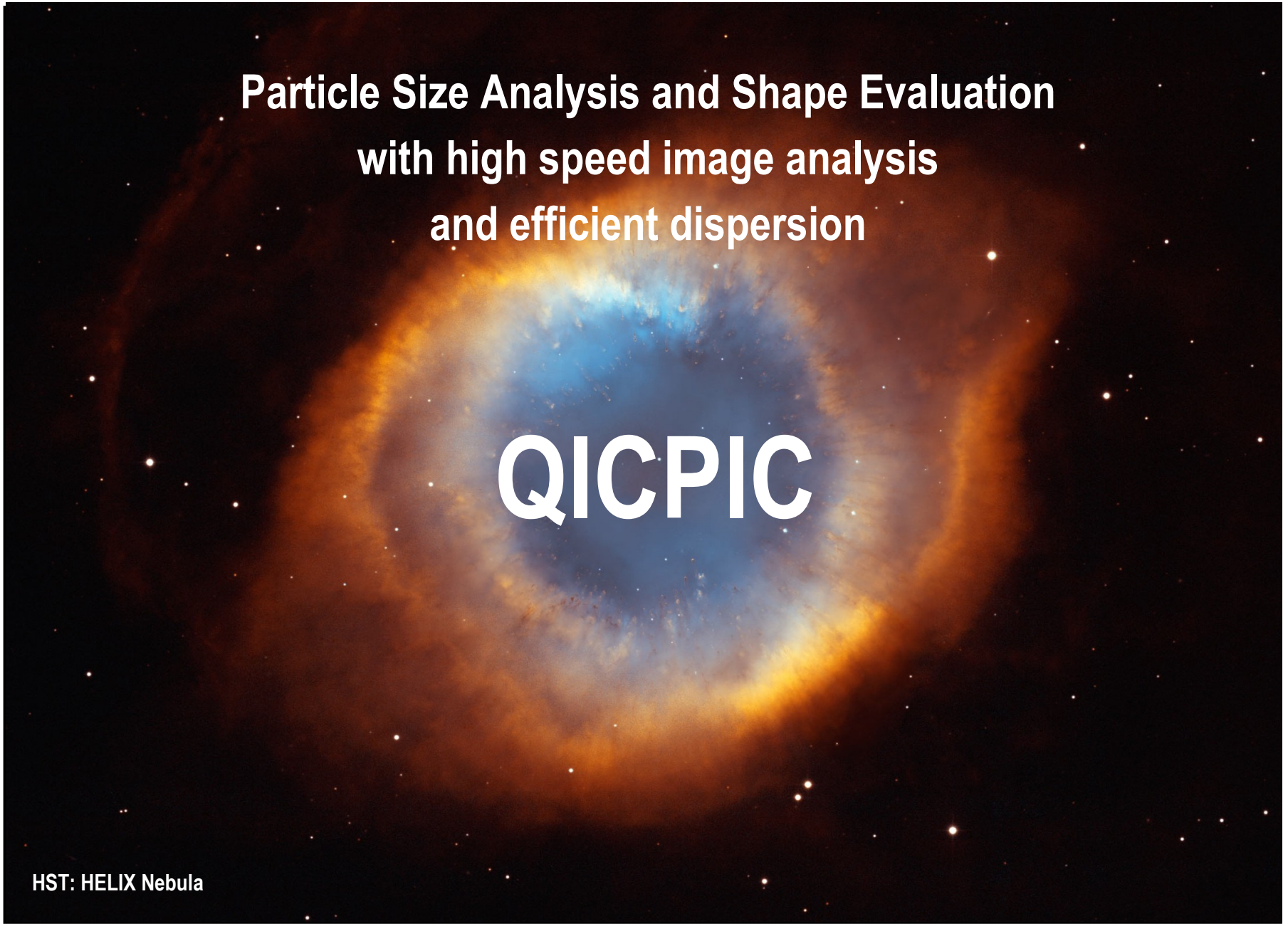
Particle Size Analysis and Shape Evaluation with high speed image analysis and efficient dispersion

QICPIC



HST: HELIX Nebula

Introduction

★ For characterisation of *real* particles *particle shape* in addition to the *particle size* gains more and more importance

☆ Image Analysis (IA) can give valuable services

★ *Static image analysis* (non-moving particles, e.g. on a microscope slide)

✔ Depth ε of the sharpness is defined

✔ High resolution for small particles

✔ Well established and standardised in ISO 13322-1 (Quimet, Kodak)

☆ Small amount of data

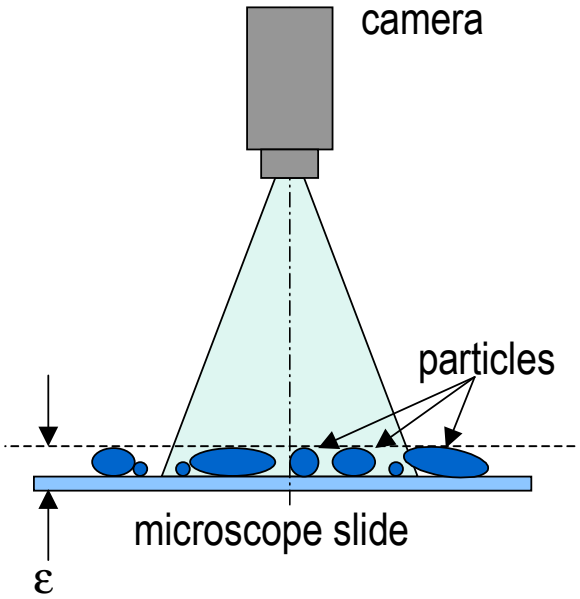
✘ Particles are *orientated* by the base

✘ Problem with *overlapping particles*

↳ High effort for software correction

✘ *Tiny sample size* → sampling problem

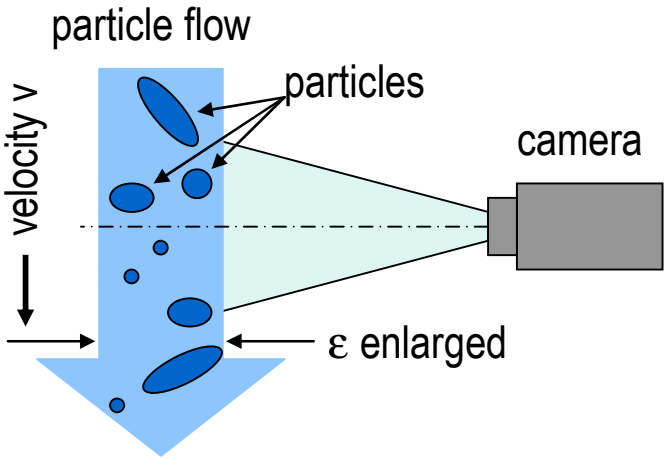
↳ Very poor statistics



Dynamic Image Analysis (Particles moving)

★ A flow of moving particles is captured

- ✔ *Enlarged sample size*
- ✔ *Arbitrary orientation*
- ✔ *Reduction of overlapping particles*



★ Several companies provide systems

- ☆ Reflection or transmission technology
- ☆ Wet dispersion or free fall
- ☆ Matrix or line scan cameras

★ Common to all systems is

- ✘ *Dry products: Free fall* → for well flowing bulk materials only, no dispersion
- ✘ *Wet dispersion* → *smallest sample sizes* and *slow particles*
- ✘ *Low frame rates* → small number of particles → *bad statistics*
- ✔ All system types will be integrated in the coming standard ISO 13322-2

Optimum

↳ **Dynamic image analysis with *powerful dispersion* and *good***

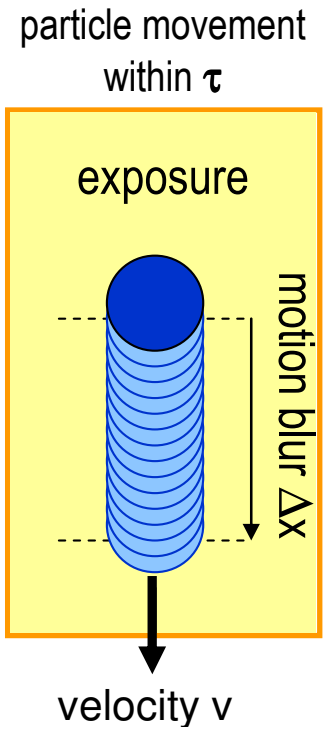
New Approach (1): Combination with Dispersers

- ★ Effective dispersion must be the prerequisite of image analysis
- ★ This adds *energy* to the particles → creates fast particles
  → which have to be *imaged clearly* within *exposure time*

Dry disperser RODOS™

- ★ Combines particle-to-particle, particle-to-wall and centrifugal forces caused by velocity gradients for effective dispersion down to *0.1 µm*
- ★ Particle velocity in aerosol beam: Up to *100 m/s*
- ★ Flash lamps with τ = 100 µs: Motion blur up to *10 mm (!)*
- ★ Best flash lamps with τ = 1 µs: Motion blur of up to *100 µm*

  ↳ Required exposure time:
  τ < *10 ns* for motion blur of < *1 µm*



New Approach (2): High Frame Rates

★ In order to obtain a *representative sample* and a *good statistical reliability* a large number of particles should be acquired in short time

Example 1 — 1 % precision requires 10.000 particles within one size class
Within 10 particles/image → 1000 images or 40 s measuring time
at 25 images/s per class. → for 30 equally populated classes:
The measuring time is about *1200 s = 20 minutes*

↳ *Not acceptable*

Example 2 — As above, but 500 images/s, and because of good dispersion
20 particles/image → for 30 equally populated classes:
The measuring time is only *30 s* !

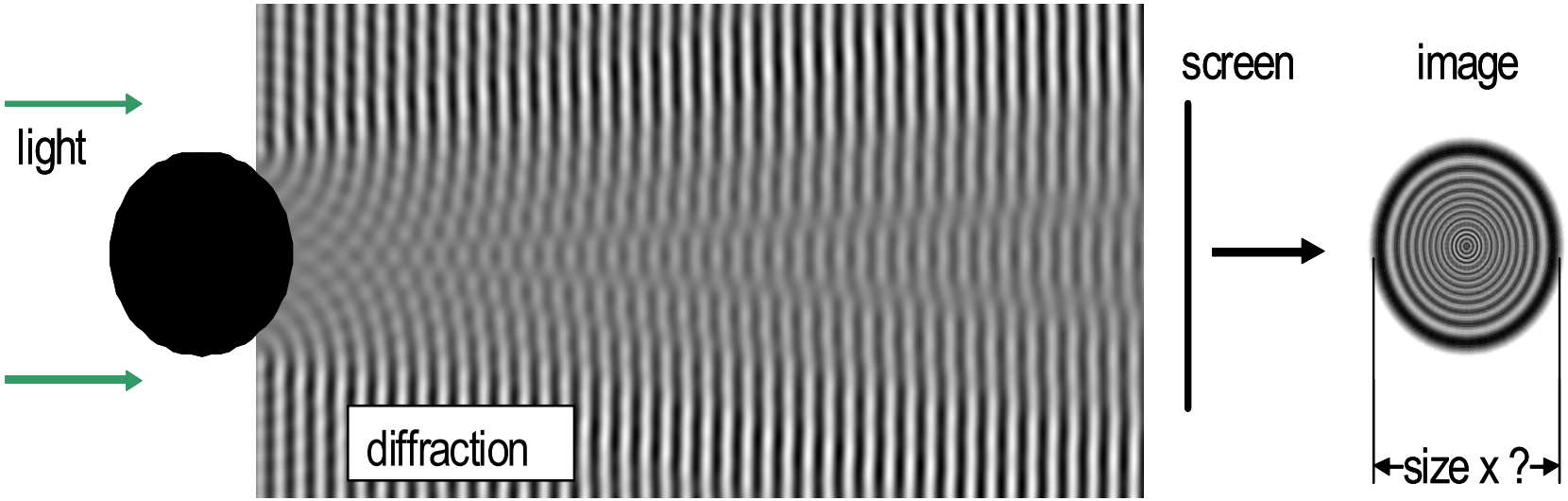
↳ *OK*

↳ *High frame rate required* (>> 25 images/s)
↳ *Extreme data volumes* (500 Mbyte/s at 500 images/s, 1 Mpixel, 8 bit)

New Approach (3): Special Optical Set-up

★ Even a *perfect parallel beam* will not produce a clear shadow of a small particle

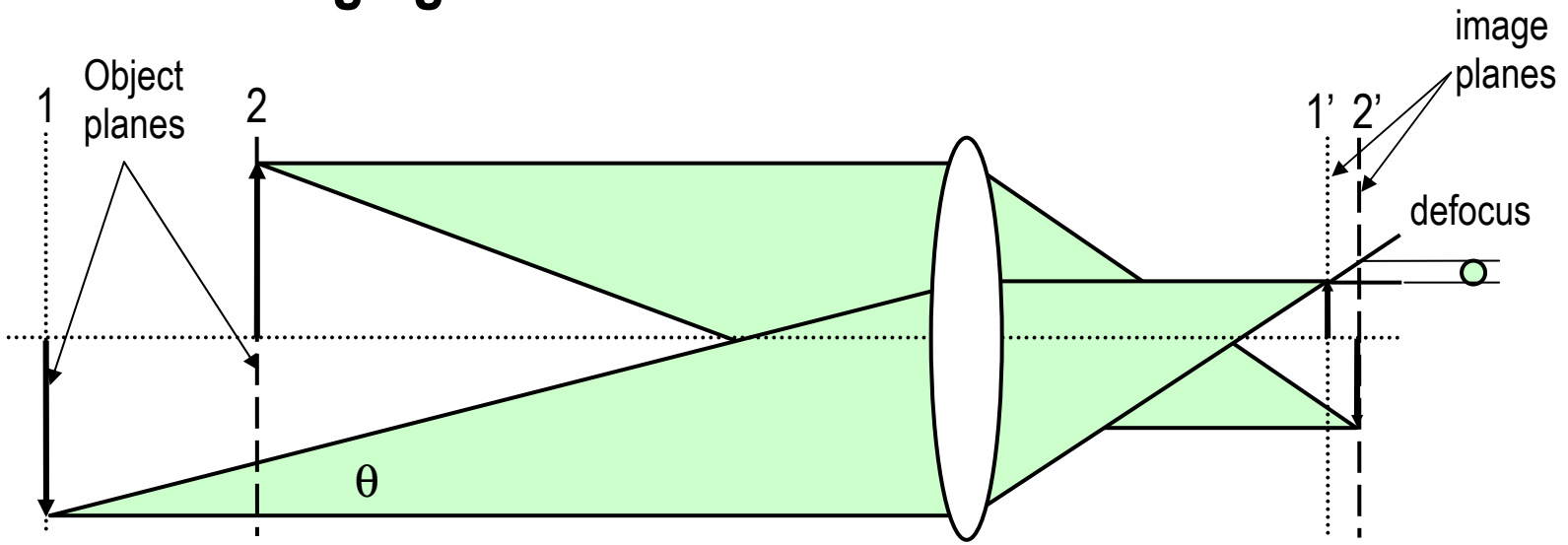
↳ The intensity pattern depends on the distance between sensor and particle



★ Without imaging optics

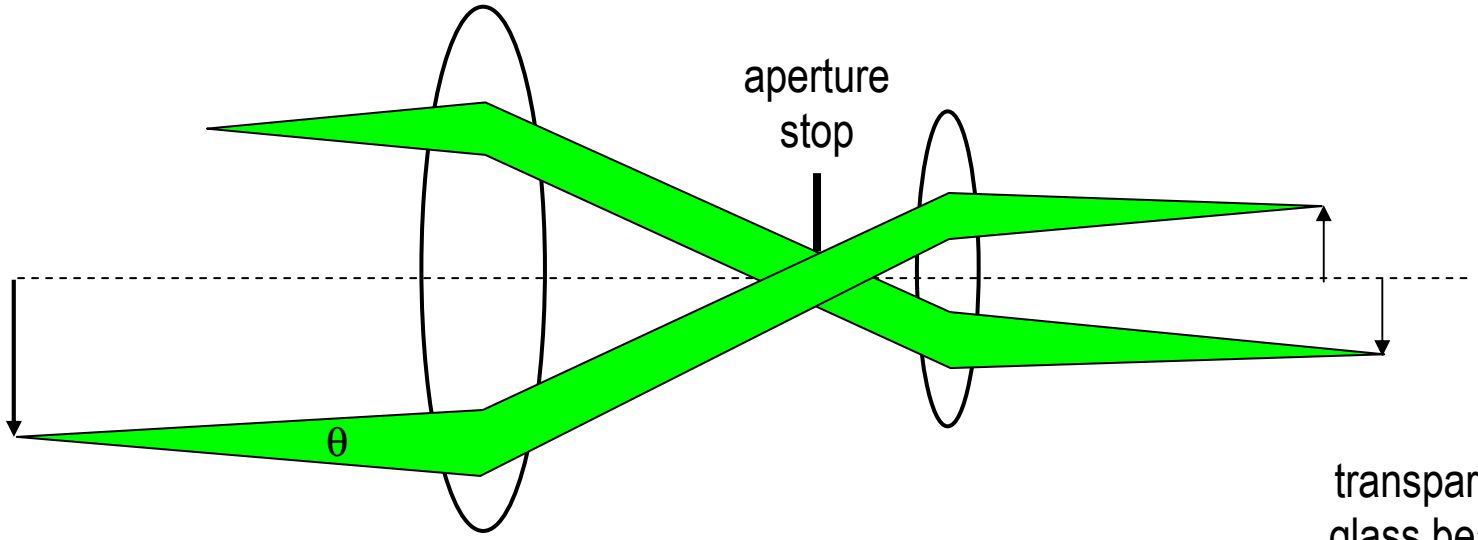
✘ Sensor must be *very close* to the particles
✘ Image quality *depends on particle size* → high calculation effort
✘ Sensor must be scaled to the particle size

Standard Imaging Lens

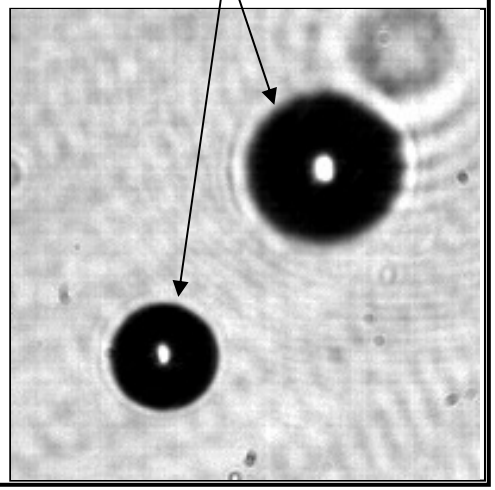


- ★ A lens transfers light from one point at the *object plane* to one point in the corresponding *image plane*
- ★ At image plane 2' the image of arrow 1 is blurred
  - ↳ Only objects located at a single object plane are in focus on a flat sensor
- ★ The size of *blurred spot* depends on *distance of the object to the object plane* and the *angle of rays* θ which passes the lens
- ✘ Magnification depends on object position (perspective image)

Special Imaging Lens

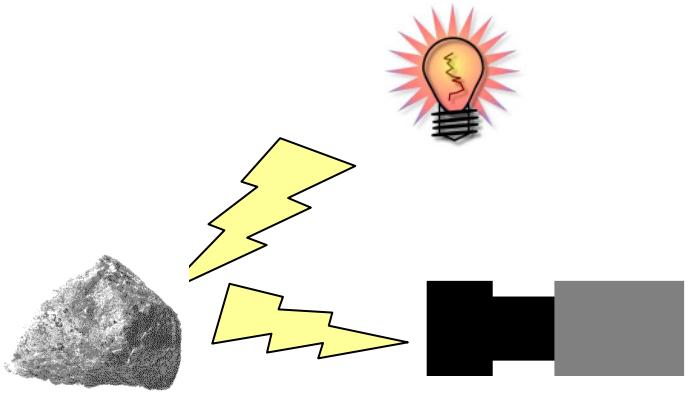


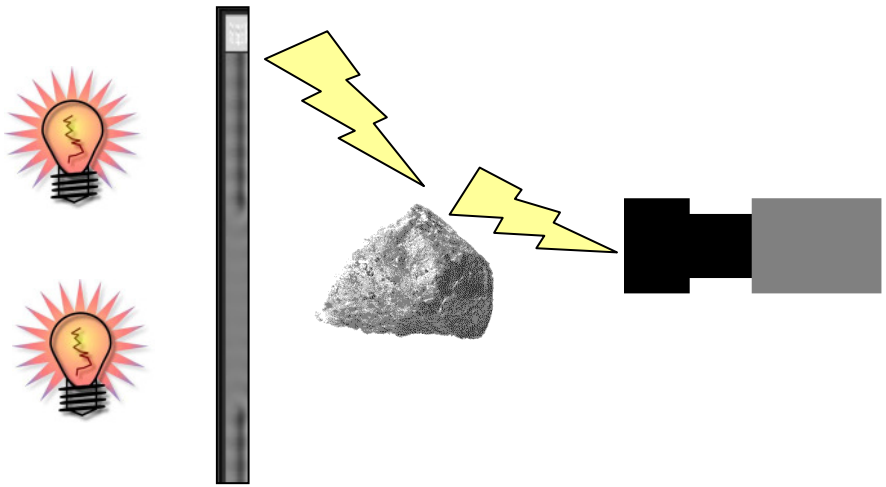
transparent
glass beads



- ★ Only ray fans *parallel to the optical axis* are used for the image
- ★ The aperture stop controls the angle of rays θ (aperture angle)
  - ✔ Image size does not depend on object position
    - ↳ Image size *less sensitive to defocus*
    - ↳ Even *transparent particles* show *high contrast*, as deflected light is not imaged
    - ↳ Calculation effort is *remarkably reduced*

Illumination





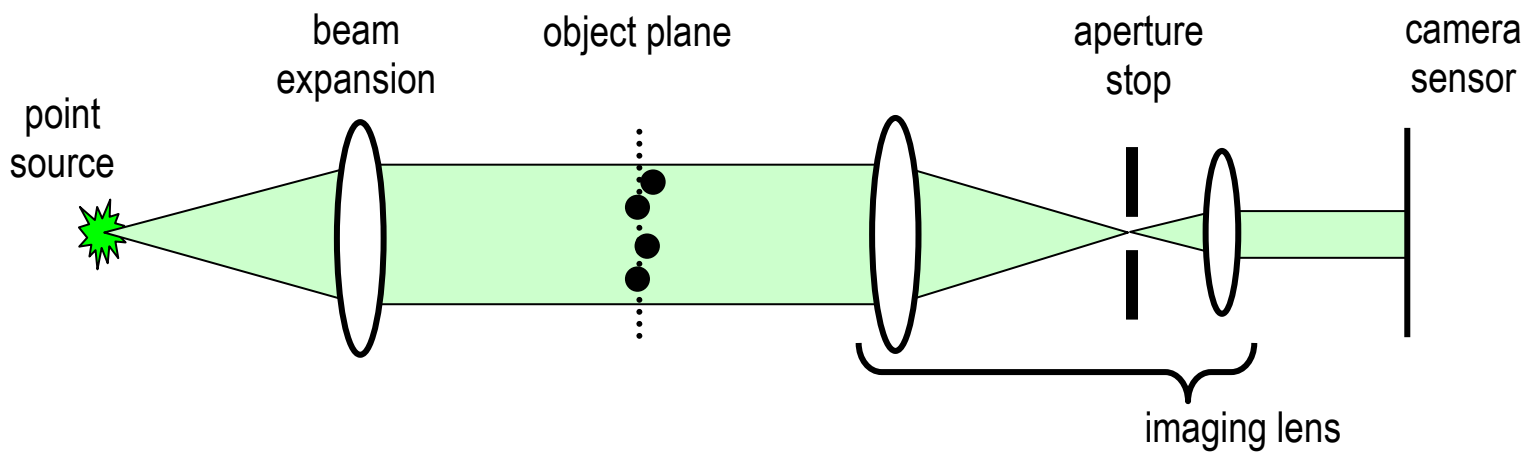
★ Front illumination

- ✔ Very *realistic images incl. surface structure*
- ✘ Reflection depends on *particle material and shape*
- ✘ Difficult and error-prone image analysis

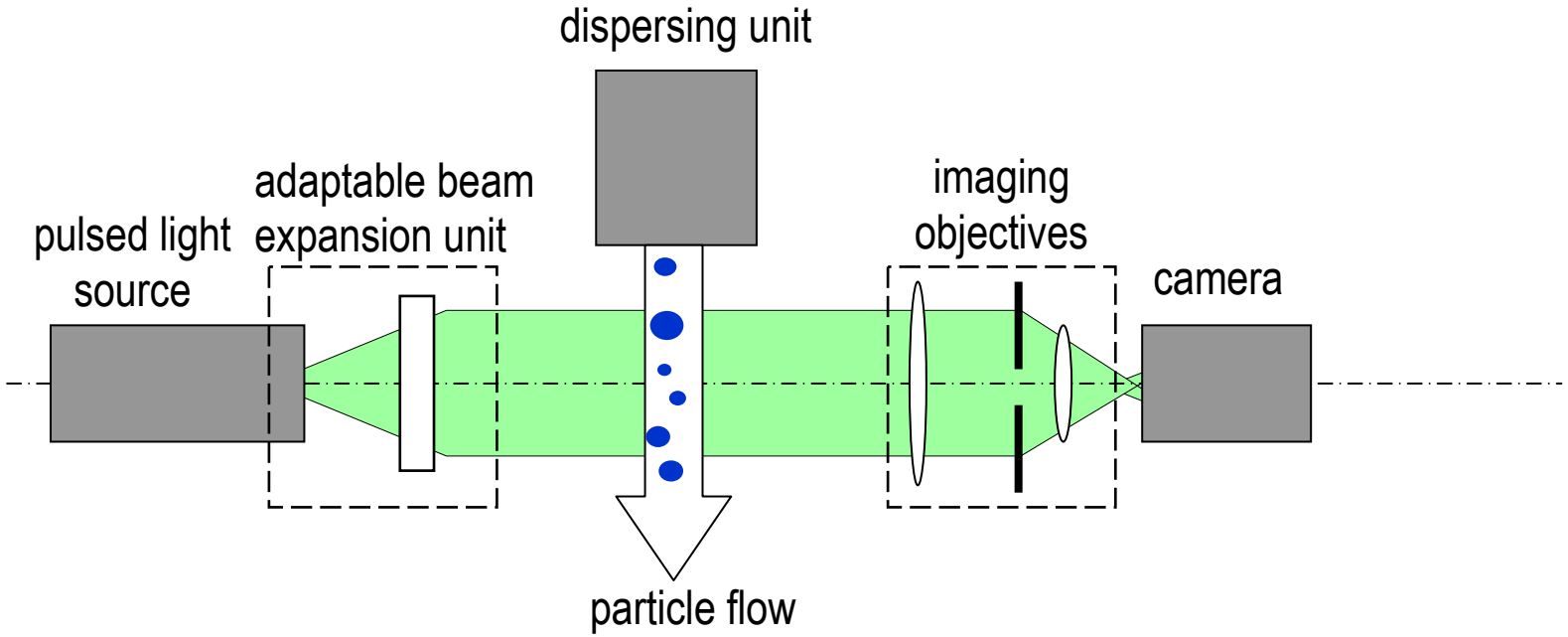
★ Illumination box

- ✘ Light can still be *reflected by particle borders*
- ✘ *Small aperture stop* is required to reduce reflection
  - ↳ Very much light is required
  - ↳ *Limited imaging* of small particles

Illumination in Transmission



- ✔ The *smallest aperture angles* can be used → highest contrast
- ✔ The aperture stop blocks only *stray light* and large angle *diffracted light* from the particles
  - ✔ The aperture stop has *no influence* on the brightness of the background → only small light intensities are required
  - ✔ The *depths of field* is limited by diffraction only → wide working distance



★ Pulsed light source

- ✔ Pulse duration: < 1 ns
- ✔ Output power: ≈ 0,15 nJ/pulse
- ✔ Light colour: visible (≈ 532 nm)
- ✔ Repetition rate: 0 to 500 Hz, adjustable

★ Beam expansion unit

- ✔ 35 mm Ø, 16 mm Ø, 7 mm Ø

★ Imaging objectives
(up to 5 on a carousel)

- ✔ 2:1, 1:1, 1:3, (1:10) …

★ Camera

- ✔ CMOS, 1024 x 1204 pixel,
- ✔ Square pixel, 10 µm²
- ✔ Up to 500 fps

QICPIC & RODOS/L

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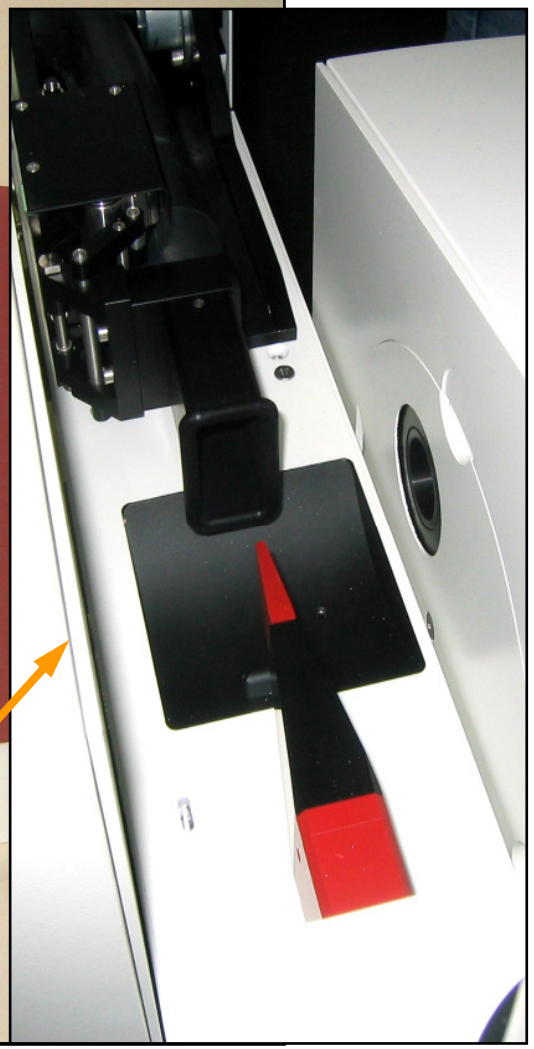


Measuring zone of
dry disperser RODOS/L

QICPIC & GRADIS/L

Gravity disperser

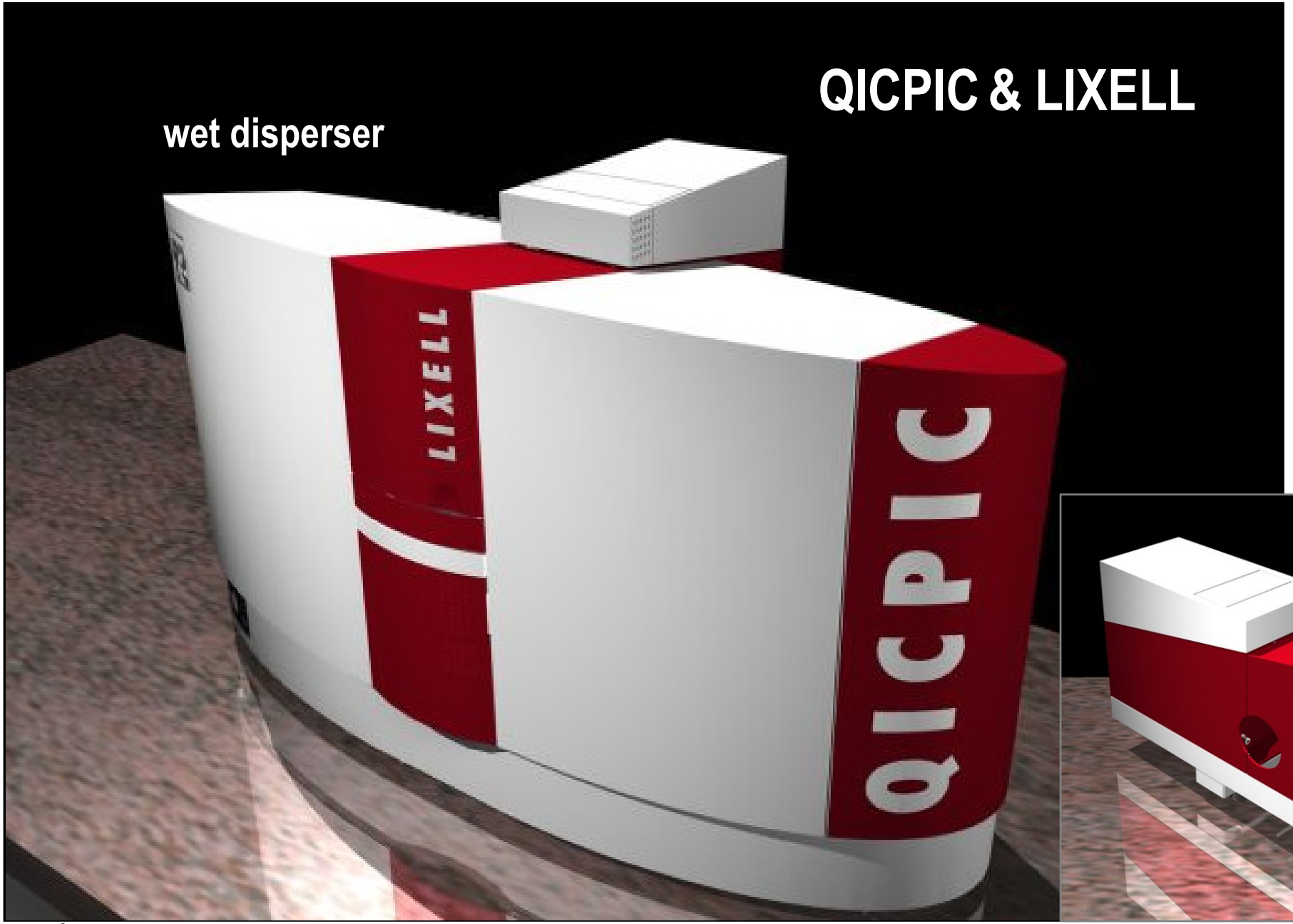
Side view

QICPIC & OASIS/L

dry & wet
disperser

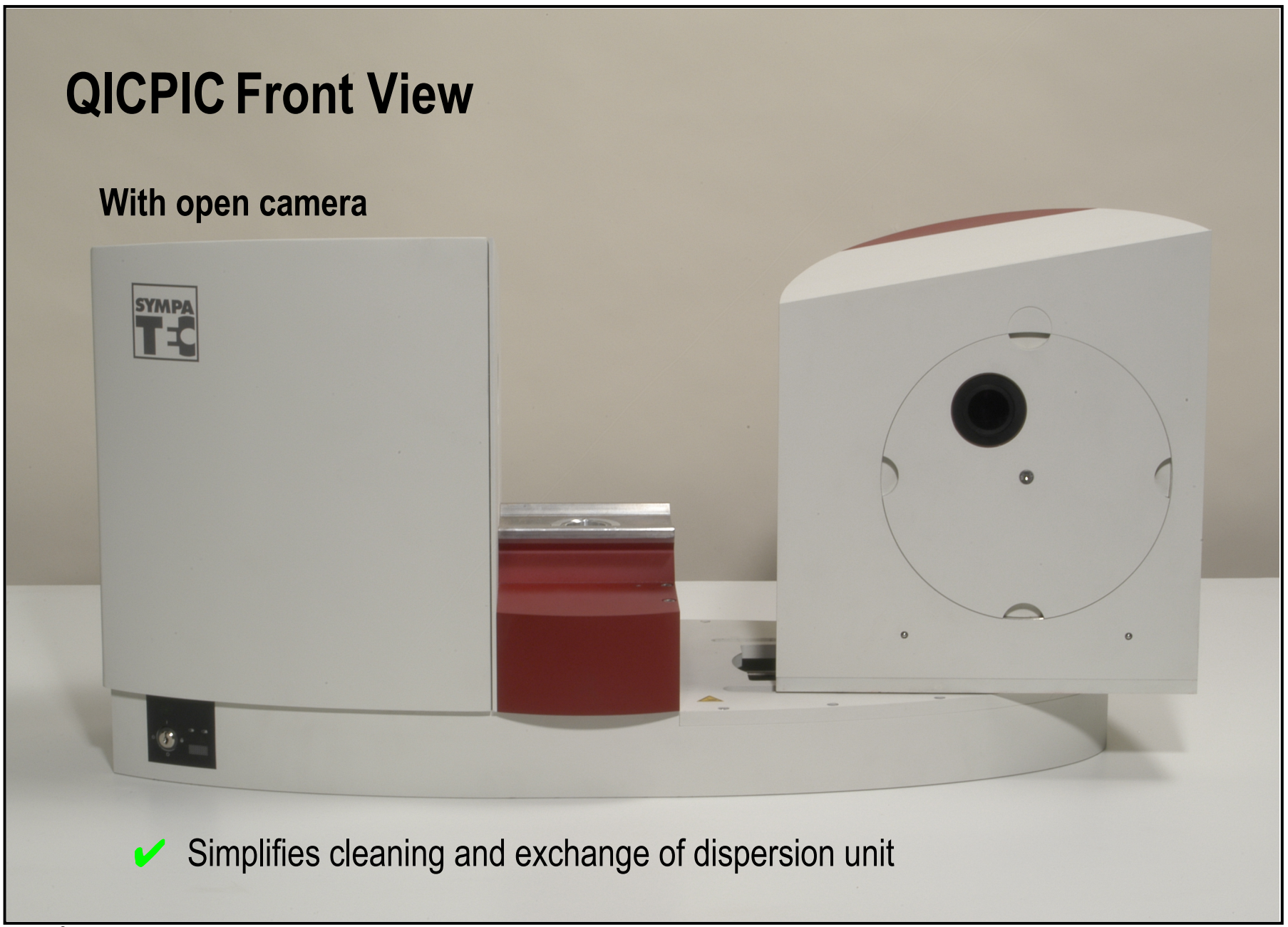
QICPIC & LIXELL

wet disperser



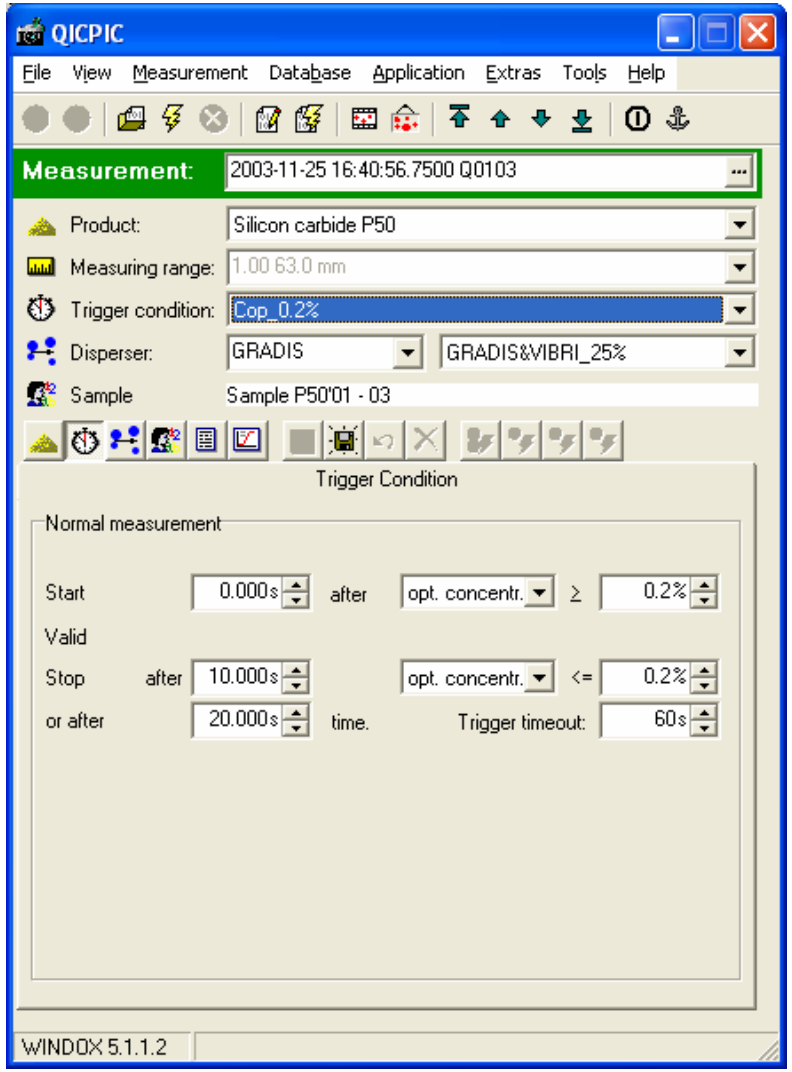
QICPIC Front View

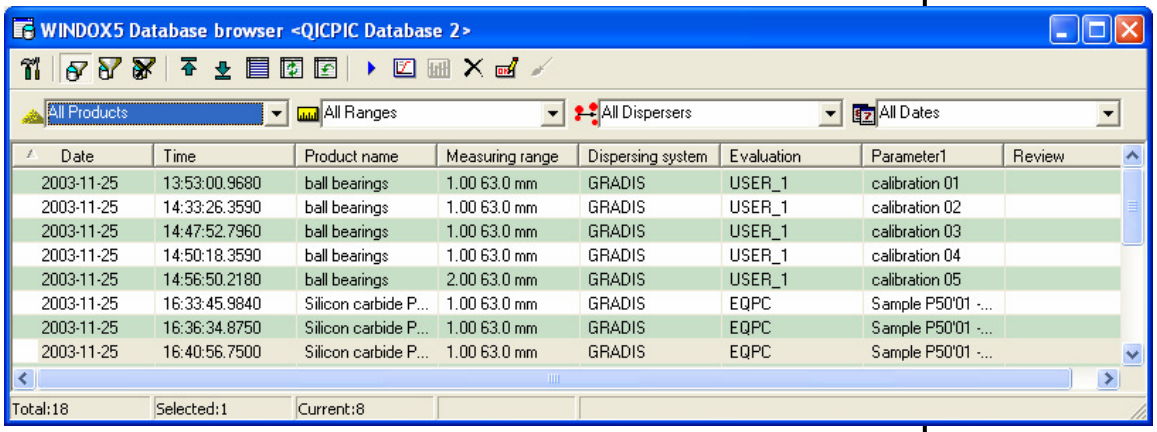
With open camera



✔ Simplifies cleaning and exchange of dispersion unit

QICPIC Sensor Control



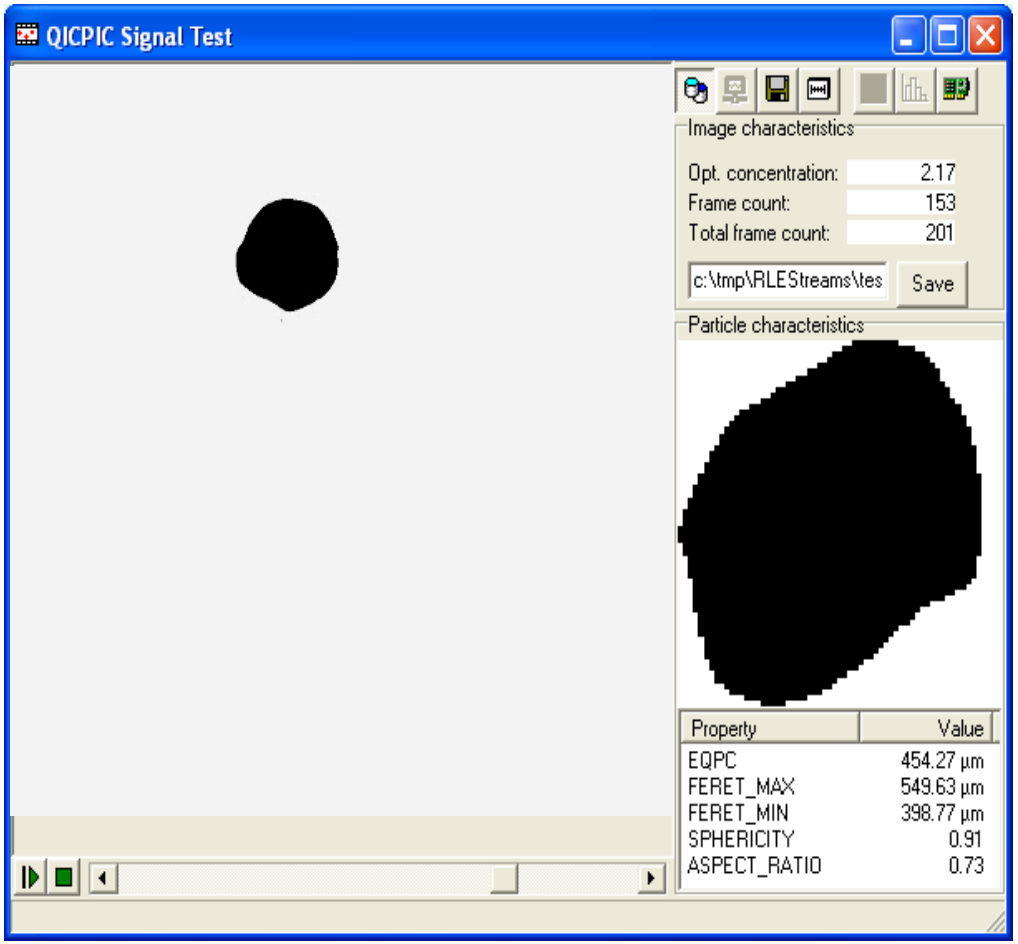


★ Every measurement is characterised with:

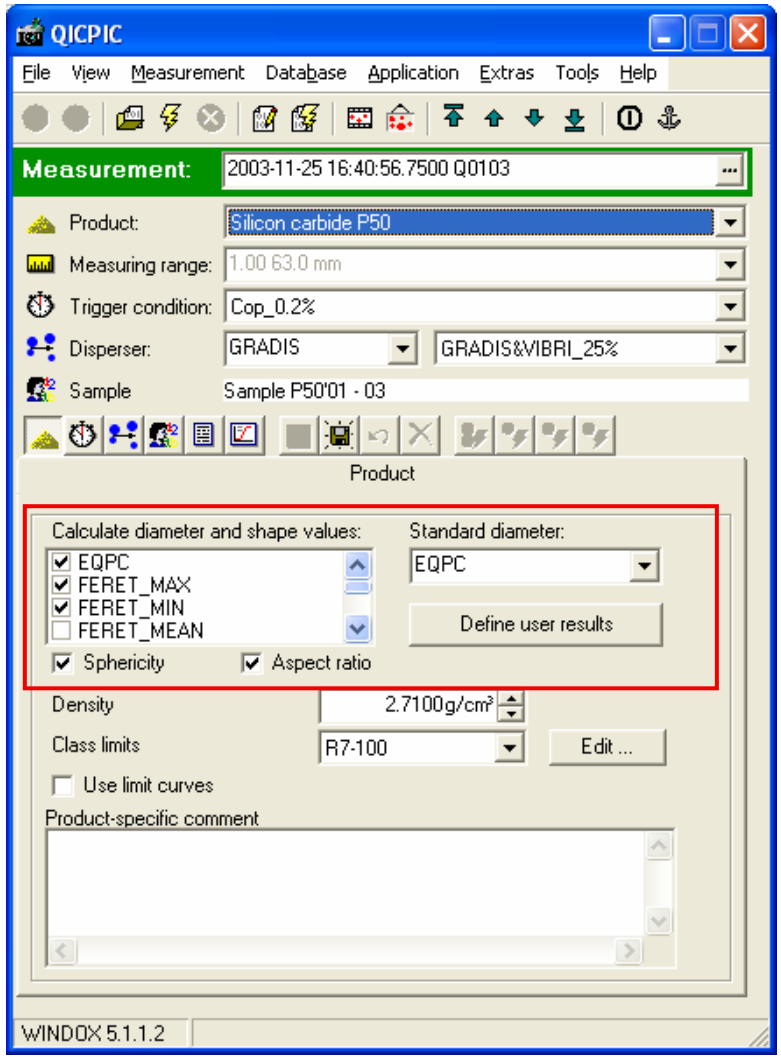
- ☆ Unique *primary key*
- ☆ Product
- ☆ Measuring range
- ☆ Trigger condition
- ☆ Disperser
- ☆ User specific parameters

Signal Test

- ✔ *On-line monitoring* during measurements (10 fps) *Video player* function in database mode
  - ☆ Scaled and non-scaled
- ✔ Selection of a particle directly: Displays size and shape parameters
- ✔ Export via clipboard or video streaming file (AVI)



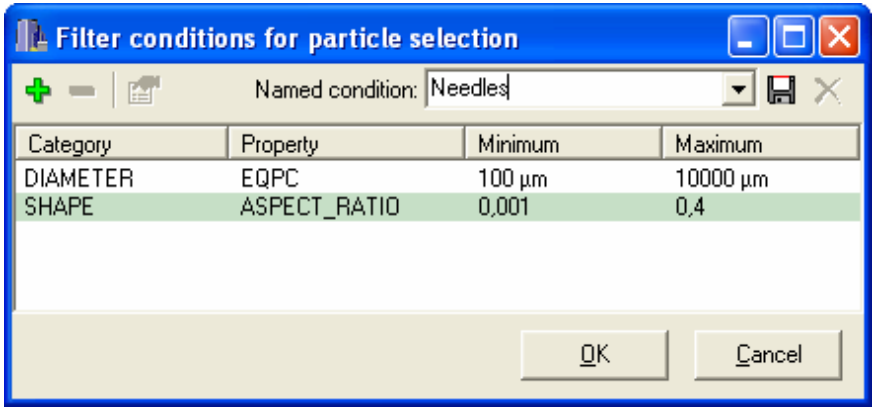
Calculation Modes

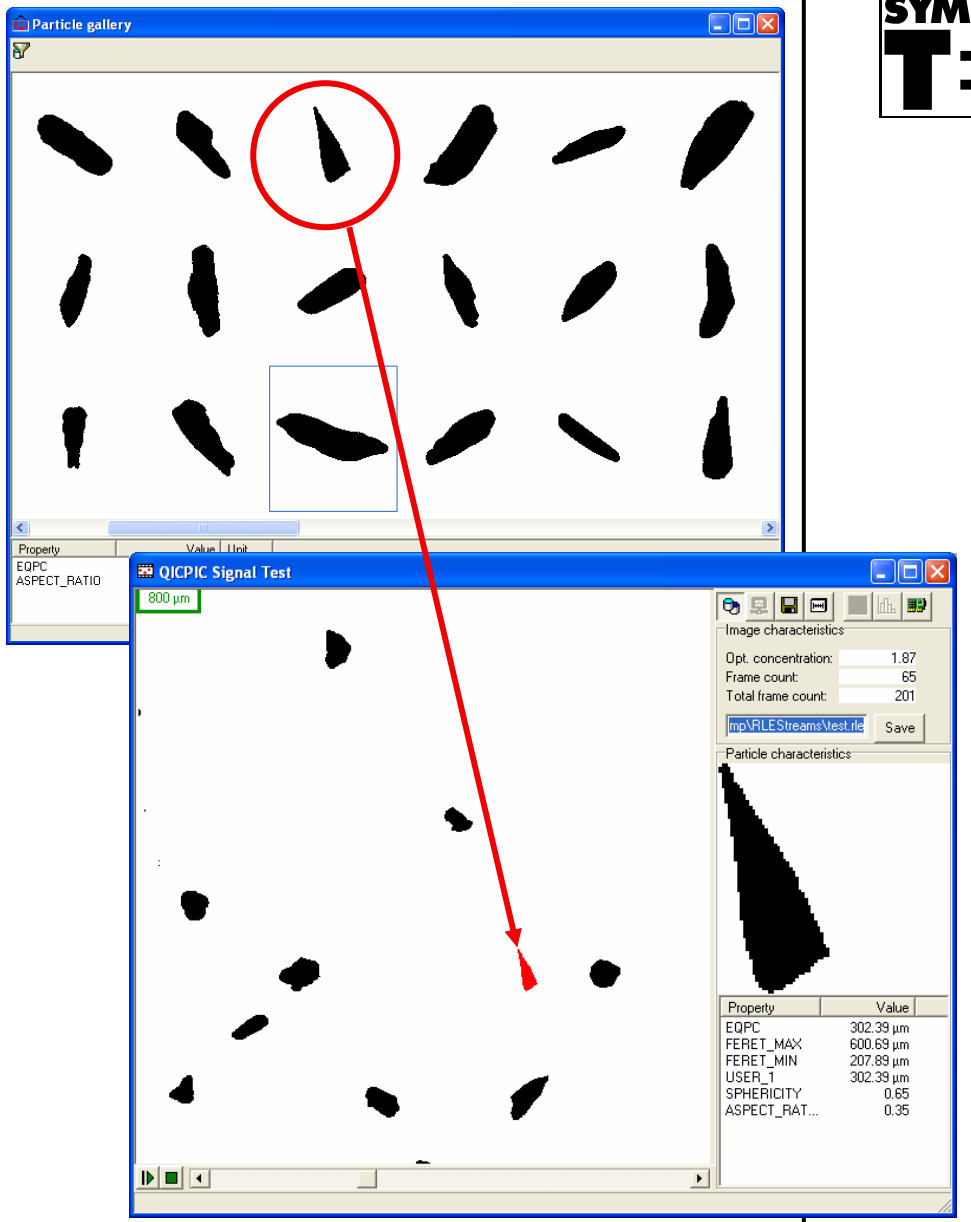


- ✔ Circle of equal projection area
- ✔ Feret Diameter
  - ✳ maximal
  - ✳ minimal
  - ✳ averaged
  - ✳ 90° to maximum
  - ✳ 90° to minimum
- ✔ Minimal area of enclosing rectangle
  - ✳ length
  - ✳ width
- ✔ Chord length
  - ✳ vertical
  - ✳ horizontal
  - ✳ maximal
  - ✳ minimal
  - ✳ 90° to maximum
  - ✳ 90° to minimum
  - ✳ averaged

Particle Gallery

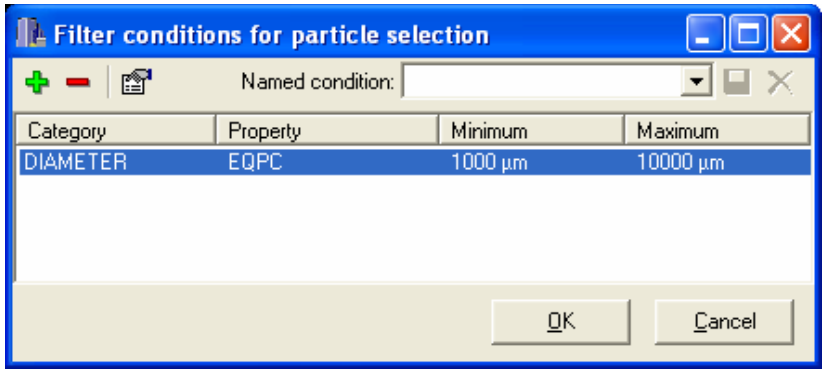
"Looking for a needle in a haystack"

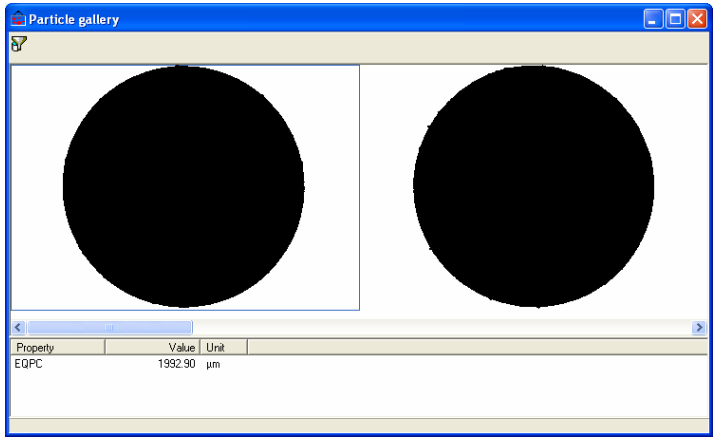




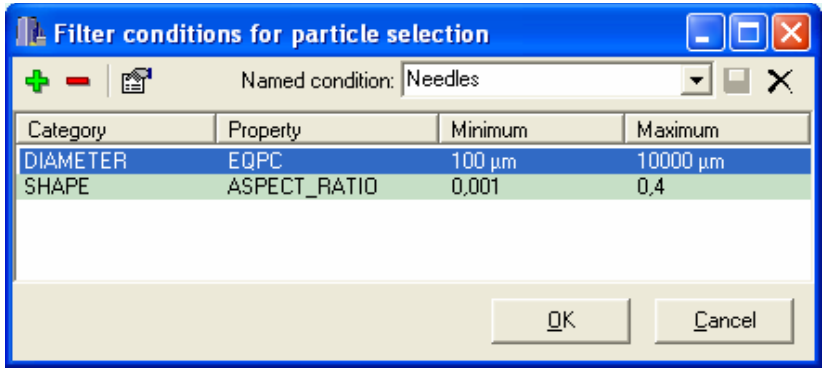
- ✔ Defined filters with *different size*- and *form conditions*
- ✔ *Unlimited number* of particles within the filter conditions are displayed in a *gallery window*
- ✔ *Ability to trace back* to particle movie

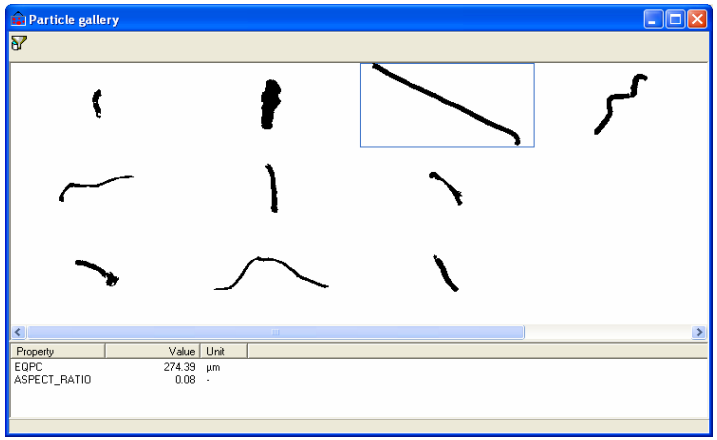
Measuring ball bearing beads





✔ Successful search for *fibres* of the tissue used for cleaning of GRADIS



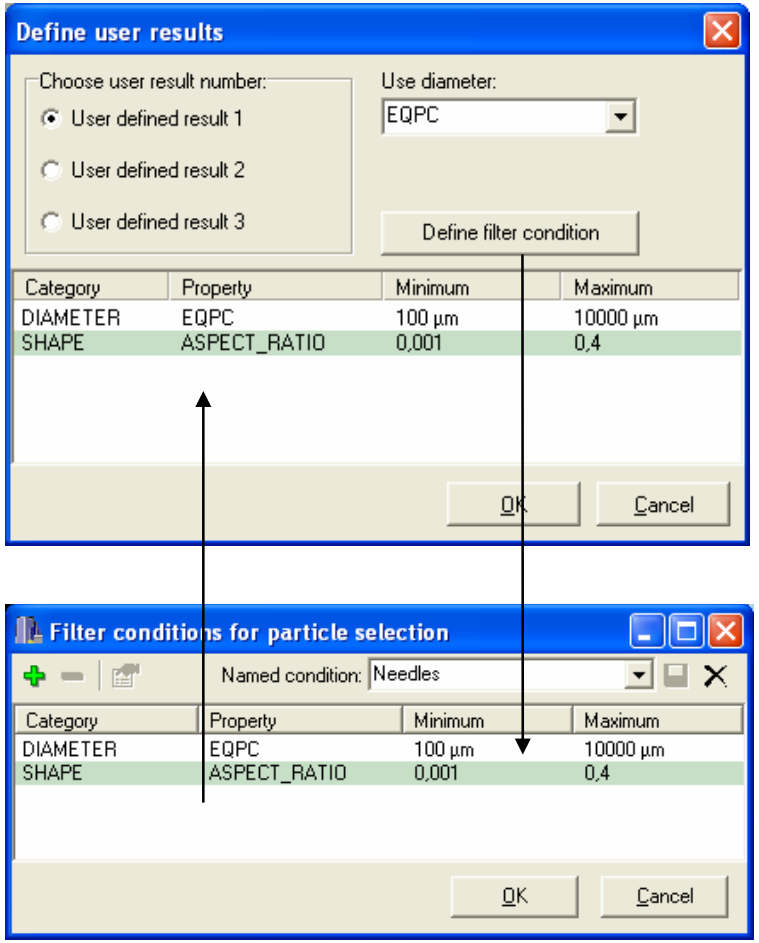


User defined Results

- ★ Considering the *really important fractions* for the evaluation
- ★ Define *new filter conditions* or transfer the filter conditions of the *particle gallery*
- ★ Re-calculate *size* and *shape information* only for particles matching the filter conditions

<u>Examples</u>

- ★ Calculate individual PSDs for the components of a mixture of components that differ in shape
- ★ Calculate the size distribution for perfect spherical particles only
- ★ Eliminate or search for artefacts



Technical Data

Sensor:	QICPIC	1 µm – 20.000 µm
Principle:	Image Analysis	in transmission
Dispersion:	Adaptable modules	for aero-dispersion, sprays, suspensions
Light source:	Pulsed light source	0 to 500 Hz, < 1 ns, 532 nm (green)
	Beam expansion	adaptable to measuring range
Measuring ranges:	Optical modules	3:1 to 1:10, on revolving disk
Measurement:	High speed CMOS camera	1024 x 1024 square pixels, 10 x 10 μm^2 256 grey levels (8 bit)
	Frame rate	up to 500 images/s
Data transmission:	Twisted, two-wire copper cable	1.25 Gbit/s, 5 meters max.
Evaluation:	Particle size	equivalent sphere, enclosing rectangle, cord length, Feret & Martin diameter
	Particle shape	sphericity, as function of particle size
	Class limits	definable, internal 20.000 classes
Visualisation:	Presentation of characteristic particle images	various selection options: movie, gallery
Software & QA:	WINDOX 5	compliant with 21 CFR Rule 11, including electronic signatures

QICPIC Conclusions

- ✔ QICPIC is an innovative measuring system for *particle size* and *shape determination* with *image analysis* in the range of *1 µm* to *20 mm*
- ✔ Extraordinary short exposure time of < 1 ns enables clear images of fastest particles and for the first time the use of approved, *cutting edge dispersing devices*, for dry, wet, spray or inhalation applications
- ✔ High acquisition rate of un-matched 500 images/second guarantees sound particle counts and *superior statistical relevance* of results
- ✔ *Permanent storage* of original information in 20.000 size classes in data base
- ✔ *Effective data compression* w/o loss of data allows retrospective modifications of evaluation and visualisation
- ✔ Various different *size* and *shape factors* are implemented
- ✔ "*Particle Gallery*" or "*Evaluation of User specific Fractions*" offer powerful tools for investigation of specialities
- ✔ WINDOX 5 software with *client/server structure* and to speed multi-user data base accomplishes *stunning data volume* and manages simultaneous operation with *Laser Diffraction*, *Ultrasonic Extinction* and *Photon Cross Correlation sensors*