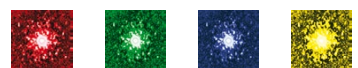


HELOS | RODOS & Co. | Laser Diffraction
Particle Measurement | Laboratory
Size and Distribution | $< 0.1 \mu\text{m}$ to $8,750 \mu\text{m}$



Sympatec develops, manufactures, sells, services and supports a range of best instruments for particle size and shape analysis for laboratory and process applications to customers worldwide. With continuous innovations Sympatec makes a prominent contribution to  laser diffraction,  image analysis,  ultrasonic extinction and  photon cross-correlation spectroscopy.



Particle Measurement

A HELOS is a HELOS is a HELOS ...

RODOS was the Beginning



A Generation of Pioneering Developments

»If a miller – when testing the flour between his thumb and forefinger – does not detect any remaining grains then nearly all particles are finer than 100 microns. This is finished finest flour!«

The *natural human measure* for fineness of particles is at **your fingertips**.

Laser diffraction is the *technical measure* for all particle sizes and its distribution between the coarsest and the finest grain – not just for the coarse. Today, about fifty years after its introduction, the size range of less than 0.1 to about 10,000 microns is mastered by this technology which acquires the particle generated scattered light.

HElium-neon Laser for Optical Spectrometry constitutes the core technology of our sensor solution HELOS.

Laser diffraction

HELOS is the modular classic among laser diffraction sensors. Within each HELOS the primary physical diffraction set-up is realised deploying a parallel laser beam. This yields the purest optical alignment for analysis of extended spacial arrangements of particle collectives which allows for size characterization beyond limiting assumptions and constraints.

Following the premise of Fraunhofer diffraction scattered light of absorbing particles is measured under low angles in forward direction only. In contrast to Mie theory only the Fraunhofer model does not require knowledge of optical properties. Hence, solely Fraunhofer is applicable to mixtures of different materials and shapes.

With laser diffraction the determined particle size always refers to the equivalent diameter of a sphere sharing the same diffraction pattern.

HELOS A-Series (1984-1994)

For the first time, all potentials of the laser diffraction principle are being unfolded uncompromisingly by using the most powerful components in a straightforward instrument set-up. Of modular design, our first HELOS sensor reveals its entire technical superiority in combination with product-adapted dispersing units. Inside the wide and spacious measuring zone interactions between the collective particles and the parallel laser beam are induced in order to analyse particle size. The 180 degree multi-element detector pays perfect attention to the symmetry of the diffraction figures and thus reliably eliminates shape effects.

Dry powder dispersion with ⊕ RODOS is the prominent landmark which proves to be the breakthrough innovation for Sympatec – leading laser diffraction to another dimension. Before RODOS, fine and adhesive dry disperse products needed to be prepared in suspension for analysis with laser diffraction. The enhanced application and extension of Fraunhofer diffraction into the submicron region – at first for suspensions –

became the next spectacular milestone. Until then considered as an unwinnable challenge. However, the modular innovative concept of the A-series was strengthened by the application of reliable Fraunhofer evaluation in the submicron region, even though going beyond basic physical fundamentals. But compared to Mie evaluation, which requires numerous and often undetermined assumptions about optical properties in order to create desired distributions, Fraunhofer delivers valid and robust results for most cases. In this way the submicron region was consequently assigned with real measuring points, supported by related evaluation modes (LD, HRLD).

In case of dependable optical parameters Mie evaluation was requested. But tribute to the limited performance of the computing devices of that time interpolated nodes had to be applied. The performance of the HELOS laser diffraction systems – comprising sensor, interchangeable optics for highest resolution and dispersing units – set new standards for reproducibility and system-to-system comparability in quality control.

⊕ RODOS: Bulk solids in funnel > material at dosing chute > particle-to-particle and particle-to-wall collisions and velocity gradients within the injector at maximum speed. The particle cloud's homogenous glow

in the laser. This is where distinct and evaluable diffraction patterns even for ultra finest arise:

⊕ DRYSUBMICRON.

⊕ Measuring ranges: The complete spectrum of ranges R1 to R8 proves the strong concept of the measuring range segmentation. Reliable insight into all details of the samples to be analysed can only be achieved with selection of the optimum measuring range. [2]

... is an R-Series HELOS

Foundations of Continuous Innovations

HELOS F-Series (1995–2010)

The submicron region was made accessible also for the dry dispersion (⊕ DRYSUBMICRON) with the redesign of the HELOS sensors introducing a fibre optic coupling of the laser beam. Now, the parallel light path was also available for smallest focal distances and the beam diameter could be adapted and optimized. For finest particle collectives highly energetic small and for coarser collectives larger illumination areas were employable. In conjunction with the improved illumination quality, the coarse particle end was extended to 8,750 microns.

The fibre optic technology allowed extremely flexible solutions for the realisation of the sensor geometry. Now light source and detector could be separated and laser diffraction became available for wide angle spray measurements. The modular instrument design was also transferred to on- and in-line installations for process control.

In succession, the variety of product-adapted modular solutions evolved innovatively. The manual lens change for range adaptation was replaced with an automated lens revolver and the best possible resolution was continuously improved to a still unrivalled measuring range covering < 0.1 to 8,750 microns.

Before leaving the manufacture, the unsurpassed quality of the measuring systems is certified with reference materials. Thus, customers and authorities can re-certify and validate the instruments, respectively.

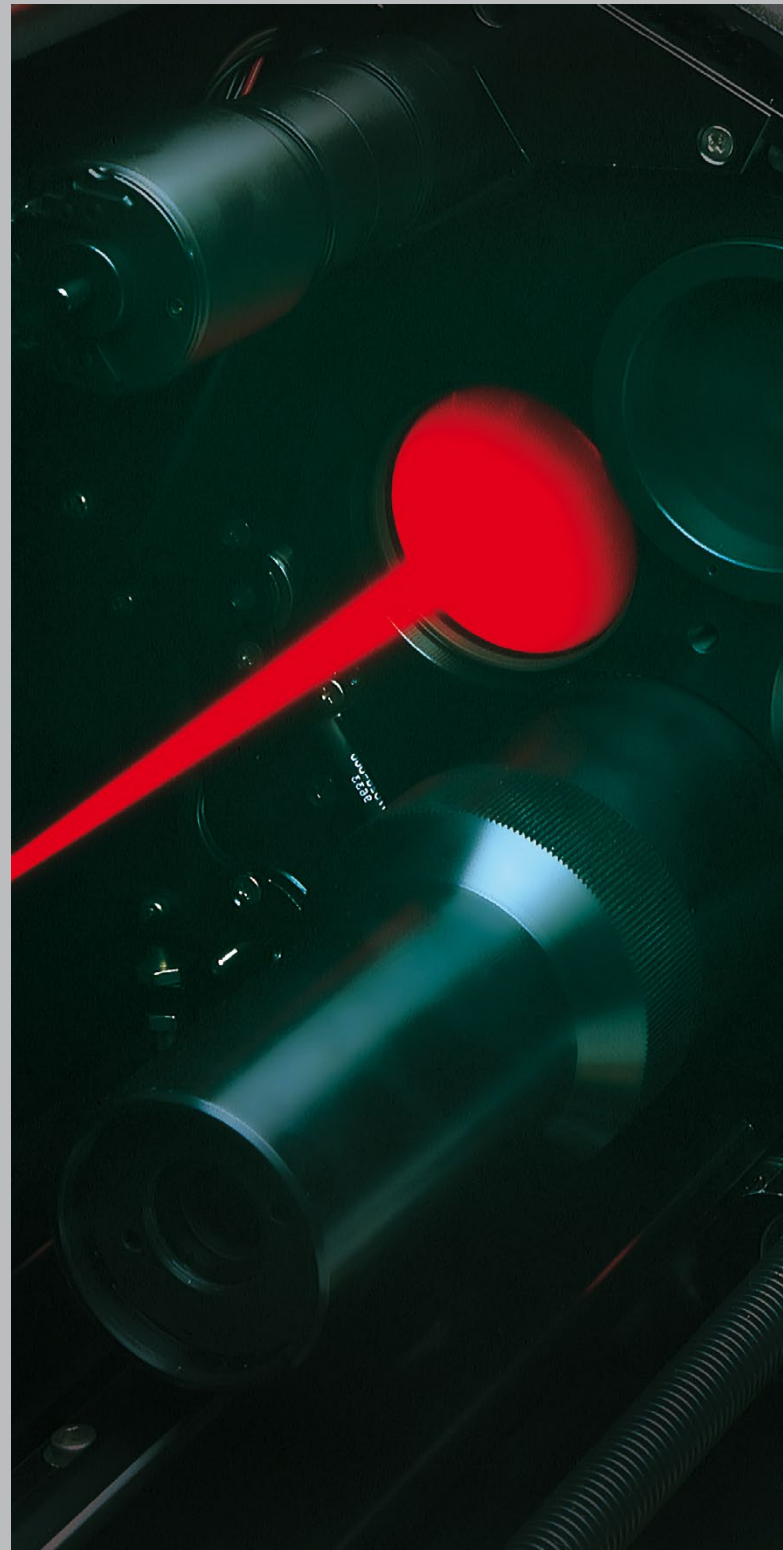
HELOS R-Series (since 2011)

Highest quality standards for resolution and reproducibility are maintained even for wide distributions. With ⊕ 8 measuring ranges, which are mastered by the 31 segments multi-element detector, range combination technology boosts the applicable measuring signal frequency to 248 intensity values.

New benchmarks for precision and accuracy have been achieved considering more efficient evaluation modes (FREE & MIEE), using more stable inversion procedures in combination with better optical models and the inclusion of signal statistics. Now laser diffraction is even closer to absolute standards. Continually, ISO 13320:2009 requirements and limits are considerably outmatched.

Evolution

Over the last 3 decades, the human sensation in the fingertips has been extended by 3 powers of 10 to the fines and to the coarse using laser diffraction technology. In the future, this technology will develop even broader fields of application by employing product-adapted dispersing units. At the same time, it will continue to set new benchmarks for quality and process control of disperse products.



[3] Typical disperse products rarely span over more than two decades. Even for these special products the concept of the multi range combination holds since the introduction of the innovative R-series. Best possible agreement with absolute values is achieved using the further matured

evaluation algorithms of FREE and MIEE. Extremely short analysis times – especially for dry dispersion – are maintained and even for the combination of four measuring ranges the time requirement typically necessary for suspension analysis is kept. Blitz dispersion

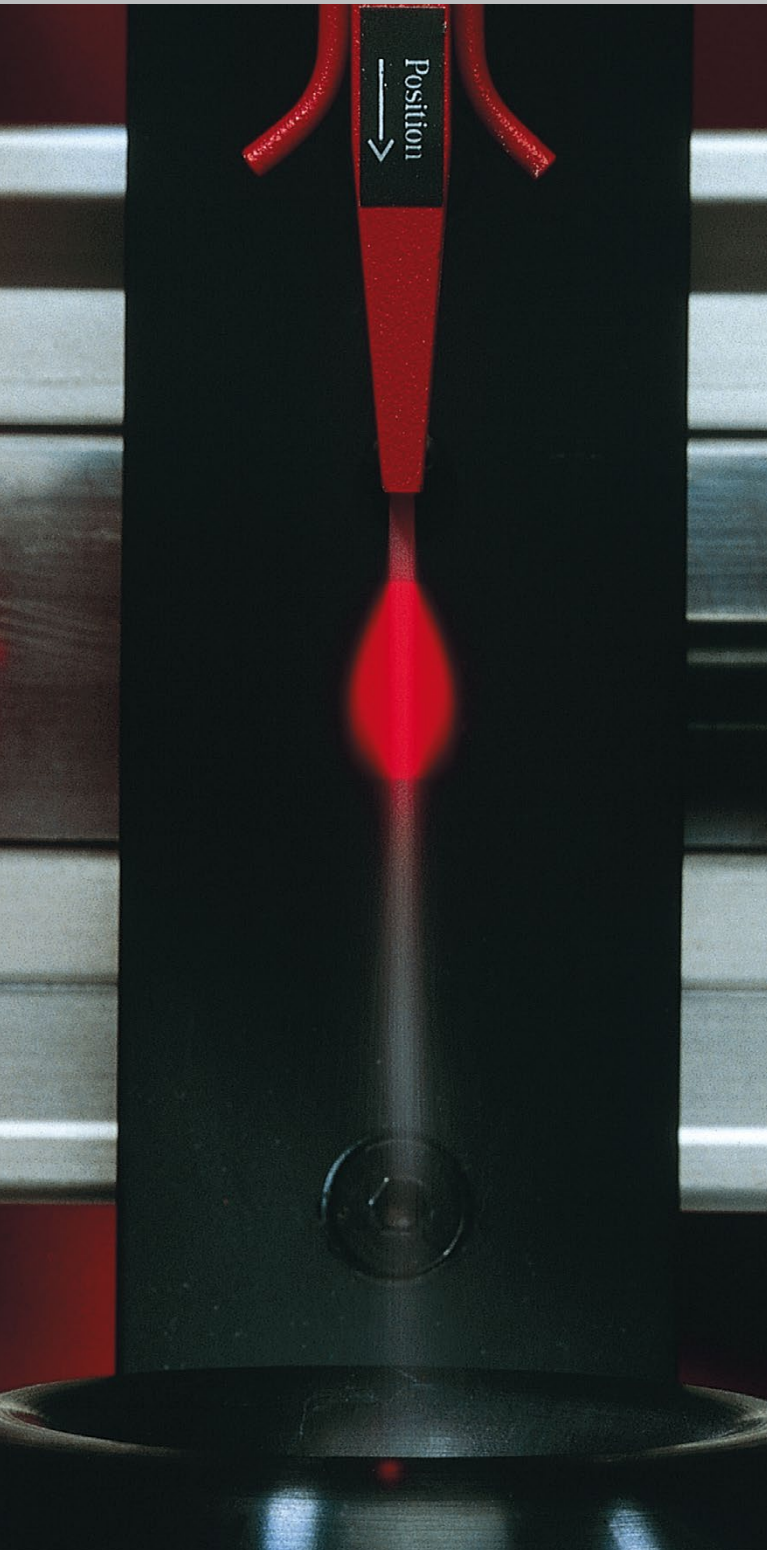
in the dry regime anyhow is faster than human conceivability; thus even doubling the time of combination only extends the pleasant astonishment of simplicity beyond the duration of snapping your fingers into the steady disposability of reliable and best analysis results.



Dry Dispersion

A RODOS is a RODOS is a RODOS ...

Break-through Innovation for Particle Size Analysis



Dispersion

Touching and rubbing between thumb and forefinger are human methods to distribute particles in a very sensitive way in order to feel the original state of fineness. With the tip of the tongue particles from 25 to 100 microns can be sensed when „wet dispersing“ in your mouth. Humans most susceptible sensor is the eye. In the eye liquid man senses even objects just greater than 10 microns. You feel "something annoys your eye".

In order for aerosols to realise a good deposition and take effect in alveoli, bronchia and tracheae of the lung (e.g., inhalants with active pharmaceutical ingredients) they need to be dispersed even further and finer than human senses can detect.

Dry and wet dispersion

In combination with product-adapted dispersing units HELOS turns into a universal measuring instrument. The modular particle measurement systems adapt in an optimum way to the products to be analysed by choosing the right dispersing unit. The quality of dispersion determines significance and reliability of the analysis.

Dry products should be dispersed as powders and wet products as suspensions, emulsions or gels. Sprays and inhalants are best characterised as aerosols.

Dry dispersion

Dry dispersion is the natural challenge and the specific interest of ambitious applications processing powders and granules. ☺ Dry dispersion is the pivotal step as dry products no longer need to be adapted to measuring instruments that only allow for analysis of suspensions. Now, with the help of effective and efficient dispersers like RODOS and GRADIS, the sensor is adapted to the requirements of dry products. Meanwhile, dry dispersion has established as a standard for most dry products and found widespread application with its abundant potential.

Prior to a successful dispersion a skilful dosing has to be accomplished. The complete innovative spectrum of dry dispersion is determined by this combined challenge. Only with well-balanced dosing mechanisms a precise control of most diverse particulate product streams is achieved. In automated applications with the precise vibratory feeder VIBRI even the height of product bed on the chute is controllable.

From a constant feed of powder effective dispersing forces provide single particles in an open aerosol jet, perfectly conditioned for reliable analysis. Dry dispersion successfully applies to agglomerates of smallest particle collectives with highly potential cohesion forces as well as for gentle dispersion of meta-stable agglomerates and coarse particles.

Best dispersion is essential for laser diffraction in order to recognise even the finest details of particle collectives when interacting with the waves of the red and coherent light.

☺ **Dry dispersion:** An essentially reduced time for sample preparation and the remarkably reduced effort for sample splitting – as bigger sample sizes can be processed – are leading to improved statistical relevance and much

shorter analysis times. As no suspensions are used, no effort for reprocessing and disposal is required. The slightly higher cost of investment for dry dispersion spreads over a much longer time of operation of minimum 10, often

up to 25 years. And cost of operation per analysis is typically less than half. Dry dispersion with RODOS and analysis within seconds was and remains unchallenged – from the submicron to the millimetre range.

... are three RODOS – T4 | M | L

RODOS Dry Dispersion



HELOS/KR & RODOS T4

The required wide velocity spectrum is mastered by highly wear resistant dispersing lines.

No other dispersing principle shows similar potentials from the sub-micron range of below 0.1 micron to coarse particles in the millimetre dimension. Of outstanding performance are speed, reproducibility, comparability and first of all highest statistical confidence with large sample volumes. At the same time, the effort for sample preparation is reduced remarkably. Also, even smallest sub-samples in the milligram region can be reliably and completely captured with highest data acquisition speed.

RODOS

The dry dispersing system RODOS has now achieved worldwide recognition as the unrivalled reference. It has pioneered dry measuring tasks for laser diffraction.

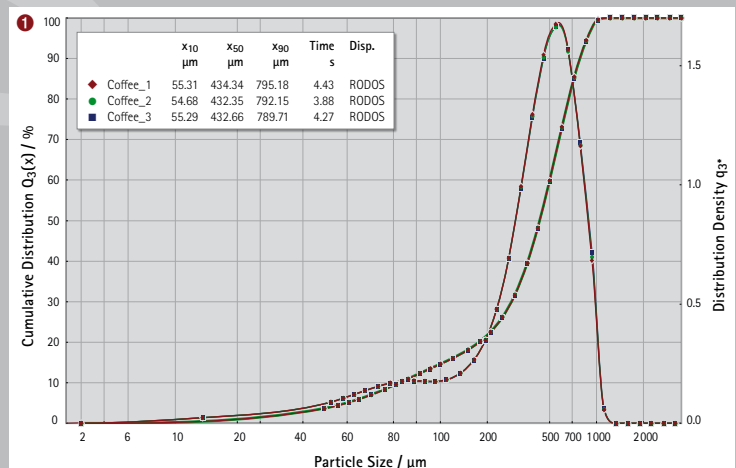
Meantime, varying applications with HELOS have pushed to the sub-micron range. Sample sizes of a few micrograms to a kilogram are analysed in milliseconds to seconds.

HELOS and RODOS

The straightforward design of the measuring principle is predestined for the development of diverse modular combinations. The multitude of modular solutions is indicated by the various images.

HELOS/KR and RODOS

The adaptable dry dispersing unit RODOS can be combined with compact sensor HELOS/BR but also with the longer versions HELOS/KR or HELOS/KR-Vario thus capturing coarse particles up to 3,500 microns.



[5] **1** Coffee: RODOS & HELOS/KR-R7-FREE
The bimodal coffee samples embrace an extreme size range of nearly three decades: running from 1 μm to 1.200 μm; X₅₀-value @ 433 μm.

The gentle separation of the agglomerated fines tail, sticking to the coarse fraction, represents the specific challenge to dry dispersion of ground coffee.

Applying a sample preparation with consistent splitting procedure an analysis time of just four seconds is sufficient for congruent repeatability.



Dry Dispersion

with RODOS and GRADIS

From compact to most flexible

Dry dispersion

HELOS/BR with RODOS

The most compact dry dispersion and laser diffraction set-up is achieved by applying RODOS with HELOS/BR. A particle size spectrum between 0.1 and 875 microns can be determined using up to five separate measuring range modules.

HELOS/BR (OM) with RODOS may be reduced to a single range module R3. This is the established standard configuration for cement covering a particle size range from 0.5 to 175 microns.

Gravitational dispersion

HELOS/KR-Vario with GRADIS

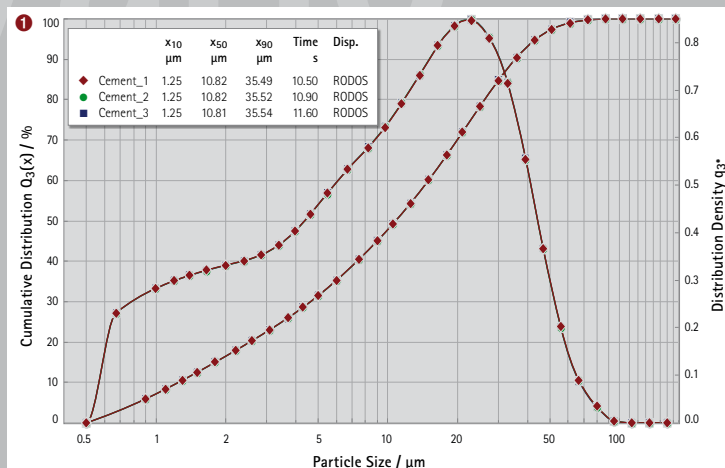
Gravitational dispersing is the method of choice for coarse, disperse, free flowing products and brittle granules providing gentle dispersion from below 10 to nearly 10,000 microns. With GRADIS, gentle dry dispersion is a remarkable option for particle sizes above 2,000 microns especially if particles of the finest fraction are coarser than 10 microns.



HELOS/KR-Vario & GRADIS

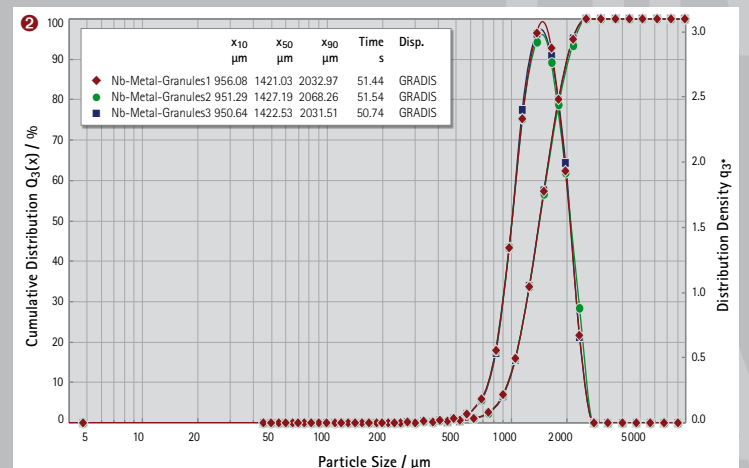
GRADIS is shown here in configuration with HELOS/KR-Vario. This sensor with its variable measuring zone width is

also the best choice for SPRAYER and INHALER as well as for customized and spacious dispersing applications.



1 Cement: bimodal (0.5 µm to 150 µm; x₅₀-value @ 10.82 µm). With measuring times of 11 ± 1 seconds
 2 RODOS & HELOS/BR-R3-FREE shows perfect

repeatability due to reliable dry dispersion – with wear-resistant dispersing line superior for decades. Even for finer cement specialties.



2 Niobium metal granules: GRADIS & HELOS/KR-VARIO-R8-FREE+ The metal granules show a very narrow distribution between 50 and 5,000 microns; x₅₀-value @ 1,420 microns.

With gentle dispersion and analysis time of less than one minute a perfect reproducibility is achieved for particles below 1,500 microns. Due to rapidly decreasing particle numbers from about and above

Aero Dispersion

with SPRAYER and INHALER, VIBRI and ASPIROS

Something's in the Air

Aero dispersion

Particles to be analysed as nebulae, clouds of droplets or spray cones remain assigned to dry dispersion as long as air or gas is the continuous phase around the disperse collectives. HELOS/KR-Vario is the preferred model for application with extended particle clouds and can also directly be applied for customer-specific aerosol or spray generators without dispersing instruments.

SPRAYER & INHALER

Installations for spray analysis round off the large variety of dry dispersers. SPRAYER for pump and propellant gas sprays and INHALER for inhalation devices tap all kinds of applications for most different customer-specific dispersers. Outstanding sensor sensitivity combining elaborate trigger algorithms with fastest data acquisition provide solutions for lowest optical concentrations with time resolutions on the millisecond scale.

The SPRAYER dispersion system for reception and actuation of customer-specific spray bottles (e.g., nasal sprays) is presented with the HELOS/BR. Spatial orientation may be controlled with optional ROTOR. The smooth "glow" of the actuated spray pulse in the laser beam proves the homogenous dispersion.

INHALER fits in the measuring zone of HELOS/BR and HELOS/KR and serves as a dispersing system for the adaptation of customer-specific inhalation devices. Analysis of various metered-dose inhalers (MDIs), dry-powder inhalers (DPIs) or nebulisers are trusted applications.



HELOS/BR & RODOS/L

VIBRI

A constant sample mass-flow is critical for efficient dry dispersion.

The precision controlled vibratory feeder VIBRI constantly feeds the sample – independent of load.

Vibration power and funnel spacing are software-controlled ensuring product-adequate and reproducible sample feeding.

In order to satisfy the demands of your product funnel and chutes are available in different shapes and sizes, specially coated, conductive or non-conductive.

ASPIROS

For analysis of small amounts of precious or toxic material, the micro-dosing device ASPIROS is on hand. ASPIROS is mounted instead of the RODOS funnel.

The encapsulated sample vial is prepared and filled with a few micrograms in a glove box or a fume cabinet. The sealed sample tube is inserted into ASPIROS and the bar code reader identifies the sample. Automatically, the desired dispersion settings are applied, the vial is decapsulated and the sample is aspirated by the RODOS injector for analysis.

With a closed measuring zone product exposure to environment is inhibited.



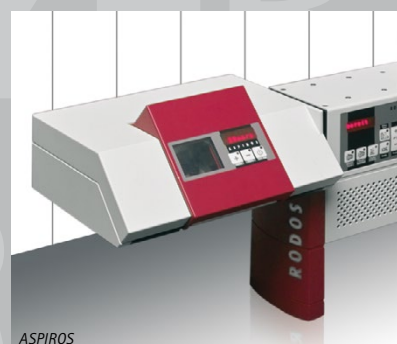
HELOS & SPRAYER



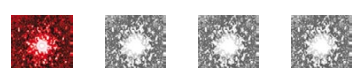
HELOS & INHALER



VIBRI



ASPIROS



[7] 2,000 microns the curves show a slight separation. Longer acquisition times and larger sample volumes could remedy here, if required.

Wet Dispersion

with QUIXEL, SUCELL, CUVETTE and LIQIBACK

Product-specific Diversity



Wet dispersion

Disperse systems in liquid or in wet state should be analysed in product-specific conditions, e.g., as suspensions, emulsions, gels or bubbles. Wet analysis has been the first approach to laser diffraction and is wide spread. This is due to the easier dispersibility of agglomerated particle collectives in liquid with capillary and cavitation forces, which can be further enhanced with surfactants and sonication.

Design options for wet dispersion units are to be found in the variation of flow cuvette depth. It defines the liquid volume in which the particles are suspended when passing the measuring beam. Thus, the suspension film presented in the measuring zone can be formed with respect to



particle concentration. Especially for small sample sizes the flow rate of the circulated suspension can be adjusted.

Sympatec's wet dispersing solutions offer applicable liquid volumes between 6 ml and several litres. At the same time, suspension films can be varied between 0.2 mm and 6 mm. With the wide-spread spectrum of

volumes and film widths sample sizes of a few milligrams to a couple of grams can be properly dispersed and reliably analysed.

With special models, this flexible equipment can be applied with heaviest particles at the coarse range or even may be fit for measurements using aggressive liquid media. If required, we provide heating and cooling options for the systems.

Wet dispersers suitable for nearly all kinds of suspension are available. ☺

QUIXEL

Equipped with a 1 litre suspension basin, centrifugal pump and a quick draining of the sample liquid after analysis, QUIXEL is first choice for the requirements of standard and complex suspension analysis. It comes with flow cuvettes of 2 and 6 mm width and a 2mm 3D-option ensuring a perfectly uniform flow. Internal or external heating facilities and external cooling options provide a wide range of applications.

CUVETTE

For smallest liquid quantities glass cuvettes are available with 6 ml and manual stirrer as well as a 50 ml model with sonication and magnetic stirrer.



☺ If laser diffraction technology requires for the adjustment of optical concentration but further dilution would effect the primary particle size distribution of

the product or if in-line applications are requested, ultrasonic extinction with OPUS or NIMBUS offers further reaching application options in wet media.

Dry and Wet Dispersion

with OASIS – that is RODOS and SUCELL

Symbiotic Dispersers



HELOS/KR & OASIS/L

SUCELL

This wet dispersing system – with a 400 ml stainless steel basin, level sensors, double stirrer unit, peristaltic pump, adjustable ultrasound transducer and media-specific tubing – can be equipped with 0.2 mm,

2 mm and 4 mm flow cuvettes.

The optional small volume adapter (SVA) reduces the liquid volume to 50 ml.

In the L-design, SUCELL can be mounted piggy-back on RODOS/L

transforming to the combined dry and wet disperser OASIS/L. OASIS is also available as M-version.

HELOS/KR with OASIS

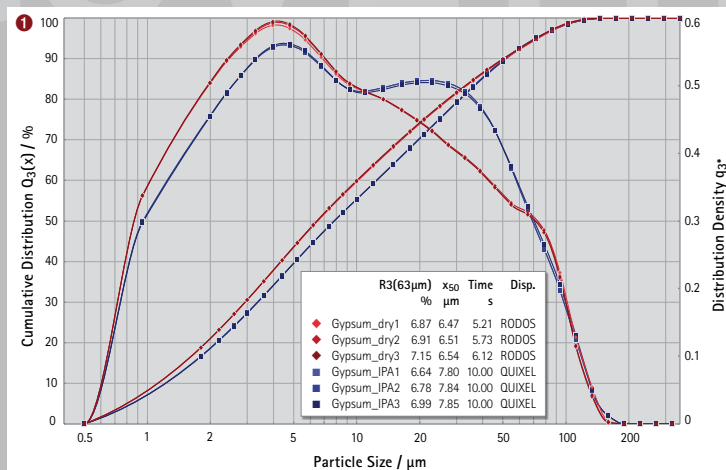
In a compact design OASIS combines the unique dry disperser RODOS with the proven wet dispersing system SUCELL. With this combination there is no need for manual change-over if frequent dry to wet dispersion is required.

For particle sizes up to 8,750 microns the elongated HELIOS/KR laser diffraction sensor with up to eight measuring range modules is available.

In combination with OASIS it provides the most powerful model for concurrent dry and wet dispersion.

LIQIBACK

A small automated device for reprocessing of dispersing liquid is available – equipped with 2x20 litres stainless steel cylinders, impeller pump, torch filters and EPDM tubing.



The bird's view on the front page unveils the cross-structure of the laser diffraction sensor HELIOS/BR with the OASIS (= RODOS & SUCELL) dispersing system installed in the measuring zone.

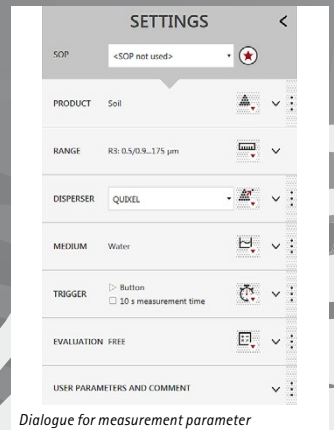
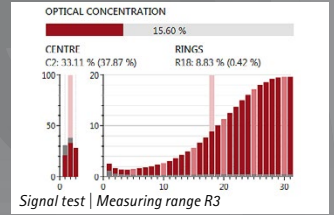
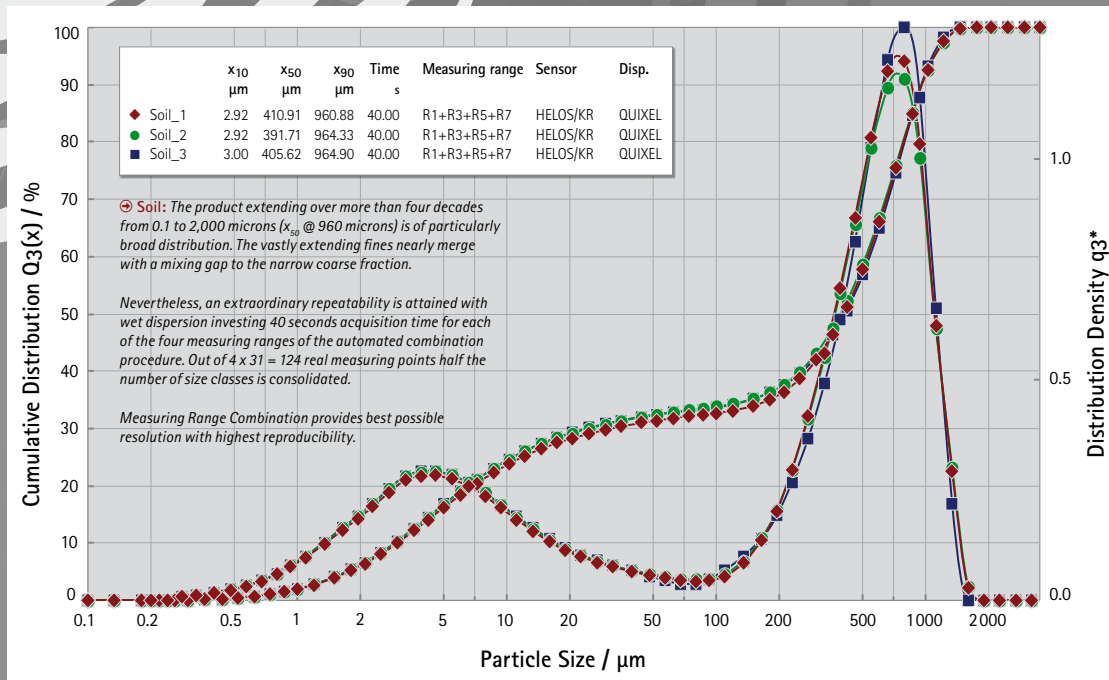


[9] **Gypsum: RODOS/QUIXEL & HELIOS/BR-R4-FREE**
Comparing dry and wet dispersion in mean size range from below 1 μm to 200 μm with $x_{50} = 7.83$ μm (wet) and 6.51 μm (dry) respectively, adequate repeatability for both dispersing methods is displayed. The respec-

tive residual values of R3(63 μm) = 6.80 % (wet) and 6.97 % (dry) show good comparability. The slightly coarser measured gypsum suspension may be caused by encased crystal water thus further enhancing the primacy of the dry dispersion. In addition, dry measurement is twice as

fast and a higher analysis frequency is possible. The significantly less time-consuming sample preparation further increases the time advantage. Additionally, with dry measurement a costly disposal of liquid dispersing media is resolved.

User-friendly Control and Evaluation Platform



PAQXOS succeeds the well proven ⊕ WINDOX application software and incorporates our collective particle sizing expertise into a user-friendly and forward-thinking framework. PAQXOS is realised as a powerful, network-ready 64-bit software for Windows® environments and serves as the joint base for our HELOS laser diffraction systems and all other Sympatec instrumentation.

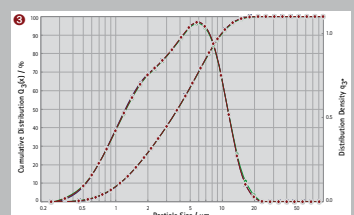
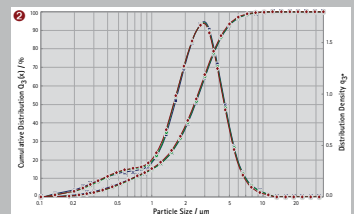
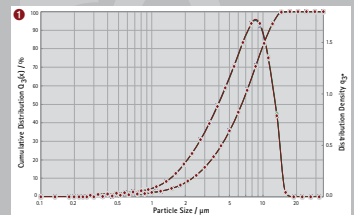
An intelligent step-by-step wizard together with the automated system detection supports the occasional or inexperienced user to get successfully started with particle sizing. PAQXOS autonomously takes care of the fundamental system settings and supports the user with built-in expert knowledge to develop a feasible measurement method and yield meaningful results.

For the experienced or expert user PAQXOS offers full flexibility to implement more demanding applications and to support the development of dependable measurement methods. All measurement parameters are set-up in specific dialogues and stored separately as retrievable templates. Completely defined parameter sets may be managed and deployed as binding ⊕ SOPs. In addition, an integrated scripting environment allows for the programming of elaborate routines to run measurements in an efficient and reproducible manner.

The graphical user interface provides optimized desktop configurations for specific tasks like measuring operations, evaluation or reporting. These default configurations can be flexibly adapted to your individual

needs. The scalable PAQXOS design ensures an optimum representation in Full HD, 4K or 5K resolution while the screen layout might span over multiple displays if desired. Drag and drop simplifies the transformation of measuring results into distribution diagrams, tables or reports and also eases method transfer from existing measurements. Result presentations and paper reports can be chosen from a variety of predefined and fully customisable formats.

PAQXOS 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 to prevent manipulation of records.



⊕ Existing WINDOX-based methods and measurement records are easily migrated to PAQXOS.
⊕ When using SOPs | Standard Operation Procedures the individual modification of measurement settings is disabled and locked.

⊕ NdFeB Challenges dry dispersion ⊕ RODOS & HELOS/BR-R1-FREE with extreme magnetic cohesion forces. Results show great repeatability. Mono-modal distribution between 0.1 microns and 20 microns, x_{90} -value @ 6.45 microns. Ultra-short measuring times: 0.65 ± 0.15 seconds.

⊕ Lactose Sticky and ultra-fine with a higher fines fraction (0.1 – 15 µm @ $x_{10} = 2.43$ µm). Perfect dispersion in the free aerosol-jet with ⊕ RODOS & HELOS/BR-R1-FREE. Repeatable measurements in 0.25 ± 0.15 seconds.

⊕ Fine Dust of Silica Bimodal and extremely fine (0.25 µm to 25 µm; x_{50} -value @ 3.81 µm). High demand on dry dispersion with ⊕ RODOS & HELOS/BR-R2-MIEE (1.457-0). Measuring times < 2 seconds. Remaining variations reduced or eliminated with increased sample size.

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."

Sensing with the tip of fingers is no longer sufficient to comply with the diversity of products for quality 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 – spear-headed by RODOS – offer you a premium performance. Our laser diffraction instruments allow for a significant extension of your particle knowledge concerning size and size distribution.

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 (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.

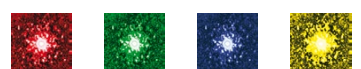
And in case particles predominantly belong to the nanometre range, we have brought the unique photon cross-correlation spectroscopy (PCCS) to market with Sympatec's NANOPHOX.

By nature, we also keep an eye on the production of disperse systems when developing methods of particle 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

Several Thousand Installations

At Particle Professionals Worldwide



Sales | Service and Partner Network



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