QICPIC | **RODOS & Co.** | Dynamic Image Analysis Particle Measurement | Laboratory Size and Shape | < 1 μm to 34,000 μm





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 D laser diffraction, D image analysis, D ultrasonic extinction and D photon cross-correlation spectroscopy.



QICPIC | Dynamic Image Analysis

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 \bigcirc 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 @

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.



● A standard deviation < 1 % for a number distribution $Q_0(x)$ requires > 10,000 particles per size class. For a volume related $Q_3(x)$ distribution the **number of** particles required depends on the size distribution itself. Here, for a standard deviation < 1 % more than 1,000,000 particles are necessary.

[2



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 in the nanosecond range 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 μ m to 34 mm can be 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 productspecific 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, convexity and roundness 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 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

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 1.8 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 0.55 μ m to 2,000 μ m. For dry samples the well-known aerosol free jet of RODOS is ready for use.

VIBRI

With the controllable precision vibratory feeder VIBRI the O 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.

Application QICPIC & RODOS

High Resolution Size and Shape Analysis of Fine Powders Efficient Dry Dispersion | Statistically Relevant and Quick Results

Metal powders | 3D printing

Additive manufacturing places the highest requirements regarding a consistent high quality of the applied metal powders. Spherical particles of high purity together with an optimized particle size distribution guarantee for reliable printing results and an immaculate, homogenous material structure. Dynamic image analysis yields meaningful measuring results to ensure optimum powder qualities from batch to batch.

QICPIC & RODOS | M5

The injection disperser RODOS provides reliable particle singling. No special preparation is required to supply a small amount of sample material with the dosing chute VIBRI for dispersion in a free aerosol jet. With the optical module M5 QICPIC covers a wide dynamic measuring range from 1.8 µm to 3,750 µm. Up to 1 million particles are captured in less than one minute. The **①** binary live image of the sensor visualises the complete dispersion of the metal powders in real time. The **②** size distributions of two powder batches are presented as volume related cumulative distributions (Ω_3) based on the diameter of the equivalent circle EQPC. The metal powder in batch A is within the expected size range of 15 to 53 µm. However, batch B has a significantly finer distribution and therefore tends to agglomerate, which reduces flowability of the powder.

Taking a look at the O PAQXOS software particle gallery, which provides an illustrative, qualitative impression of individual particles, reveals that some particles of batch A contain O satellites. The presentation of the O aspect ratio exhibits that in the fine range of batch B the particles are stronger elongated. Not until around 30 µm are the values of the aspect ratio about the same for both batches.

The ③ sphericity is the ratio of the surface area of a sphere with equal volume and the surface area of the particle. In this example the surface areas of finer particles are smoother than the surface area of larger particles in both batches.



Binary image of dispersed particles

0





Less porous, spherical particles improve the flow properties of metal powders and optimise the powder fusion. The printing process is positively influenced and leads to components with high density.



[5] ● EQPC is the diameter of a circle that has the same area as the projection area of the particle. Satellites are smaller particles adhering to bigger particles during the solidification process. The fewer satellites are present, the better the printing result.

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 μ m to 34,000 μ m length and 1.8 μ m to 5,000 μ m 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.

Efficient industrial vacuum cleaners are available for the extraction. If sample recollection is required, cyclones are a viable option.

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 **8** 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 Q₂ 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 $S_{ro} \approx 0.69$. In the presentation of the aspect ratio related to the size it is shown that smaller particles tend to be more compact and round than larger ones.









 Triggered with the optical concentration VIBRI generates a continuous, reproducible particle feed during the complete measuring period. The Aspect Ratio is defined by the ratio of FMin to FMax. Values range between 0 (strongly elongated) and 1 (compact and round). Regarding the volume-based **Q₃ distribution** it is recommended to use the EQPC evaluation for compact, irregular particles. EQPC yields the best approximation of the actual particle volumes.





Wet Dispersion

with SUCELL | 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.



SUCELL

The size and shape of particles in suspensions or emulsions in a range from 0.55 μ m to 2,000 μ m are reliably characterized with SUCELL.

The wet dispersion system with 500 ml stainless steel basin, level sensors, double stirrer unit, peristaltic pump and variable tubing is equipped with a precisely controllable ultrasonic transducer. To ensure optimum focusing on particles in different size ranges, flow-through cells with adapted layer thicknesses from 0.3 mm to 4 mm are available. With the small volume adapter SVA, the nominal volume can be reduced to 50 ml if required. SUCELL works as a stand-alone device or is upgradable to an integrated dry and wet dispersion system in combination with RODOS/L.

LIXELL & LIQXI

Due to its open and modular design LIXELL represents the most versatile wet dispersion system over a particle size range of 0.55 µm to 2,000 µm. Numerous applications can be realised with a variety of Θ cuvettes and application kits starting at a sample volume of 20 ml. True-tomethod 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.

MULTISAMPLER w



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 11 μ m to more than 10,000 μ m. 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 SUCELL 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.

 ⊕ LIQIBACK A reprocessing unit for the recycling of dispersion liquids is available as an accessory. LIQIBACK can be integrated into the MIXCEL circuit and allows for
 a multiple, sustainable use of dispersion liquids. The cleaning is performed in a parallel pumping circuit with filter cartridges, i.e. two stages of 1 µm and 0.45 µm. ⊕ Cuvettes are available with depths of 30 µm to 4 mm. Optional accessories include different pumps, stirrers, syringes, adapters, tubing and stopcocks. [8]

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: 1 intact, juice filled cells are found in batch A and Ø disintegrated cells in batch B.

The ⁽³⁾ particle size distribution now quantifies the differences in quality of the supplied raw juices. Batch A shows a mean particle size of $x_{ro} \approx 9,785 \,\mu m$ 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 *⊕* sphericity, a measure of closeness to a perfect sphere. The smaller the value for the sphericity the more irregular a



[9]
 The mean Feret diameter is determined as the average value out of all possible orientations (0° to 180°) of the particle projection. Transparent juice cells thus

are interpreted as solid particles. Whereas the EQPC would reduce the particle size by the transparent parts.

area is related to the real particle perimeter. The result is a value between 0 and 1.

0.1

0.2



Sphericity

0.5

0.6



0.7

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

4 100

Cumulative Distribution Q₃(S) / %

90

80

70

60

50

40

30

20

10

the particles than the cumulative curve of batch A with its undamaged,









1



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



TWISTER | in-line Sampling

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 **1** size distributions of both batches display the narrow grain range of about 900 µm to 1,500 µm, 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 μ m and $x_{50,EPS} \approx 1,151 \ \mu m$.

More critical is the **2** residue at 1,400 µm. In order to comply with the specifications, the coarse fraction is not to exceed a value of $R_{31400} < 0.5$ %. 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.



heat insulation materials for construction and shock absorbing packing material.

compact EPS raw beads with steam and moulded to solid masses and final shapes depending on the final application. The beads are growing to a multiple of its initial volume throughout the (repeated) swelling process. Typical applications include e.g., noise and

Pulverhaus

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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 – 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 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 (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 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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