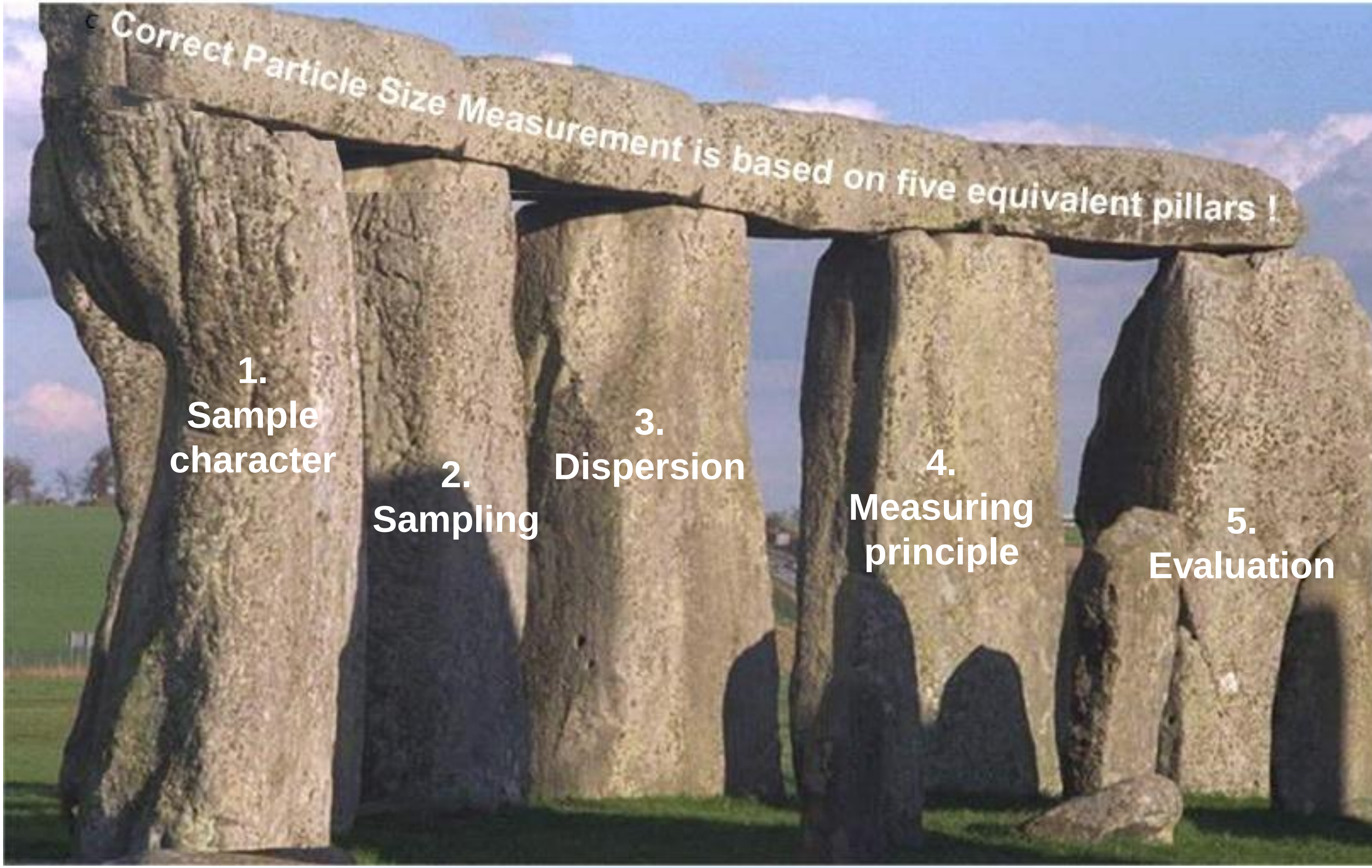
A course on image analysis of particles

Based on my experience at/with Sympatec GmbH
(employing excerpts of Sympatec GmbH publications)
http://www.sympatec.com/EN/ImageAnalysis/ImageAnalysis.html

Particle size measurement of granules and important basic precautions to be taken

„Which measuring instrument does measure correctly?“



1. Sample character

★ **Samples from dry processes have to be measured in a dry way**

drydispersion that is not based on single particle detection is able to analize even large sample sizes in short measuring time

★ **Samples from wet processes have to be measured in wet dispersion**

ISO 13320-1 : 1999(E)

“The minimum volume of sample, required for repeatable measurement, increases as the width of the size distribution becomes greater in order to allow a sufficient number of large particles to be present. Accordingly, the volume of the dispersion fluid required to suspend these samples also increases if the limits of optical concentration are to be observed.

For example, for a sample with particles in the approximate size range of 2 µm to 200 µm, a sample volume of at least 0,3 ml is needed. This will require at least 500 mf of suspension fluid for its dispersion. Also, the measurement time or the number of detector readings within one measurement should be sufficient to reach a reasonable precision. Appropriate measurement conditions should be established experimentally, in relation to the desired precision. „

2. Sampling

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- ★ Representative cross section of the total
- ★ Statistical relvant size

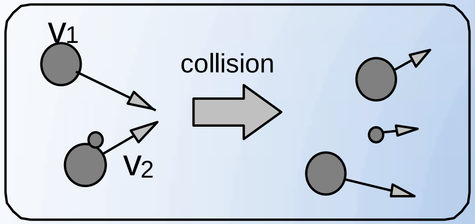
  For a representative sample and a good reliability of the statistical base, a large number of particles has to be selected in an applicable short time:

  1 % precision requires 10.000 particles per size class

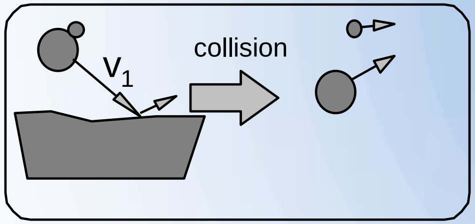
- ★ Split into representative test portions
- ★ Also the test portions have to be of statistical relevant size

3. Dispersing

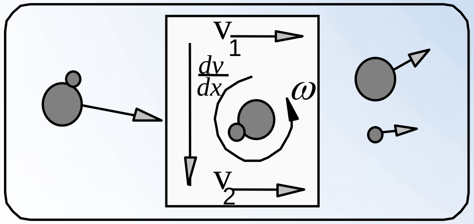
- ★ Complete but without communition / dissolving
- ★ Stable atleast during the measuring time, no recrystallisation / reagglomeration



Particle to particle collision



Particle to wall collision



Velocity gradients caused by shear stress

Perfectly dispersed aerosols tend to reagglomerate during subsequent transport through hoses and cuvettes.

Dispersion based on surpressure often causes instability. As in case of using the surpressure of the extraction unit to provide the dispersing force, the pollution of the filter changes the available surpressure permanently.

4. Measuring principle (available methods)

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Method	Priciple	Restriction
Sieving	Passing through a mesh	Only smallest spherical diameter is detected, no shape information
Sedimentation	Separation by gravity	Flakes are „parachuting“ -> too small sperical diameter, no shape information
Centrifugation	Sep. by enforced gravity	Rotation start problems s.Sedimentation
Coulter-Counter	Electr. Field disturbance	Low concentration = limited statistics Small dynamic size range only
Time of flight	Light beam disturbance	Low concentration = limited statistics
Diffraction	Diffraction pattern	Equivalent spherical diameter only, no shape information
Ultrasonic extinction	Attenuation of ultrasonic frequencies	Only spherical diameter based on calibration, no shape information
Microscopy	Static image analysis	Low concentration = limited statistics Orientation -> Limited form information
Image Analysis	Dynamic image analysis	Depth of sharpness

4. Measuring principle

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★ Preferential based on basic physical principles

★ No mix of methods, no hybrid instrument

↳ *pretended universality compromises accuracy*

★ Adequate dynamic measuring range

↳ *peripheral zones often are critical areas*

★ Statistical relevant numbers of measured effects

↳ *the charme of exact measured single particles looses qualification very fast if statistical relevance is missing.*

5. Evaluation

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- ★ **Physical basic principles can always be evaluated without input or specification of model assumptions**

  ↳ ***specification of mono or muptiple modality is nothing but a cruch to enable evaluation by highly instable evaluation modes***

- ★ **Physical basic principles cannot be „calibrated“ just by using software without (wanted) backlash to results.**

  ↳ ***Set up errors e.g.focal errors have to be corrected in the instrument, not by software***

ISO 13320-1 : 1999(E) *

6.5.1 Calibration

„Laser diffraction systems are based on first principles, though with idealized properties of the particles (cf. Annex A) Thus, calibration in the strict sense is not required. However, it is still necessary and desirable to confirm the correct operation of the instrument by a validation procedure (see 6.5.2).“

Image analysis instrument QICPIC

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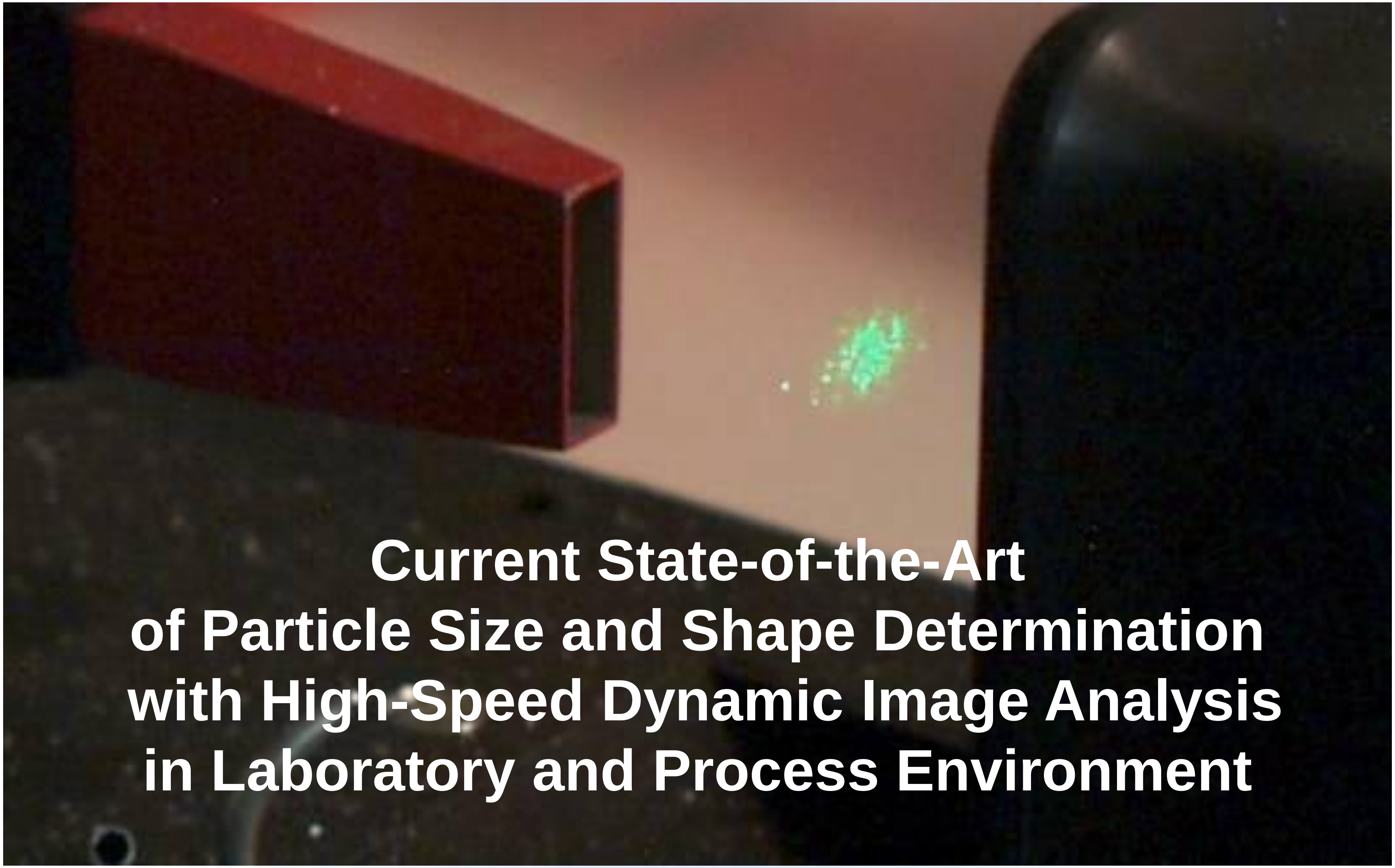

Introduction

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What do we want?

State-of-the-Art Technology for :

Particle Size and Shape Analysis

results as close as possible to reality

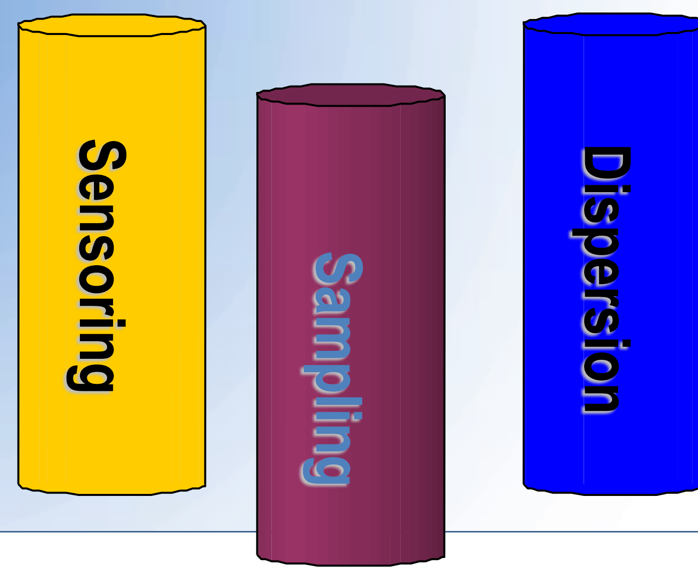
analysis as fast as possible

for all kinds of disperse matters, i.e.

- powders
- suspensions
- emulsions

What do we need?

1. representative amount of sample
2. complete, product adapted sample preparation, i.e. dispersion
3. dynamic Image Analysis Sensor



Solution

Measurement of statistically relevant Samples

Combination of Image Analysis Sensor with

effective, product adapted Disperser

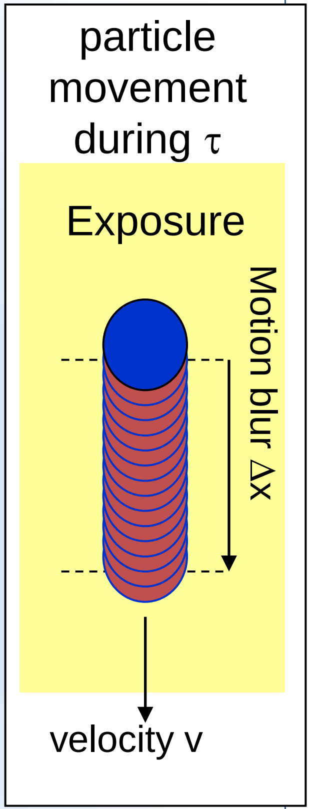
avoiding overlapping particles

no "software dispersion" requesting time consuming data manipulation

Analysis of very large particle numbers ($>10^6$)

improving the *statistical relevance* of the results

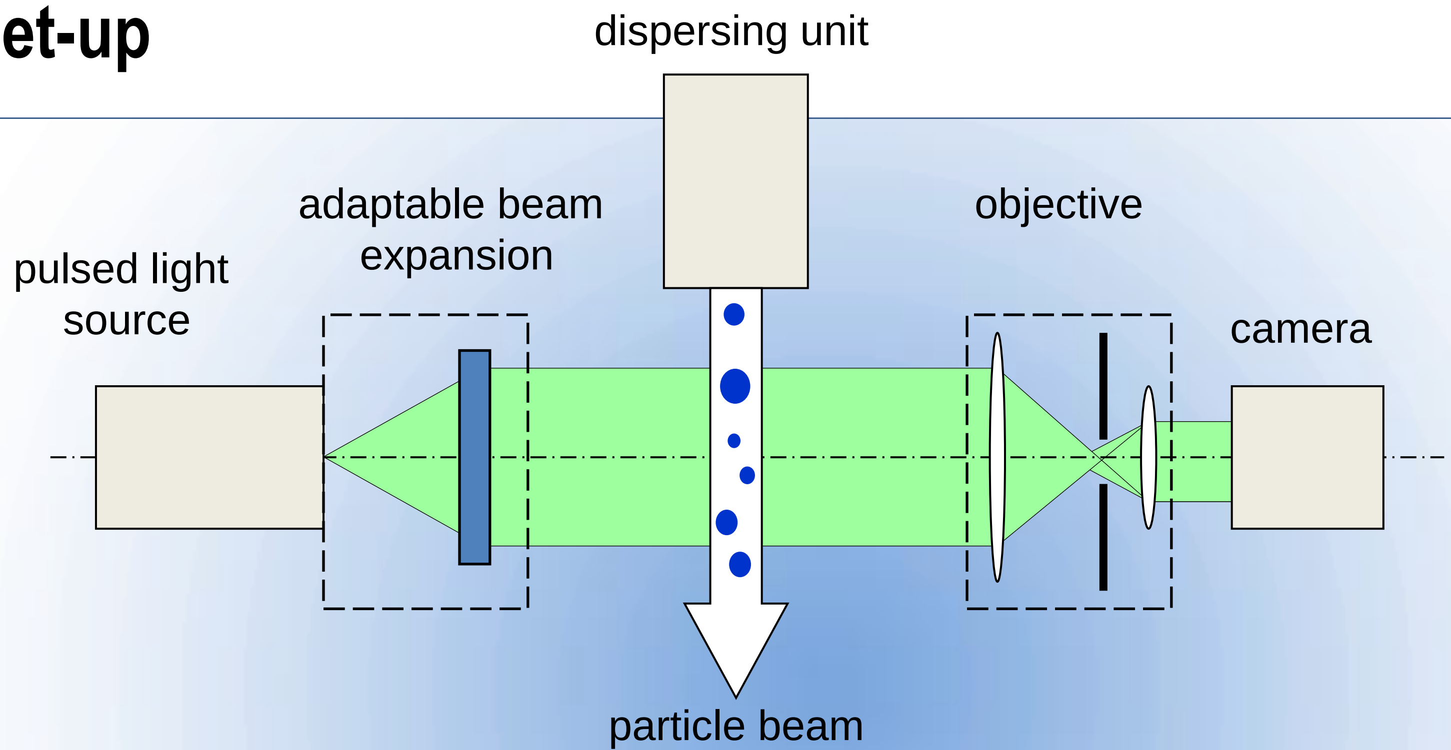
to values with σ_{max} < 1% (comparable to laser diffraction)



Challenges

- ☆ Light source → *< 10 ns* exposure time (dry dispersion, e.g. with RODOS at 100 m/s)
- ☆ Camera → acquisition of *10^4 to 10^5 images* in less than a minute
- ☆ Optics → imaging at *highest contrast* (reduced computing time)
- ☆ Evaluation → fast processing of *GigaByte of data*

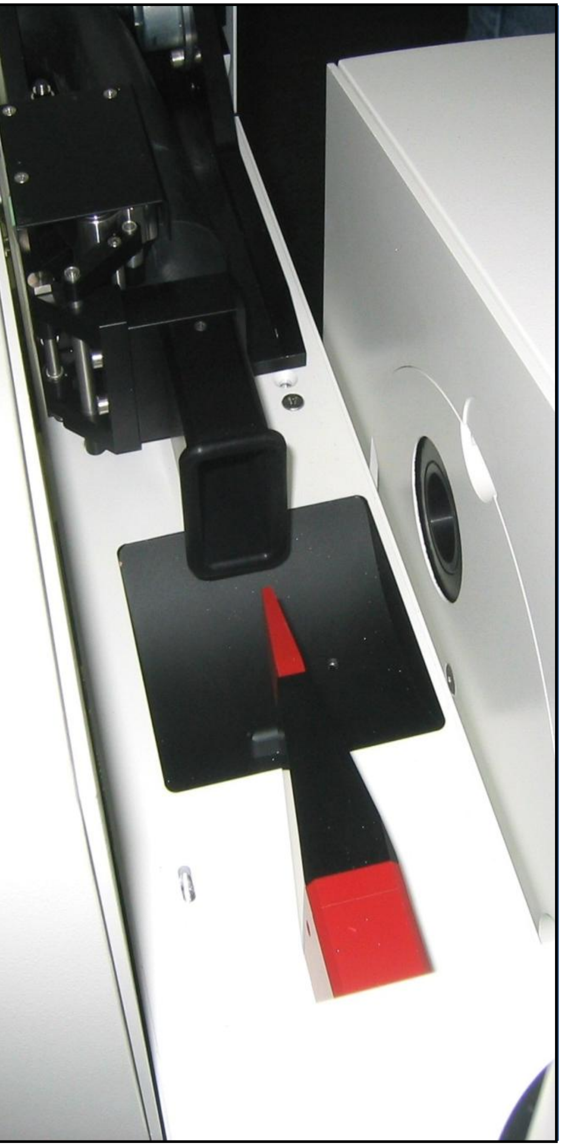
Set-up



- ★ pulsed light source
  - ☆ pulse duration: < 1 ns
  - ☆ energy: ≈ 0,15 nJ / pulse
  - ☆ colour: visible (≈ 532 nm)
  - ☆ pulse rate: 0 - 500 Hz, selectable
- ★ beam expansion unit
  - ☆ 35 mm Ø, 16 mm Ø, 7mm Ø
- ★ objectives (up to 6 on a carousel)
  - ☆ 2:1, 1:1, 1:2, …
- ★ camera
  - ☆ CMOS, 1024 x 1204 Pixel,
  - ☆ 10 µm x 10 µm Pixel
  - ☆ up to **500 fps**

Dry Dispersion: QICPIC & RODOS

for fine dry particles



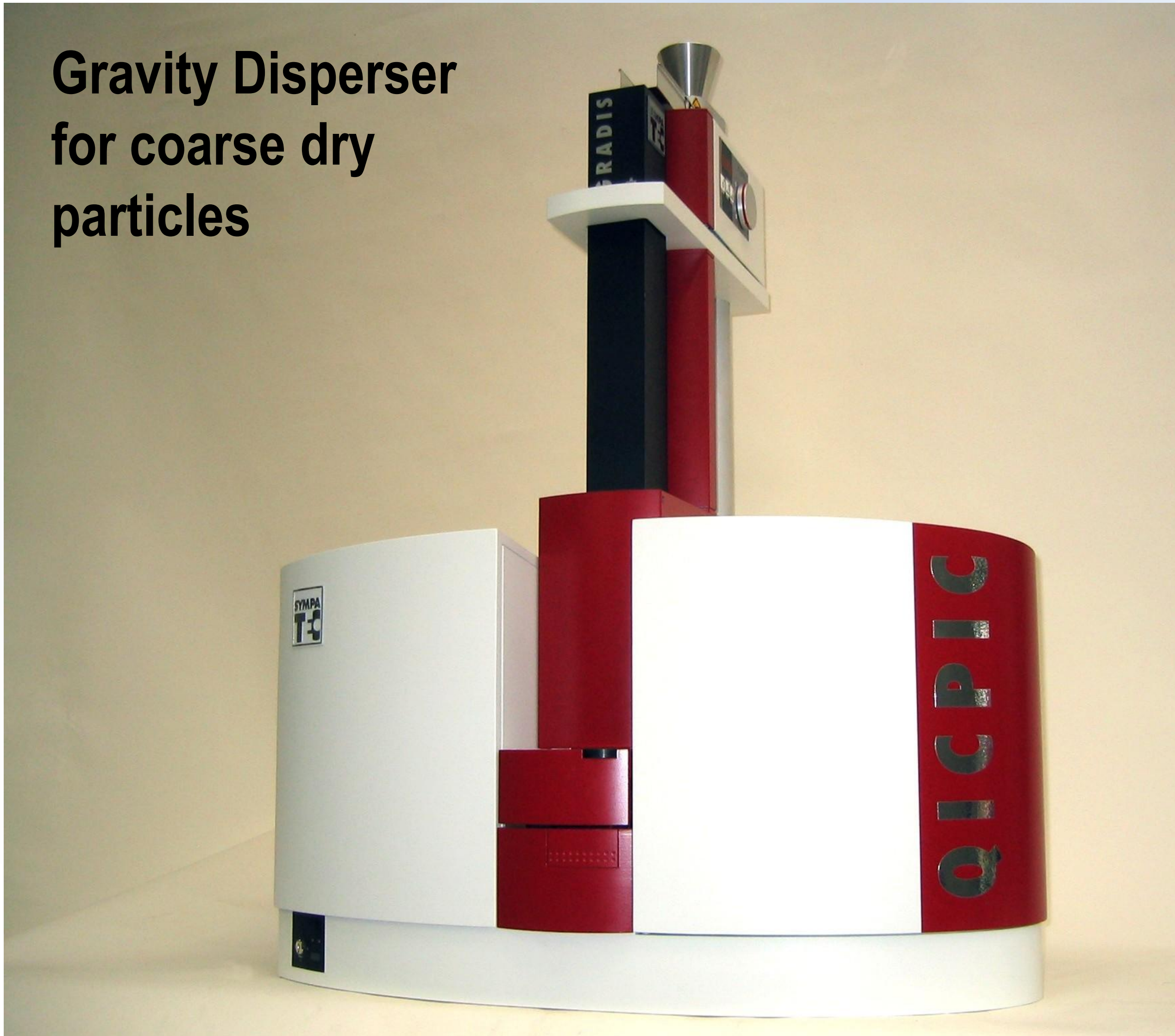
QICIPIC & RODOS: Operation

Measurement with QICPIC and Dry Disperser RODOS

1. *filling* of sample into hopper of the feeder
2. *constant feeding* into funnel of RODOS dry powder disperser
3. *imaging* of particles dispersed in aerosol cloud with 500 flashes/s of 1ns duration
4. *life display* of imaged particles on monitor

QICPIC & GRADIS

Gravity Disperser for coarse dry particles



Investigation of Slow Particles: 3D shape

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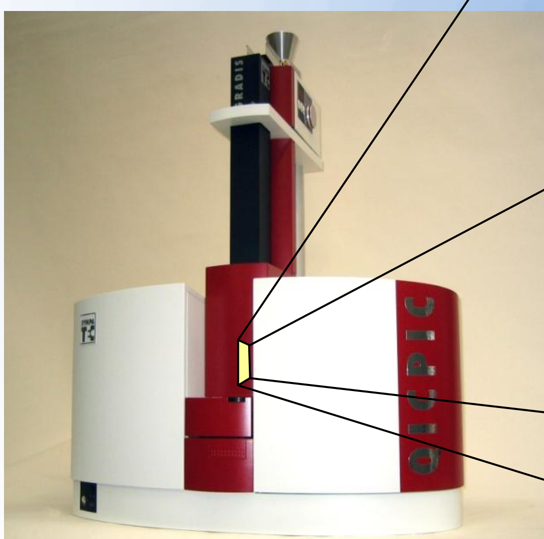
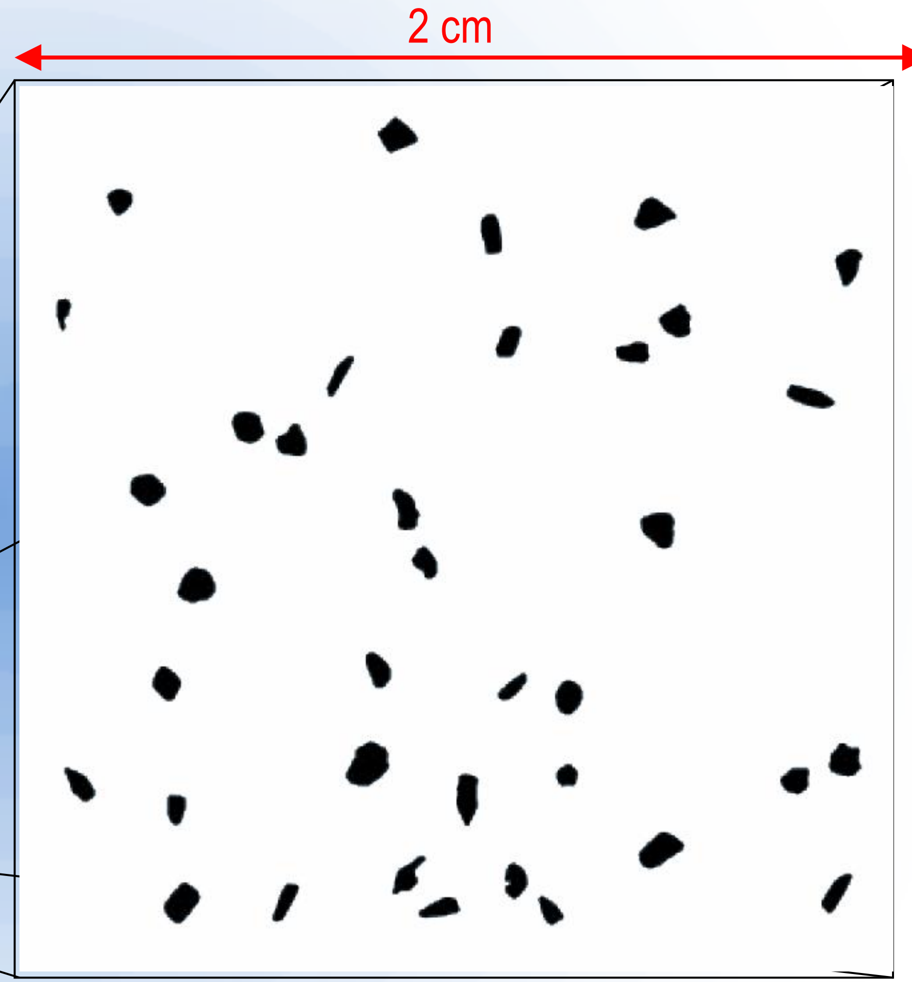
- ☆ due QICPIC's high frame rate of 500 fps, slowly moving particles are *imaged more than once*
- ☆ this feature can be used to study the 3D shape of particles under different orientations



QICPIC & gravity disperser GRADIS



sample: SiC–P16, measured at 500 fps

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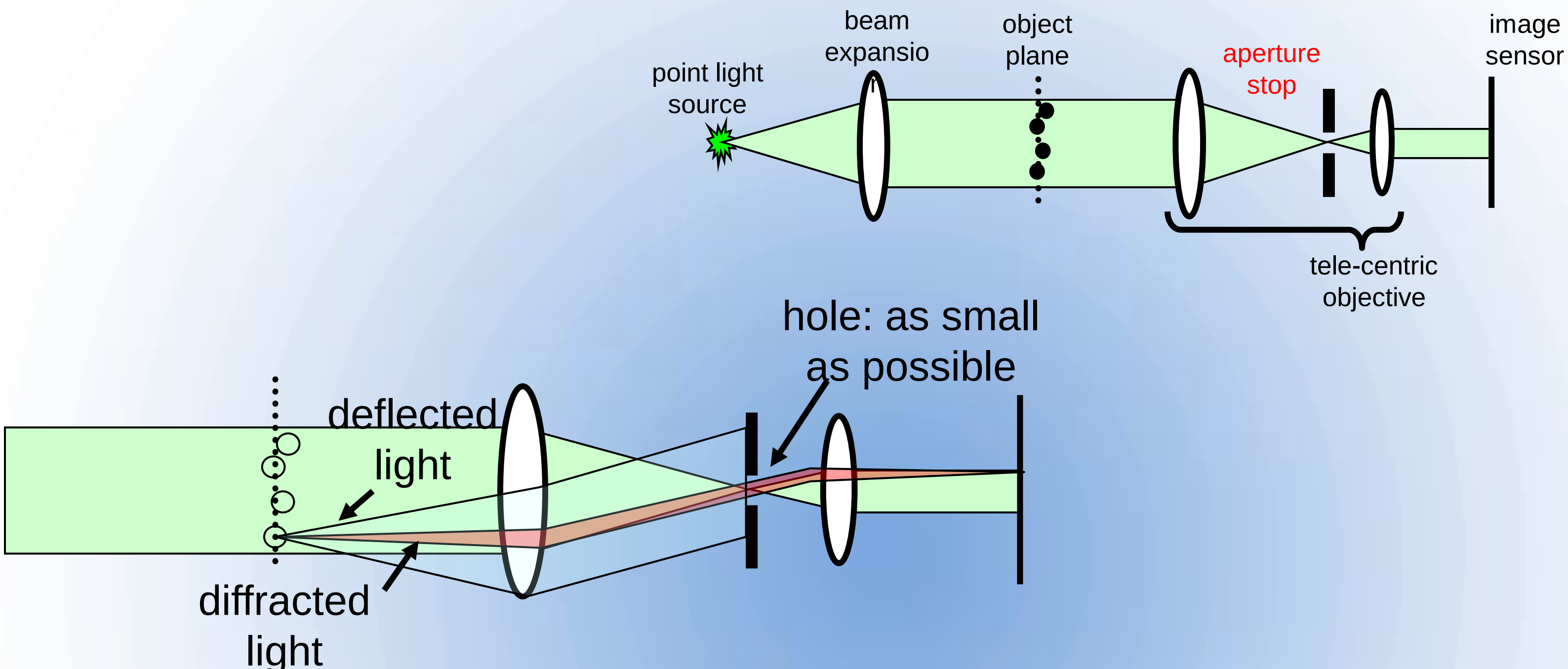
Wet Dispersion

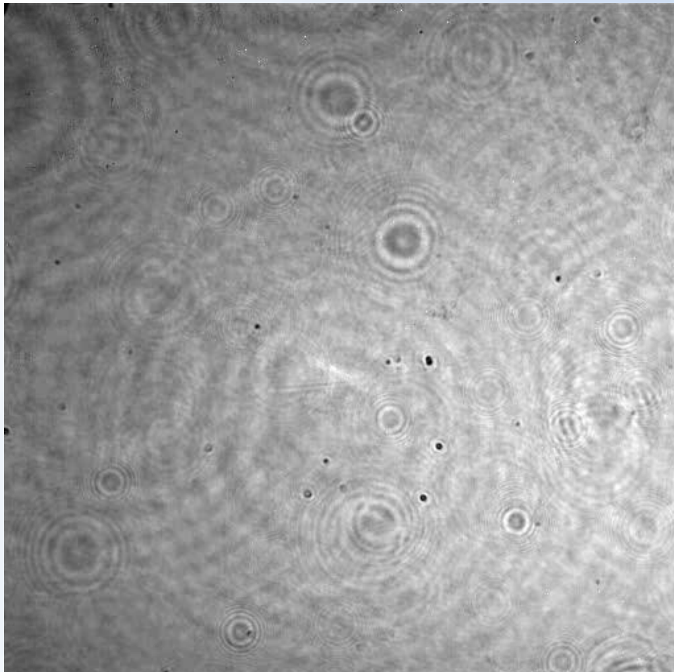
Dry and wet dispersion
with QICPIC & OASIS

The Optical Set-up

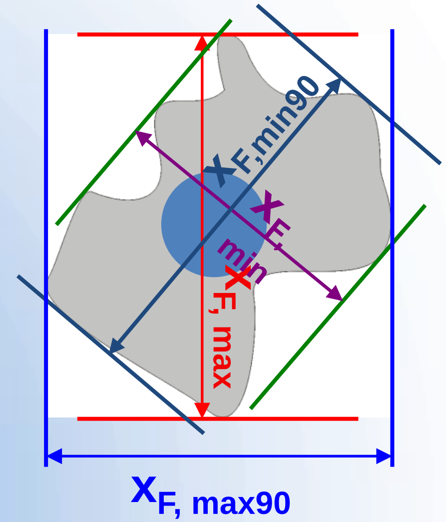


- ★ a *background illumination* with *parallel light* is used in combination with an objective with aperture stop
- ★ the aperture stop eliminates
  - ☆ *stray light*
  - ☆ *deflected light*
  - ☆ *diffracted light* scattered to large angles

Application with transparent particles



unprocessed, analogue “life” image of particles

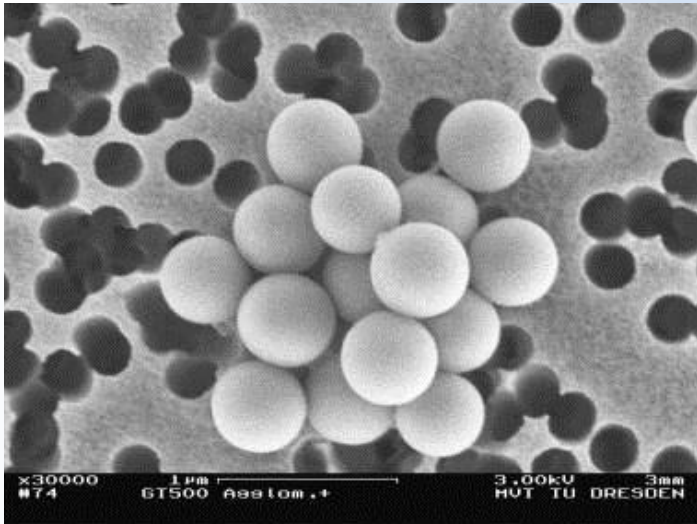


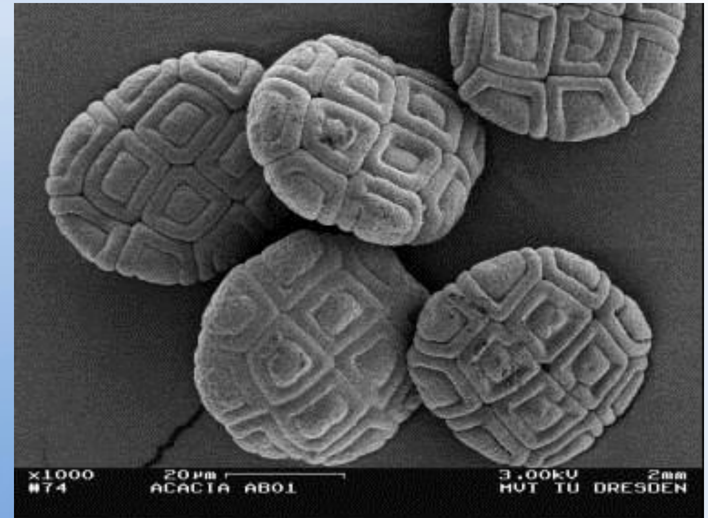
Feret diameter for particles with „hole“

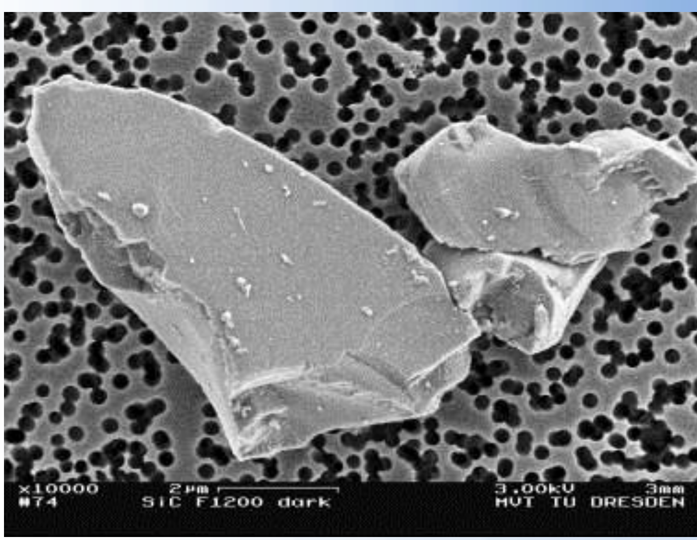
- ☆ all particles are images in black colour, as *deflected light* cannot reach the camera
- ☆ the *centre is illuminated*, as the light beams here propagate nearly parallel to the optical axis

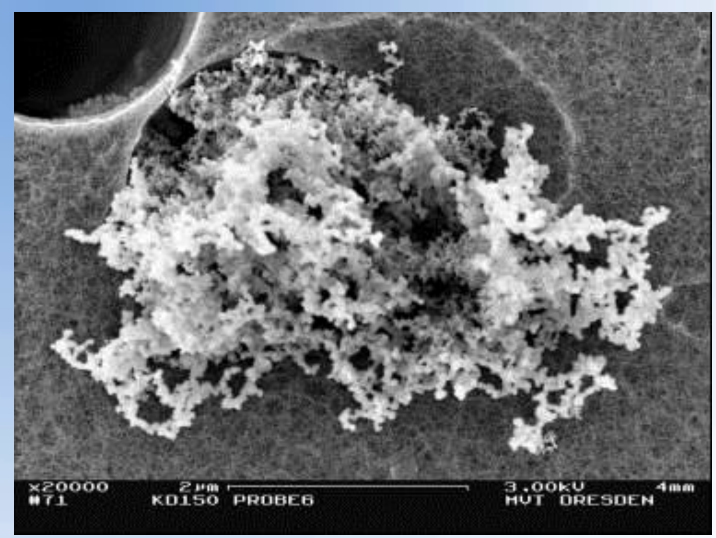
➩ highly transparent particles can be acquired at *high contrast*

Variaty of shapes, how to describe?









Diameter Definitions (1)

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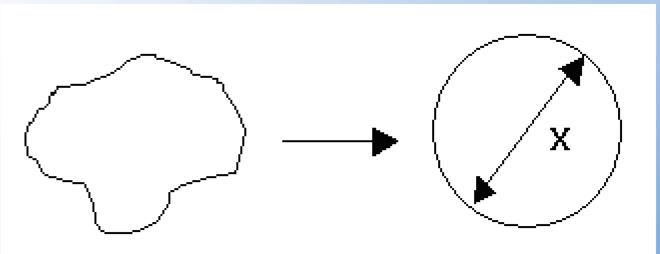
★ Diameters Derived from the Equivalent Circle



Diameter of a Circle of Equal Projection Area (EQPC)

This is the diameter of a circle that has the same area as the projection area of the particle.

Diameter of a Circle of Equal Perimeter (PED)

This is the diameter of a circle that has the same perimeter as the particle image.

Diameter Definitions (2)

★ Feret Diameter

This is not a diameter in its actual sense but the common basis of a group of diameters derived from the distance of two tangents to the contour of the particle in a well defined orientation. In simpler words, the method corresponds to the measurement by a slide gauge (slide gauge principle).

Diameter Definitions (3)

Feret Diameter, Maximum (FERET_MAX)

Maximal Feret diameter after consideration of all possible orientations (0°...180°). Internally, the Feret diameters for as many angles as possible are calculated, and their maximum is selected. If a particle has an irregular shape, the Feret diameter usually varies much more than with regularly shaped particles. The maximum can therefore be significantly larger than the diameter of the equivalent circle.

Feret Diameter, Minimum (FERET_MIN)

Minimal Feret diameter after consideration of all possible orientations (0°...180°). Internally, the Feret diameters for a sufficient number of angles are calculated, and their minimum is selected. If a particle has an irregular shape, the Feret diameter usually varies much more than with regularly shaped particles. The minimum can therefore be significantly smaller than the diameter of the equivalent circle.

Feret Diameter, Mean Value (FERET_MEAN)

Mean value of the Feret diameters over all orientations according to the principle described above.

Diameter Definitions (4)

Feret Diameter, 90° to the Maximal Feret Diameter (FERET_MAX90)

First, the maximal Feret diameter, FERET_MAX, is calculated. The result is the Feret diameter measured at an angle of 90 degrees to that of the maximal Feret diameter.

Feret Diameter, 90° to the Minimal Feret Diameter (FERET_MIN90)

First, the minimal Feret diameter, FERET_MIN, is calculated. The result is the Feret diameter measured at an angle of 90 degrees to that of the minimal Feret diameter.

Diameter Definitions (5)

Minimum Area Bounding Rectangle

The calculation of the smallest encasing rectangle is based on the Feret diameter. The value is calculated as the minimum of the product of every possible pair of (x_{Feret}, $x_{Feret,90}$).

Minimum Area Bounding Rectangle, Length (BR_MAX)

The larger dimension of the smallest encasing rectangle is output.

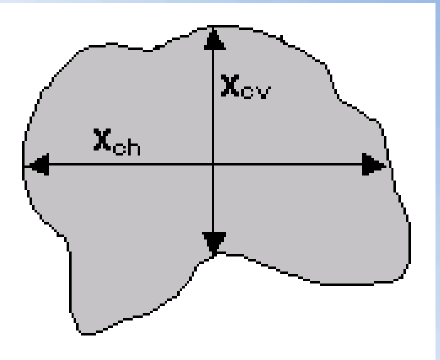
Minimum Area Bounding Rectangle, Width (BR_MIN)

The smaller dimension of the smallest encasing rectangle is output. This dimension corresponds quite well to the results of a sieve analysis.

Diameter Definitions (6)

★ Chord Length

This is not an diameter in its actual sense but the common basis of a group of diameters.
A chord length is defined by the distance of two points of the contour, measured exactly across the centre of gravity of the projection area. This is why all methods of evaluating the chord length imply an evaluation of the centre of gravity of the projection area.
Warning:
Chord length methods are problematic with strongly concave contours of a particle.



Example:
x_{cv} = Chord length vertical
x_{ch} = Chord length horizontal

Diameter Definitions (7)

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Chord Length, Vertical (CHORD_VERTICAL)

The result is the chord length measured vertically across the centre of the projection area.

Chord Length, Horizontal (CHORD_HORIZONTAL)

The result is the chord length measured horizontally across the centre of the projection area.

Chord Length, Maximal (CHORD_MAX)

The result is the largest chord length measured across the centre of the projection area.

Chord Length, Minimal (CHORD_MIN)

The result is the smallest chord length measured across the centre of the projection area.

Chord Length, 90° to the Maximalen Chord Length (CHORD_MAX90)

First, the maximal chord length is calculated. The result is the chord length at an angle of 90 degrees to that of the maximal chord length.

Chord Length, 90° to the Minimalen Chord Length (CHORD_MIN90)

First, the minimal chord length is calculated. The result is the chord length at an angle of 90 degrees to that of the minimal chord length.

Chord Length, Mean Value (CHORD_MEAN)

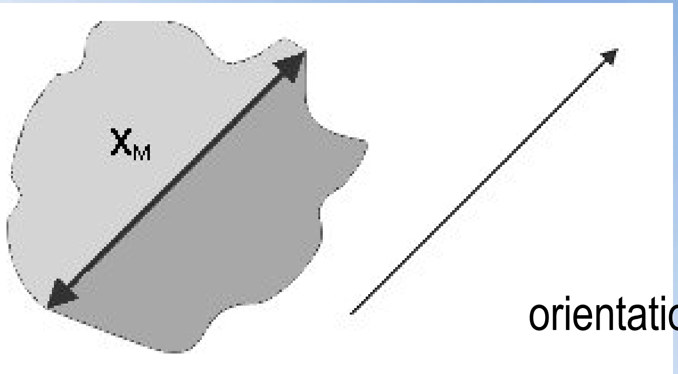
First, chord length values for a sufficient number of orientations are calculated. Their mean value is output.

Diameter Definitions (8)

★ Martin Diameter

This is not an diameter in its actual sense but the common basis of a group of diameters.
The Martin diameter, x_M, is that chord dividing the projection area of the particle into two equal halves.

Warning:
The Martin diameter is problematic if a particle has many concave parts of the contour and should be avoided in such cases.



orientation of measurement

Diameter Definitions (9)

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Martin Diameter, Maximal (MARTIN_MAX)

This is the maximal Martin diameter after consideration of all possible orientations (0°...180°). Internally, the Martin diameters of all possible orientations are calculated, and their maximum is output.

Martin Diameter, Minimal (MARTIN_MIN)

This is the minimal Martin diameter after consideration of all possible orientations (0°...180°). Internally, the Martin diameters of all possible orientations are calculated, and their minimum is output.

Martin Diameter, Mean Value (MARTIN_MEAN)

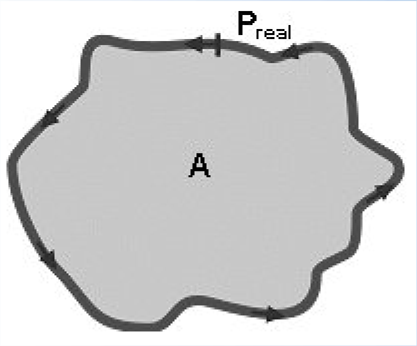
This is the mean value of the Martin diameters of all possible orientations according to the priciple described above.

Shape Factor Definitions (1)

★ Shape Factor Derived from the Equivalent Circle

Sphericity (SPHERICITY)

The sphericity, S, is the ratio of the perimeter of the equivalent circle, P_{EQPC}, to the real perimeter, P_{real}.



P = perimeter
A = area

The sphericity is defined by the formula below:

$$S = \frac{P_{EQPC}}{P_{real}} = \frac{2\sqrt{\pi \cdot A}}{P_{real}}$$

The result is a value between 0 and 1. The smaller the value, the more irregular is the shape of the particle. This results from the fact that an irregular shape causes an increase of the perimeter. The ratio is always based on the perimeter of the equivalent circle because this is the smallest possible perimeter wit a given projection area.

Shape Factor Definitions (2)

★ Shape Factor Derived from the Feret Diameter

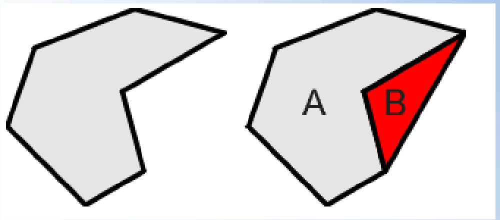
Aspect Ratio

The ratio of the minimal to the maximal Feret diameter is another measure for the particle shape.

★ Other Shape Parameters

Convexity

The convexity is an important shape parameter describing the compactness of a particle. The figure below shows a particle with projection area A (grey/light) leaving open a concave region of area B (red/dark) on its right hand side.



The convexity is defined as follows:

$$\psi_c = \frac{A}{A+B} = 1 - \frac{B}{A+B}$$

The WINDOX software calculates the convex hull of the particle projection. The convexity is the ratio of the projection area itself (A) and the area of the convex hull (A+B).

The maximum theoretical convexity is 1, if there are no concave regions. Due to the detector design (square pixels), however, all particles seem to have small concave regions, corresponding to the tiny steps with every pixel in the perimeter line. Therefore, the maximum convexity calculated in reality is mostly limited to 0.99.

Characterisation of Fibres

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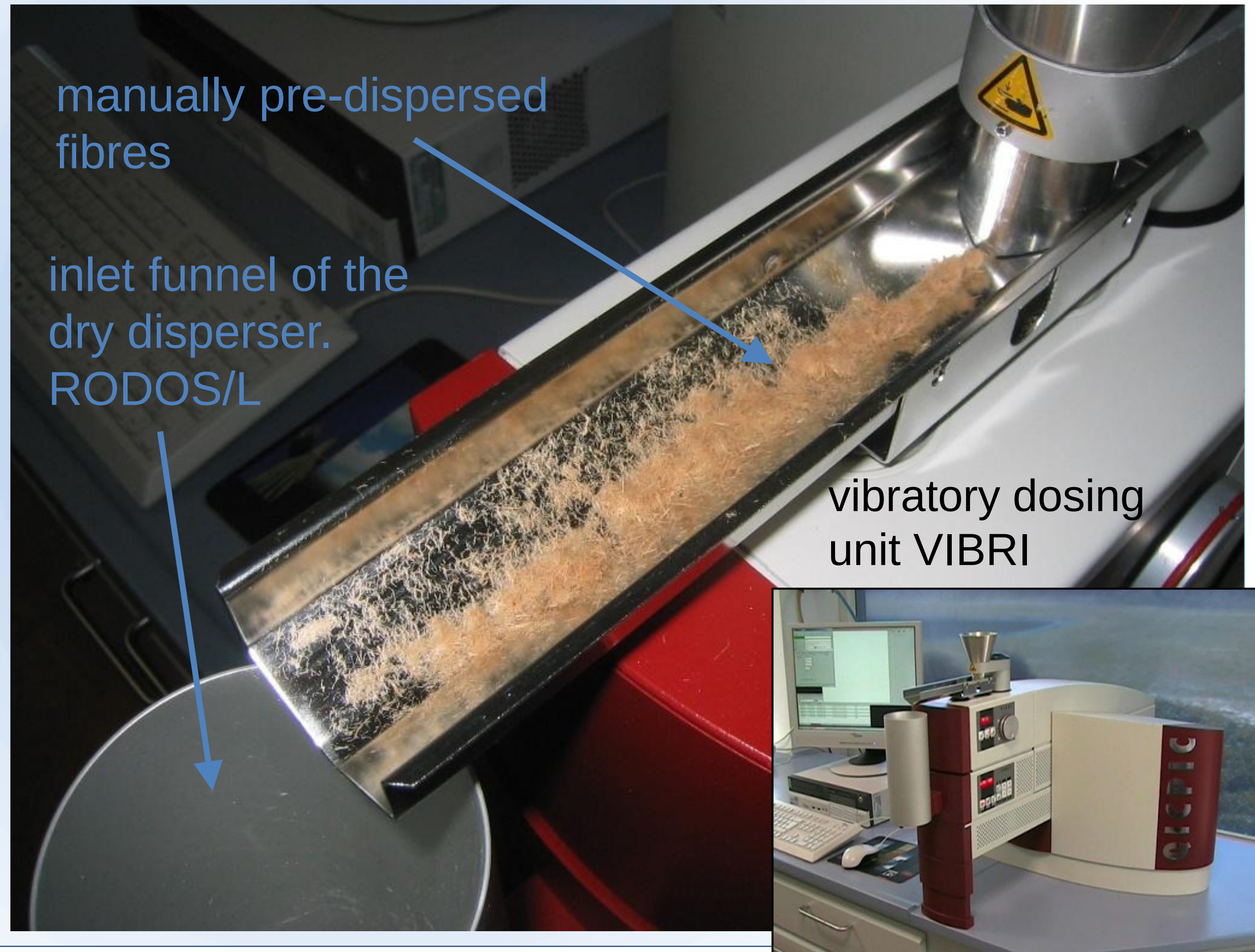
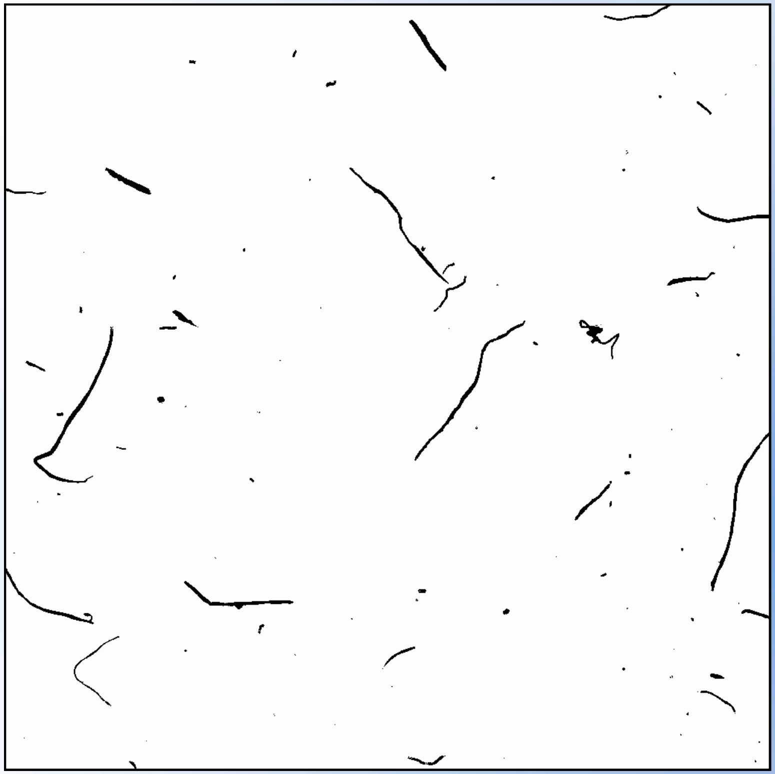
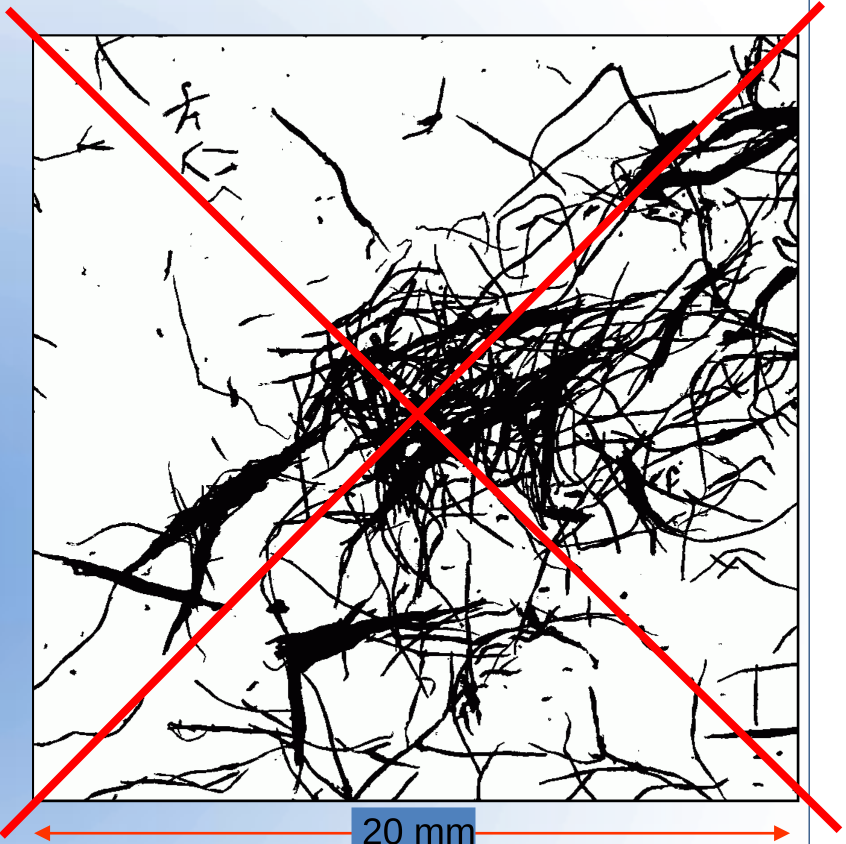
Example: wood shavings

- ☆ the fibres are *not free flowing*
- ➯ using *RODOS* dry disperser
- ➯ a manual *pre-dispersion* of the sample is required
- ➯ a V-shaped chute is recommended to *smoothen* the feed



Result: Dispersion of Fibres (wood shavings)





20 mm = 1024 pixel

20 mm

☆ because of use of *effective disperser*

- *overlapping fibres* are *avoided* as much as possible
- *high numbers of particles per image* are obtained
- well *de-agglomerated, diluted fields of fibres* are generated

Evaluation of Fibres

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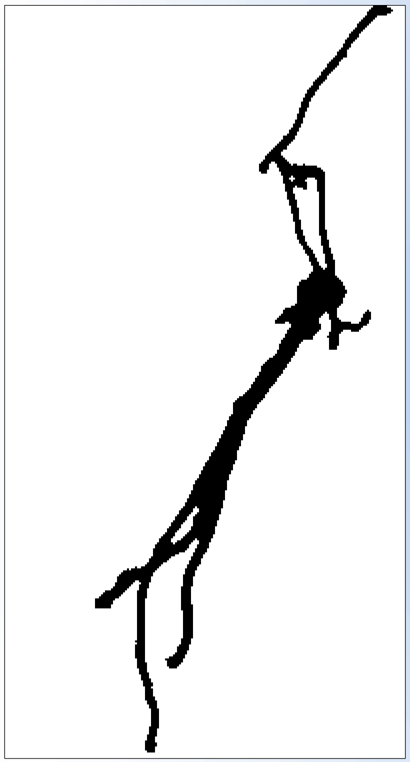
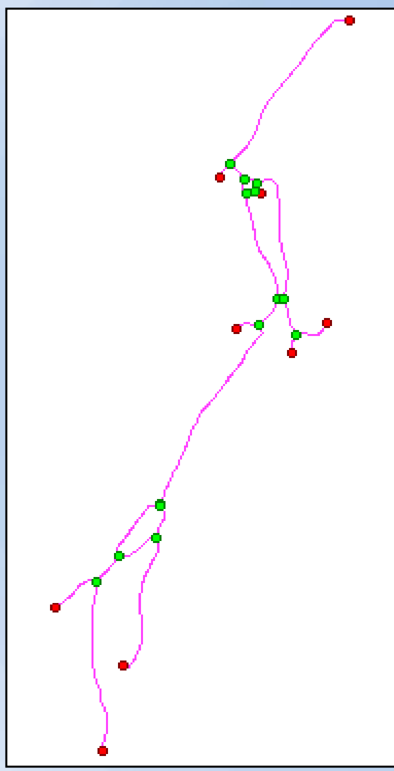
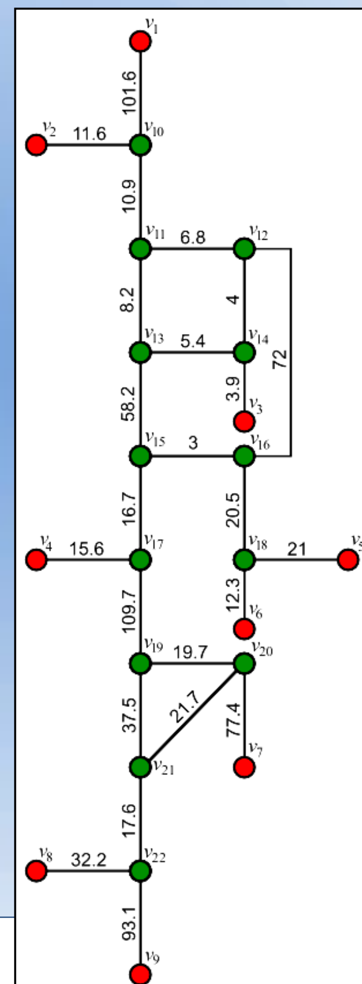
1. skeletonisation (erosion) Ahmed & Ward



2. search for all crossings, endpoints …



3. conversion of the fibre skeleton to a *graph* (*Dijkstra algorithms*)



4. **graph theory** can be used, e.g. to detect the *longest path between all endpoints*

- **LeFi** is *the shortest connection* between the *most distant end points*
- **DiFi** yields from total projected area *divided* by the sum of the length of all segments d_i

Size Definitions for Fibre-Shaped Particles (1)

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Length of Fibre (LEFI)

The length of a fibre is defined as the direct connection between its opposite ends, this is the longest direct path from one end to another within the particle contour. (Direct means without loops or deviations.)

Diameter of Fibre (DIFI and DIFIX)

One could imagine a number of ways to describe the diameter of a fibre by one mean value. The method implemented in WINDOX is to divide the projection area by the sum of all lengths of the branches of the fibre.

The calculation of DIFI is applied to those fibres only that are completely within the image frame, whereas the calculation of DIFIX also includes fibres touching the edge of the image.

Size Definitions for Fibre-Shaped Particles (2)

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Volume Based Fibre Diameter (VBFD)

This diameter is defined as the diameter of a sphere which has the same volume as the respective fibre. It is calculated by:

$$x_{VBFD} = \sqrt[3]{\frac{3}{2} x_D^2 \cdot x_L}$$

with xD, the fibre diameter (DIFI) and xL, the fibre length (LEFI).

The volume based fibre diameter is very useful if sample material consists of a mixture of granulate and fibres, and a distribution diagram of volume over particle size is desired. Neither LEFI nor DIFI can be used appropriately for the x-axis of a volume distribution diagram but VBFD contributes to an informative representation.

Size Definitions for Fibre-Shaped Particles (3)

Straightness of Fibre Shaped Particles

Most fibres, especially longer ones, tend to curl, and there have been several efforts to describe this phenomenon in terms of a single parameter.
One of the possible definitions is the straightness, proposed for the coming ISO standard 9276-6: STRAIGHT = FERET_MAX / LEFI
A value of 1 of the straightness represents a perfectly straight particle while values close to zero represent a greater deformation (curled fibres).

Curl Index

In earlier Versions of the WINDOX software, the Curl Index was used:
CURL_INDEX = LEFI / FERET_MAX - 1
which reflected the tradition in some industries, mainly the wood processing industry.

Elongation

This is the ratio of diameter and length of a fibre as defined by the formula, DIFI / LEFI. This parameter is also called excentricity.

Particle Gallery of Wood Fibres

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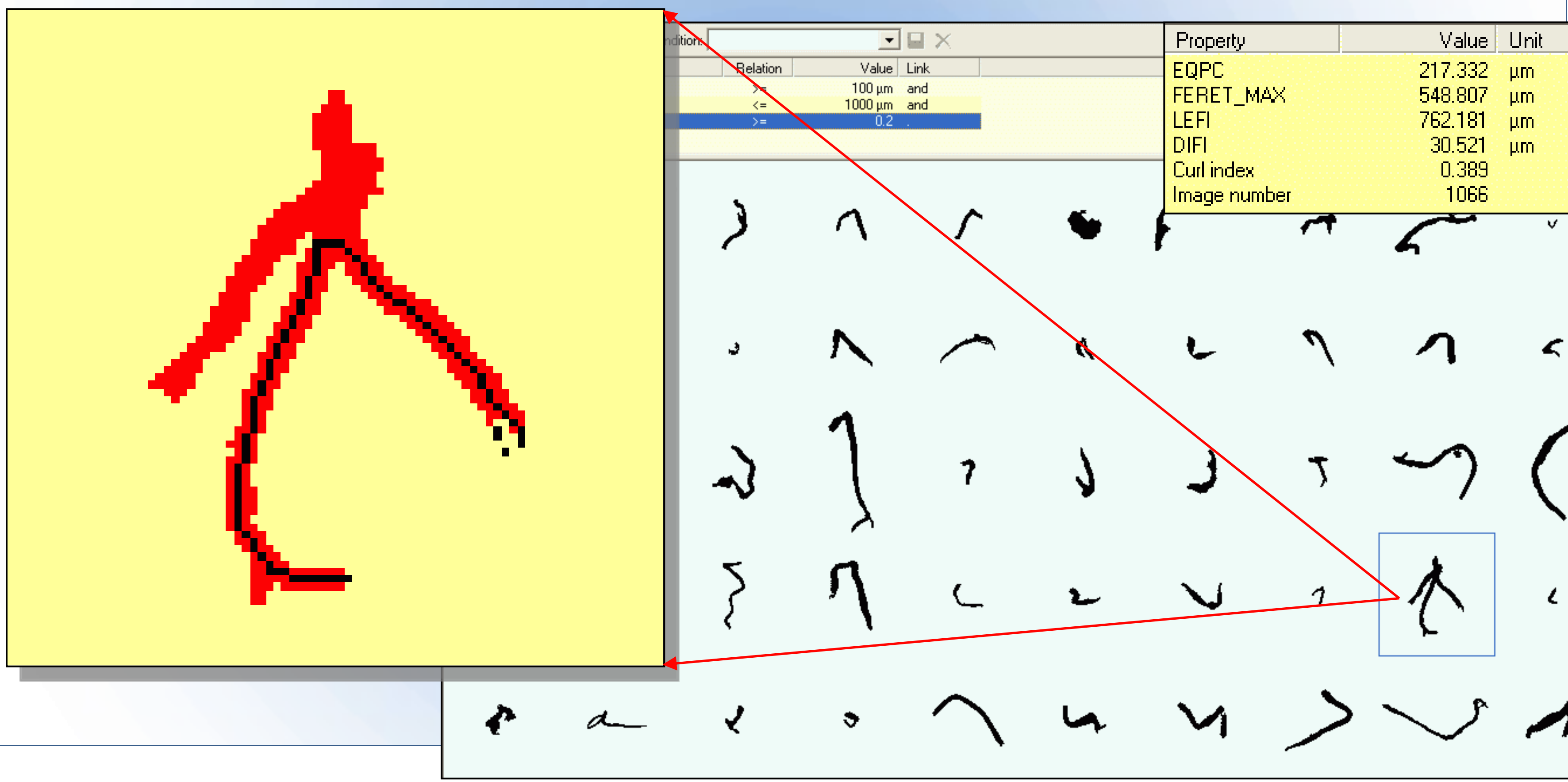
Individual shape information via filter conditions, e.g.

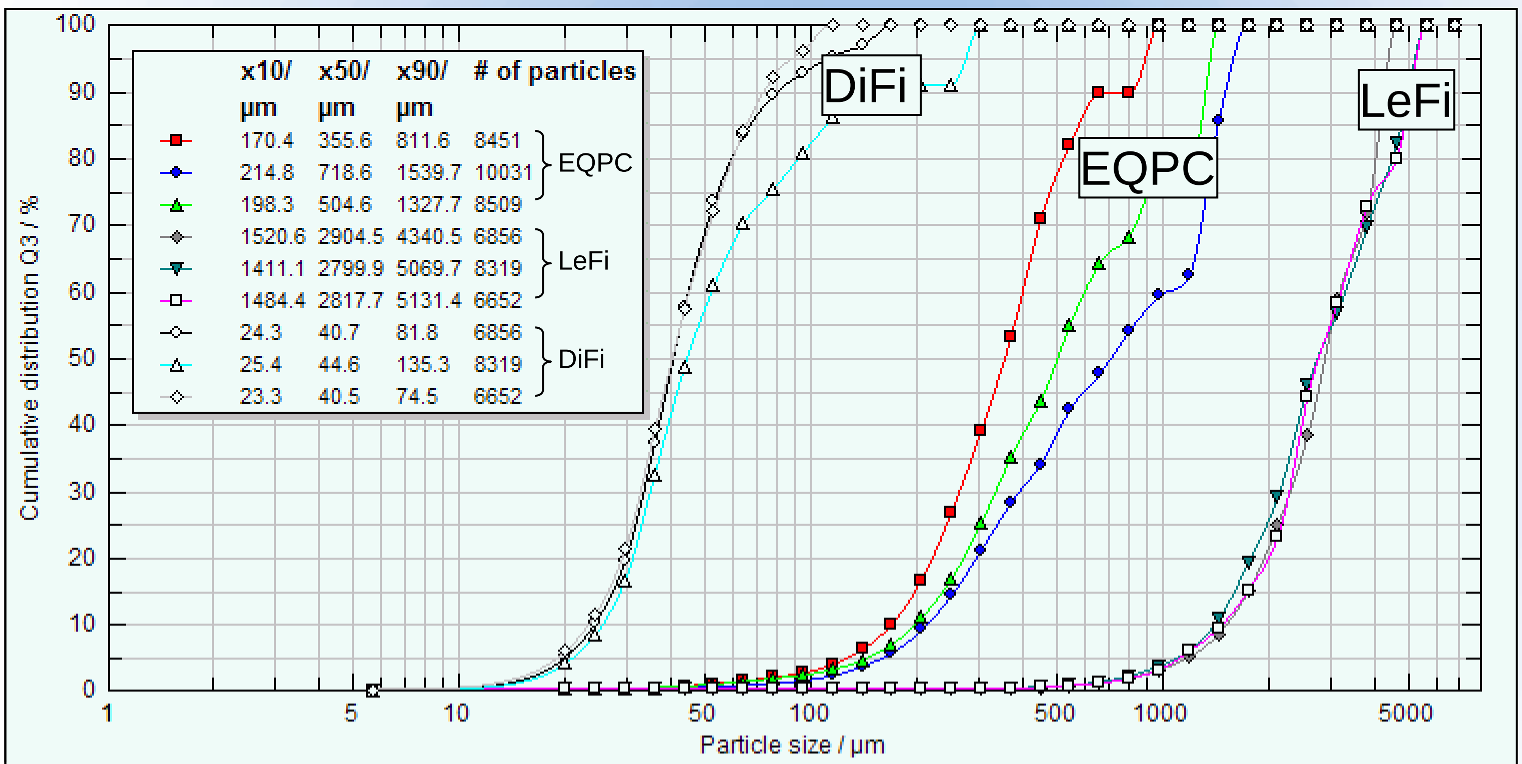
- ☆ 100 µm ≤ *LeFi* ≤ 1000 µm and *Straightness (curl-Index)* ≥ 0.2
- ☆ *back-traceability* to the original image

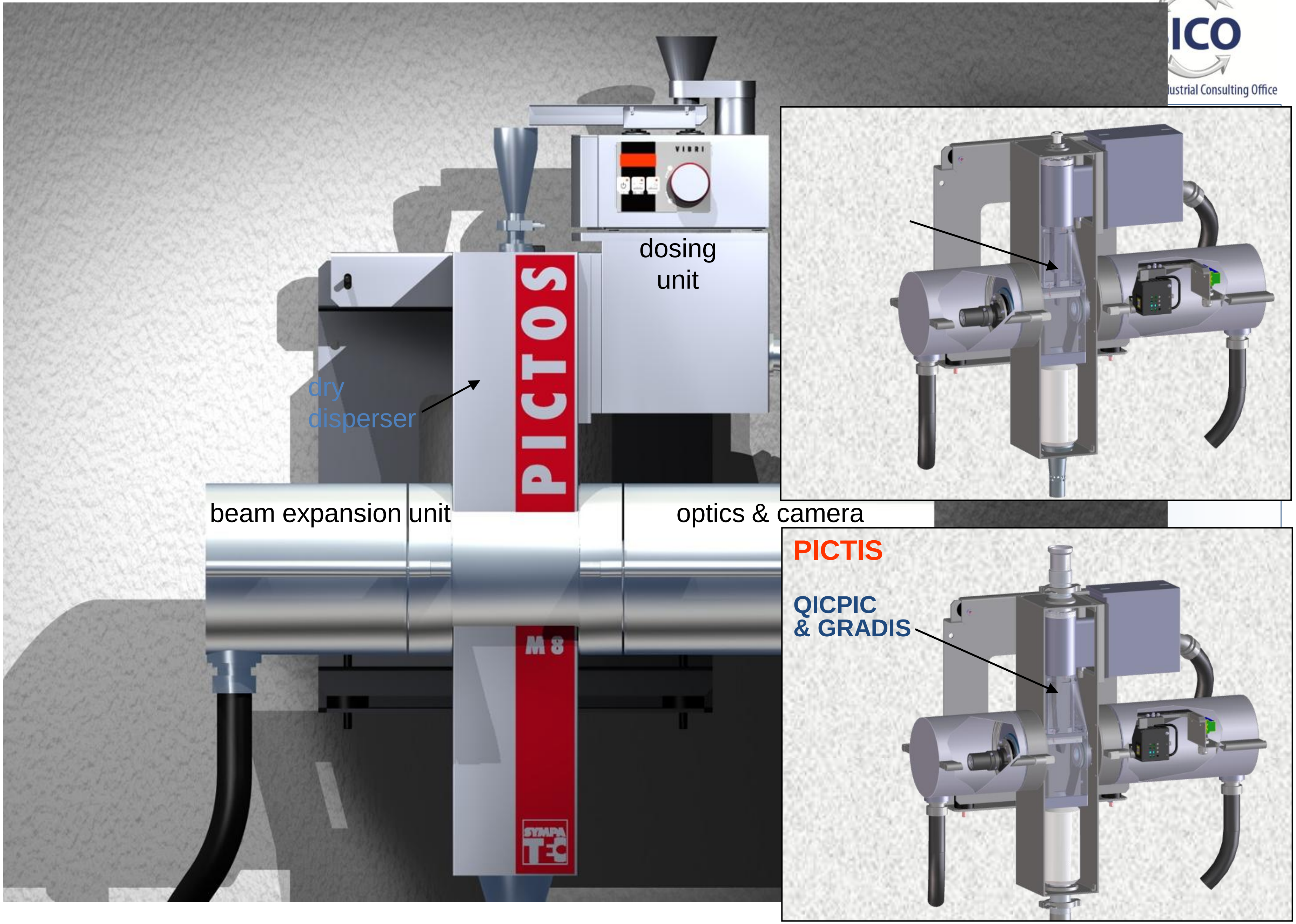


Results of Wood Fibres

Various evaluation modes are possible :

☆ EQPC, LeFi, DiFi, Feret$_{max}$, straightness, etc.




dosing
unit
dry
disperser
PICTOS
beam expansion unit
optics & camera
M 8
SYMPATEC
PICTIS
QICPIC
& GRADIS
VIBRI

Statistical Relevance: Number of Particles

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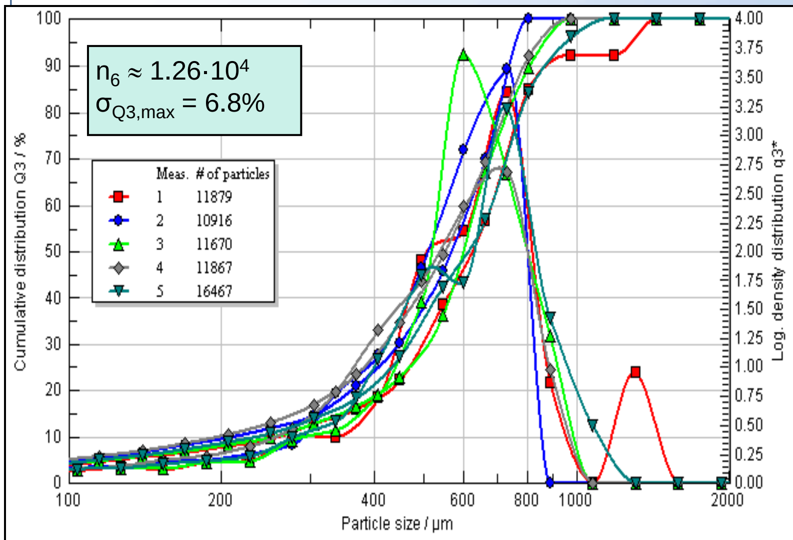
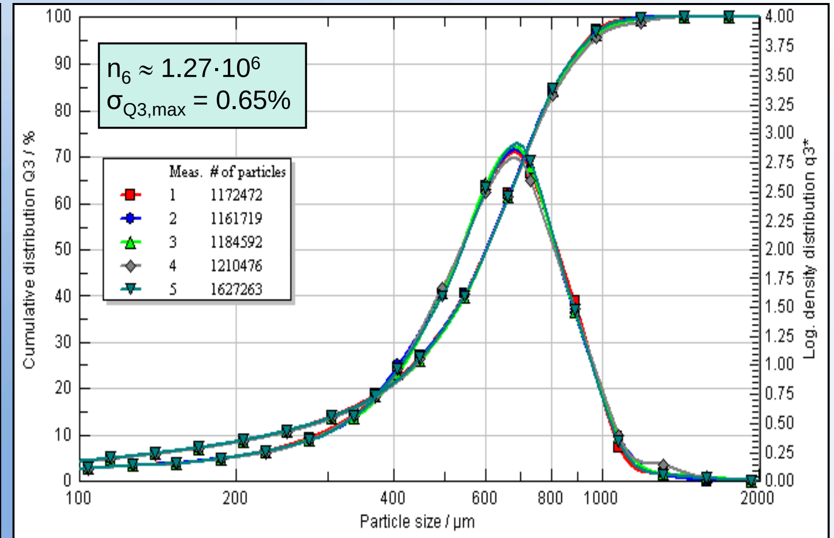
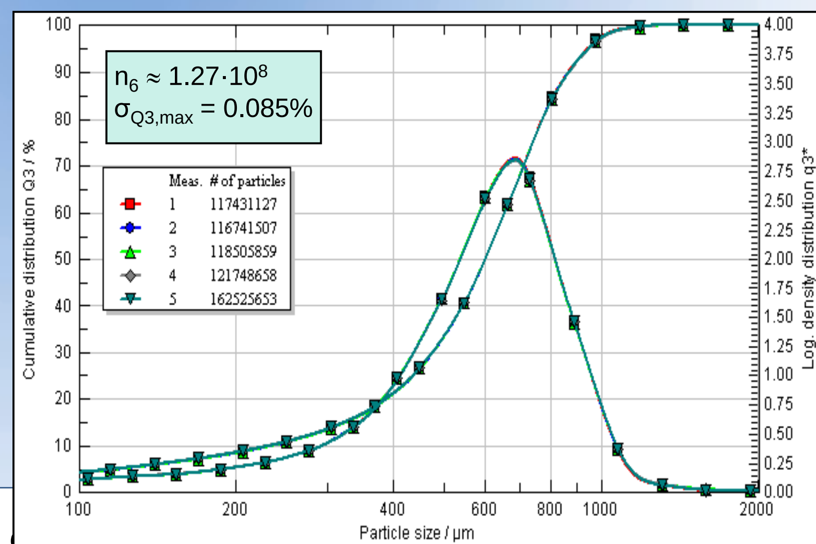
possible number of particles per measurement > 10^8





<u>Sample:</u>
coffee powder
75 mg



Statistics on Shape Parameters

SICO
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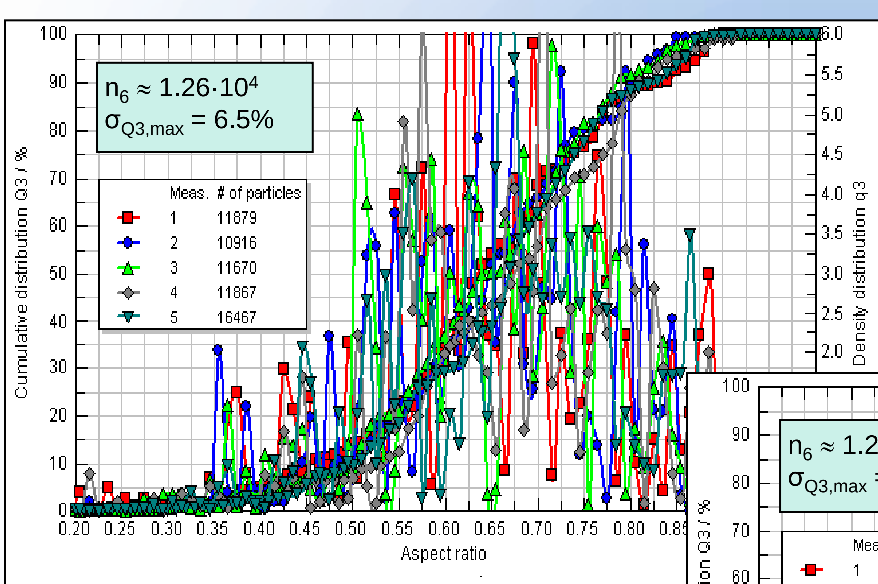
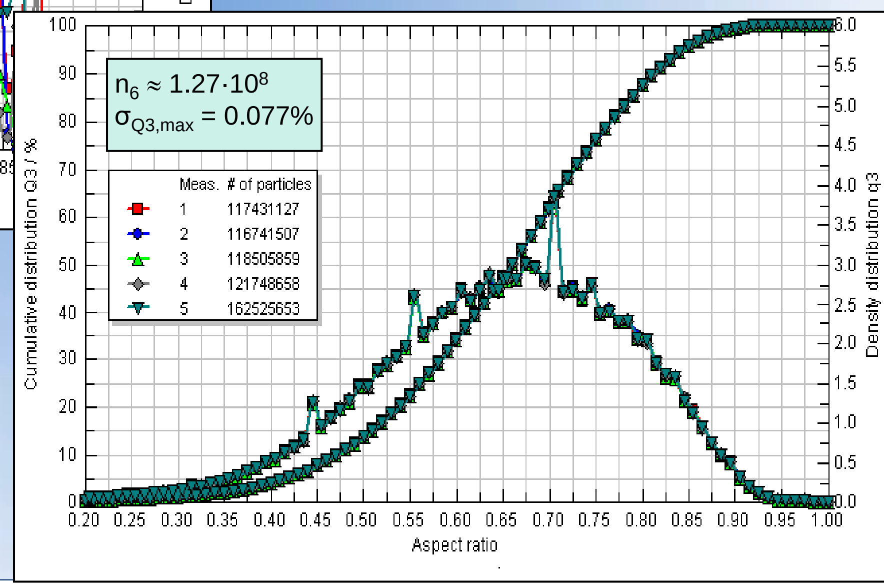
Number of particles per measurement raised to > 10^8

☆ What happens to the shape parameters, e.g. the aspect ratio?





<u>Sample:</u>
coffee powder
75 mg

Conclusion

- Since its introduction, QICPIC, the *combination* of *product adapted*, *powerful RODOS dispersion* and *high speed Image Analysis* has become a unique tool in laboratory and process for dry and wet applications

Main Benefits

- for dry and wet products
- size range *1 micron* to 20 mm
- *time of analysis for > 1 million particles in less than 1 minute*
- sample size of *> 100 million particles* (up to about 1 kg) per measurement
  - *time of analysis & statistical relevance* of results is comparable to well established Laser Diffraction
- *various size* and *shape parameters* available, including the characterisation of *fibres*
  - all results are *traceable* down to the *single particle*
- the method is traceable to the *standard metre*

QICPIC has opened new fields of applications in psa at highest precision

References

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- ☆ WITT, W., KÖHLER, U. and LIST, J., (2005), Experiences with Dry Dispersion and High-Speed Image Analysis for Size and Shape Characterisation, Particulate Systems Analysis, Stratford-upon-Avon, UK
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