was result of the fractionation. There is no significant deviation between the two batches, regarding the respective values for mean particle diameter of $x_{50,EPS_A} \approx 1,157 \ \mu$ m and $x_{50,EPS_B} \approx 1,151 \ \mu$ m.

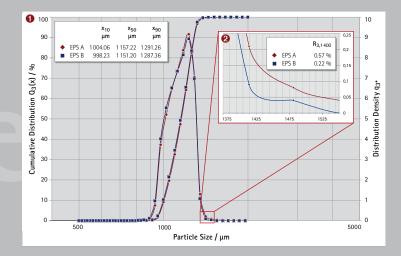
More critical is the ② residue at 1,400 μ m. In order to comply with the specifications, the coarse fraction is not to exceed a value of $R_{3,1400} < 0.5$ %. The contaminated batch EPS A displays a slightly increased amount of coarse material of 0.57 %, indicating a screen break-



age at a very early stage. PICTOS is capable of resolving these fine variations reliably thus inducing a timely change of screen decks.

A powerful computing infrastructure guarantees for fastest measurement

cycles for all nine production lines. Based on our control and evaluation software, three dedicated computers take care of device control and data acquisition, data evaluation and visualisation of measuring results at the master display.



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Development of Innovative Methods for Particulate Systems Characterization Laser Diffraction | Image Analysis | Ultrasonic Extinction | PCCS



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 unique, powerful selection of dispersing units – spear-headed 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 boundless 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 (NIMBUS).

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 should particles predominantly belong to the nanometre range, we have brought the unique Photon Cross-Correlation Spectroscopy (PCCS) to market with Sympatec's NANOPHOX.

By nature, we also keep an eye on the production of disperse systems when developing methods of particle characterization. Hence, you may also have confidence to 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- and 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. 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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