Introduction to Image Analysis

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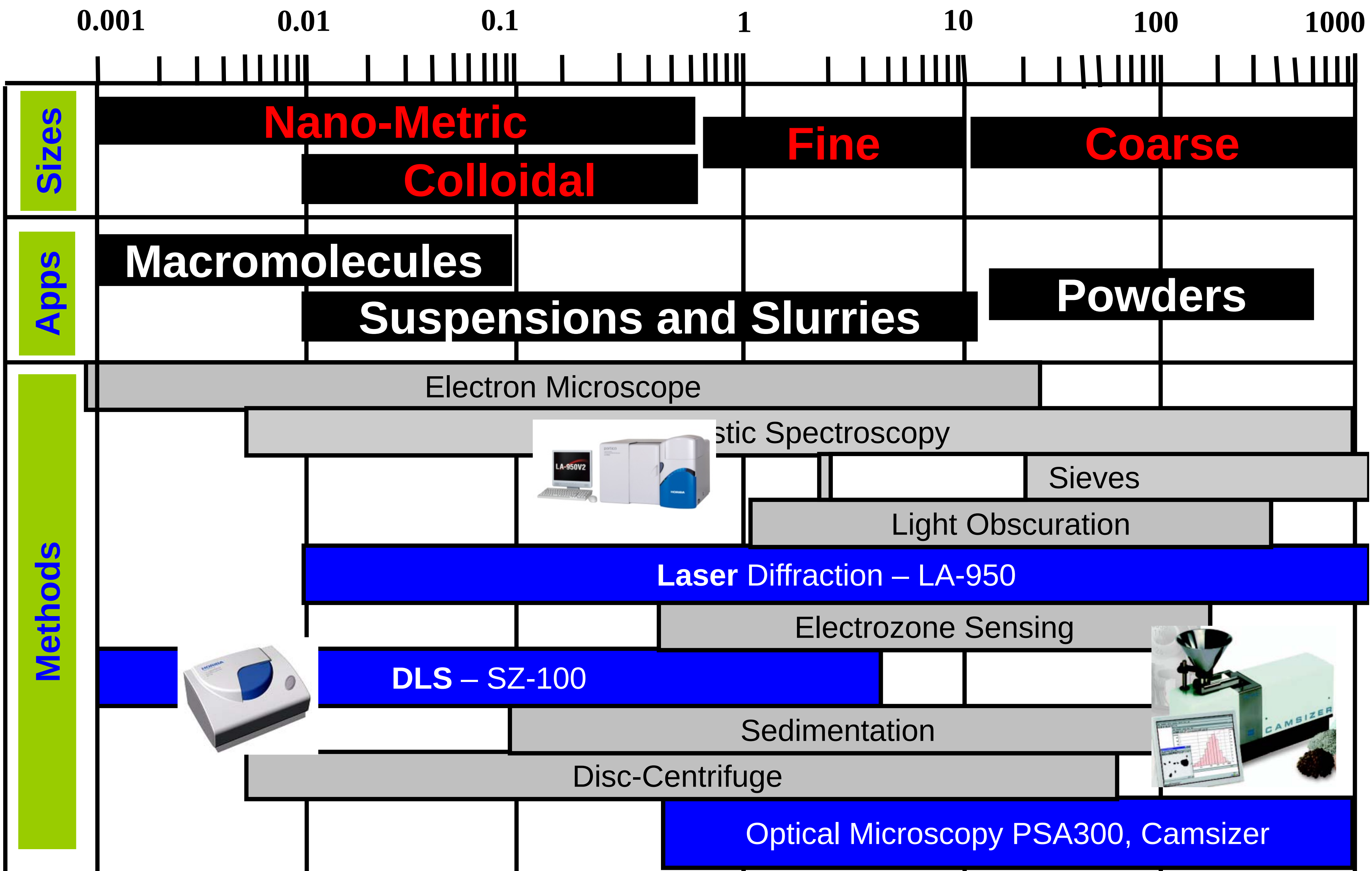
HORIBA Scientific
www.horiba.com/us/particle

Size: Particle Diameter (µm)



Why image analysis?

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- Replace sieves (really!)
- Verify/supplement laser diffraction results (orthogonal technique).
- Need shape information, for example due to importance of powder flow

These may have the same size (cross section), but behave very differently.

Why image analysis?

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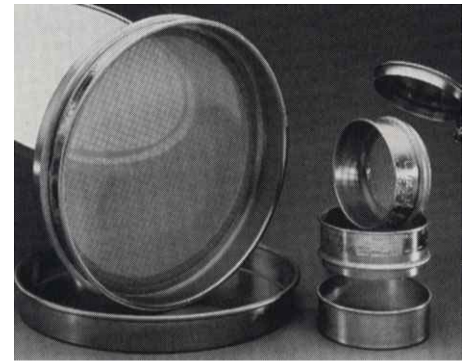
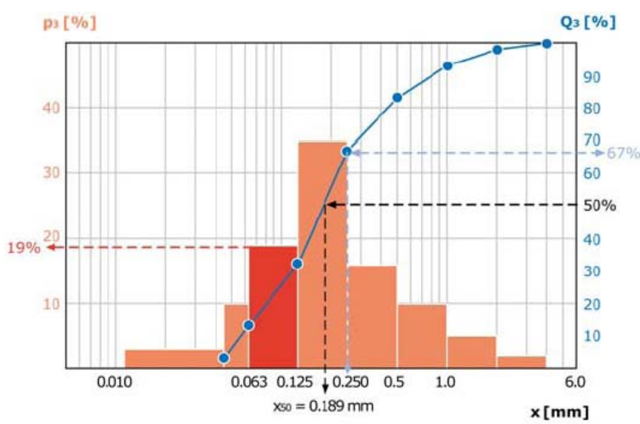
Replace Sieves

- Tend to wear over time. It is difficult to tell when sieve results are "drifting" due to wear
- Results depend on nature of shaking and loading leading to operator to operator variations in results.
- Small number of size classes





Size class [mm]			p_3 [%]	Q_3 [%]
	<	0.045	3.0	3.0
0.045	-	0.063	10.0	13.0
0.063	**-**	**0.125**	**19.0**	**32.0**
0.125	-	0.250	35.0	67.0
0.250	-	0.500	16.0	83.0
0.500	-	1.000	10.0	93.0
1.000	-	2.000	5.0	98.0
2.000	-	4.000	2.0	100.0
> 4.000			0.0	100.0

x_{50} = 0.189 mm

More information available through www.retsch.com

Why image analysis?

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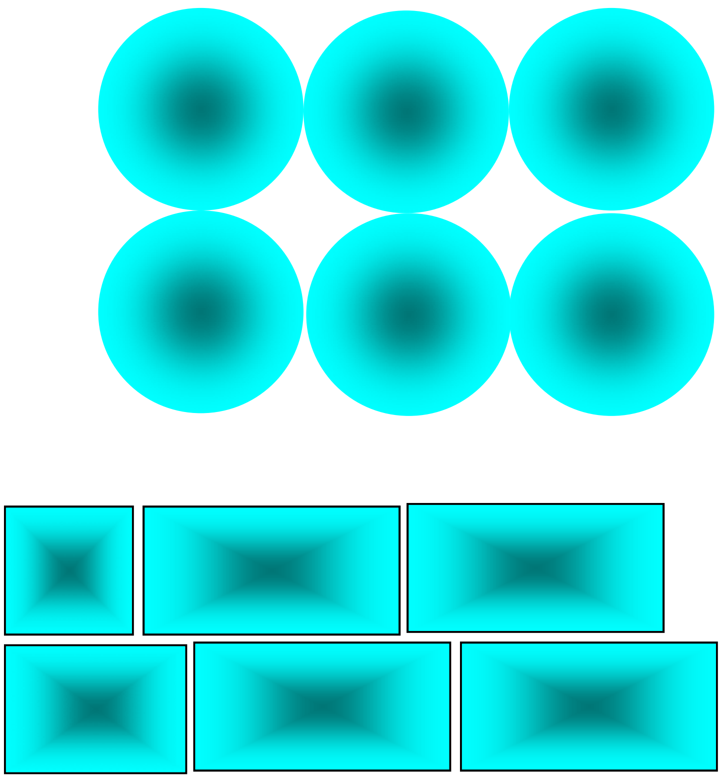
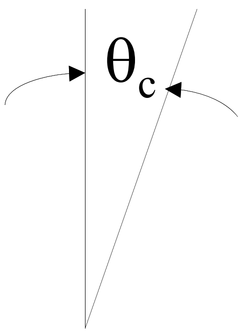
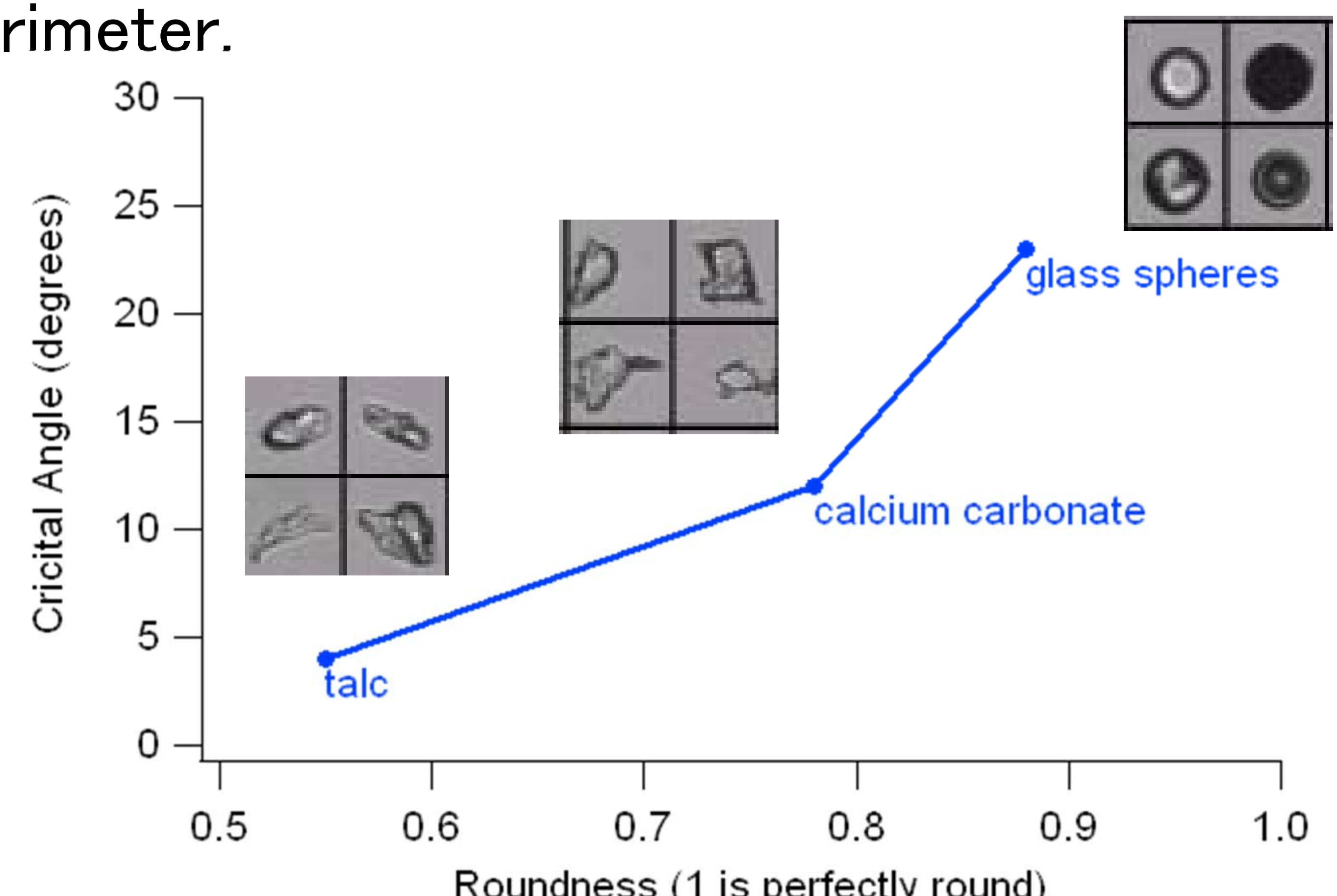
Need shape information for evaluating packing and flow.



Effect of Shape on Flow

- Yes, I assumed density doesn't matter.
- Roundness is a measure based on particle perimeter.





Why image analysis?

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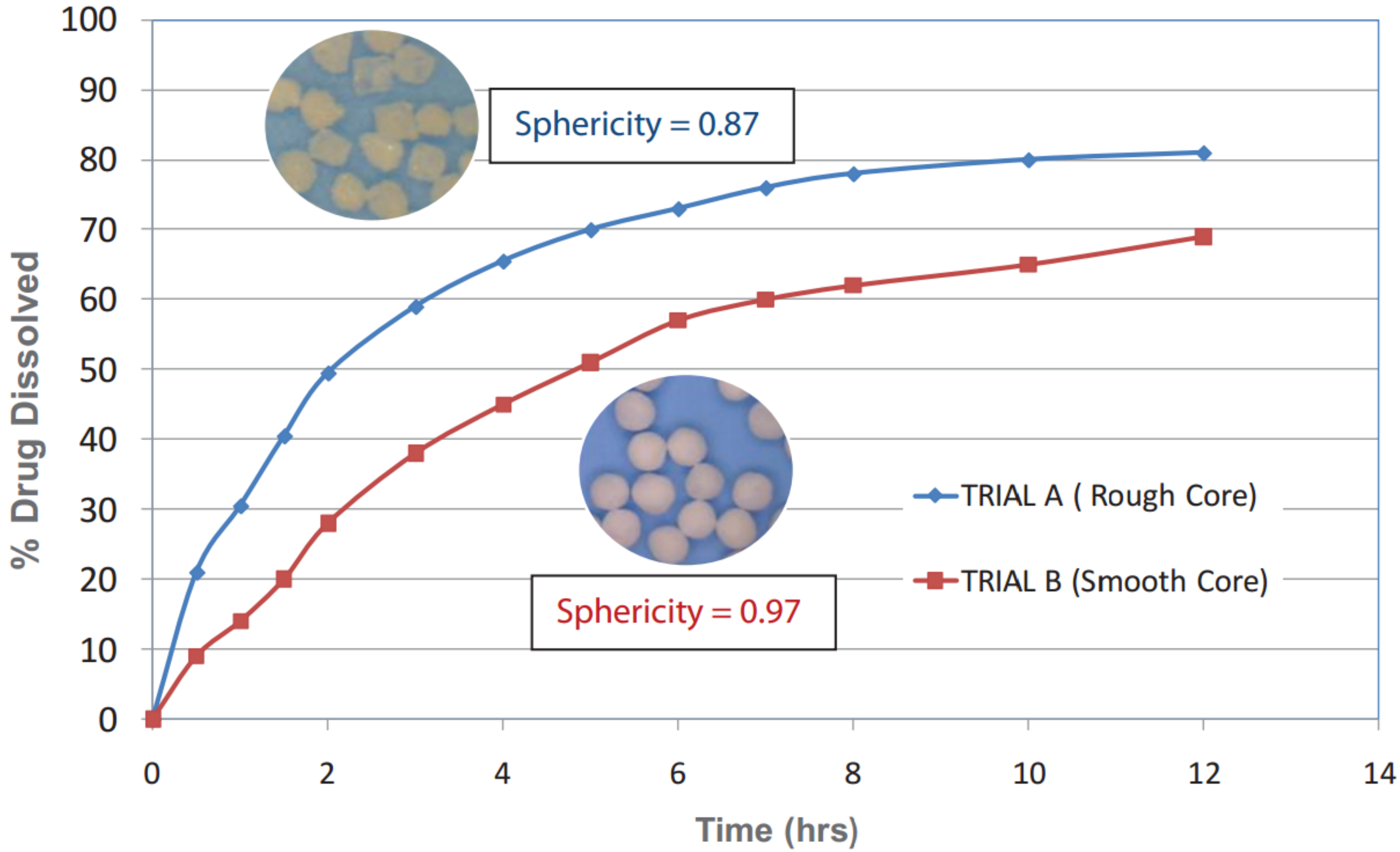
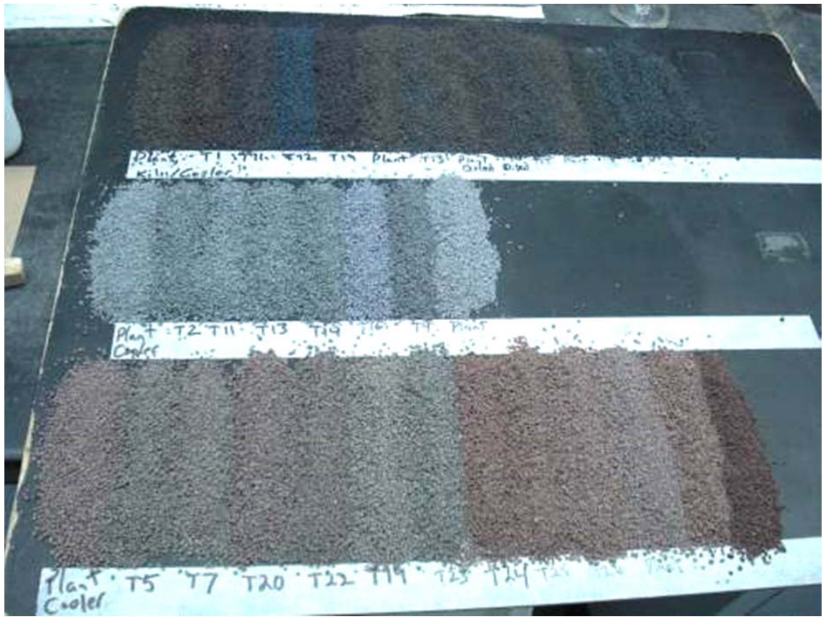
Shape affects drug release profile



Why image analysis?

Shape is important for roofing granules that block sunlight from reaching next layer.



Why image analysis?

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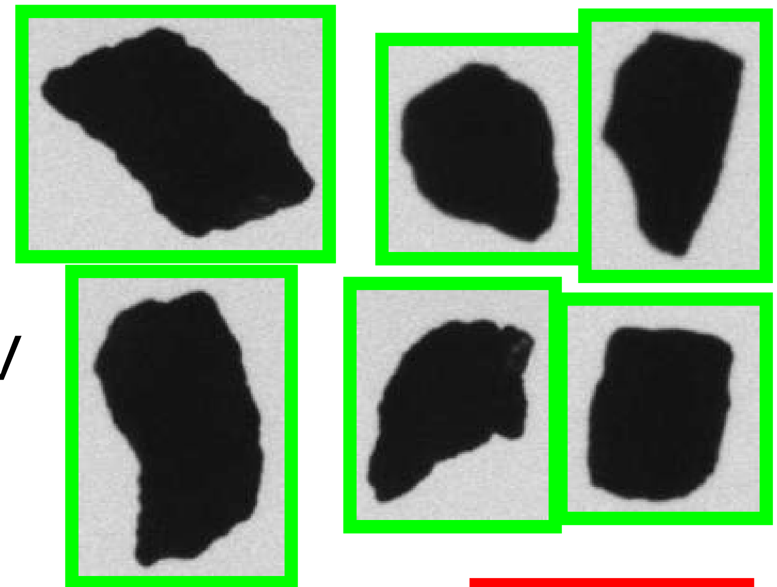
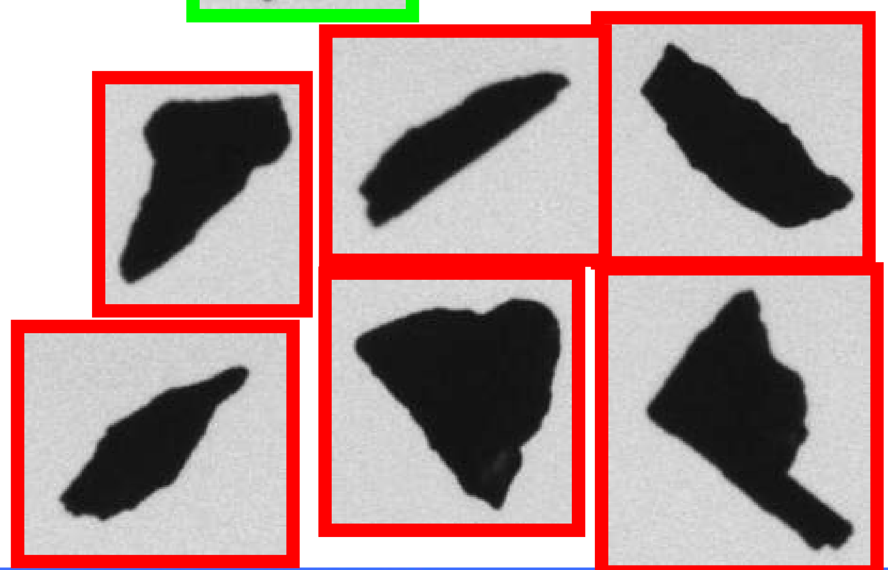
Good Product



Compact particles that fully block UV

BAD Product



Flaky, angular, leaves gaps for UV to pass

Why image analysis

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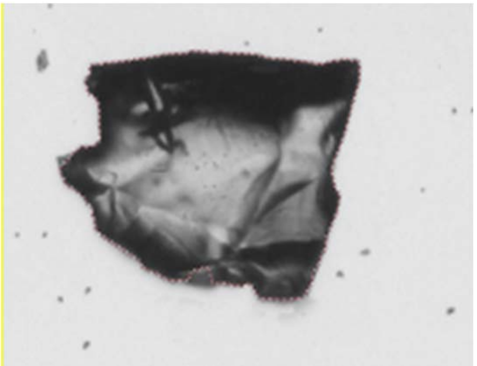
- Pictures: contaminants, identification, degree of agglomeration
- Screen excipients, full morphology
- Root cause of error (tablet batches), combined w/other techniques
- Replace manual microscopy



Major Steps in Image Analysis

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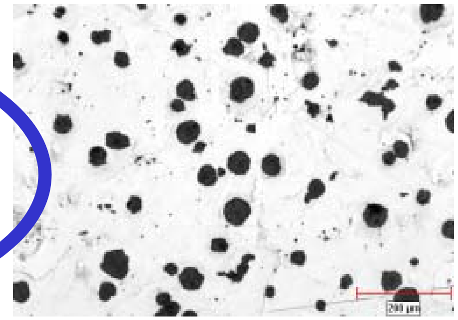
Image Acquisition and enhancement

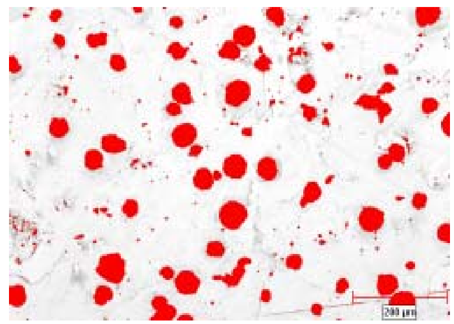
↓

Object/Phase detection

↓

Measurements





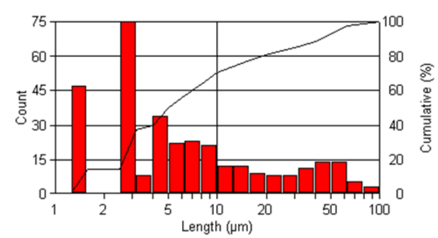


Image Analysis

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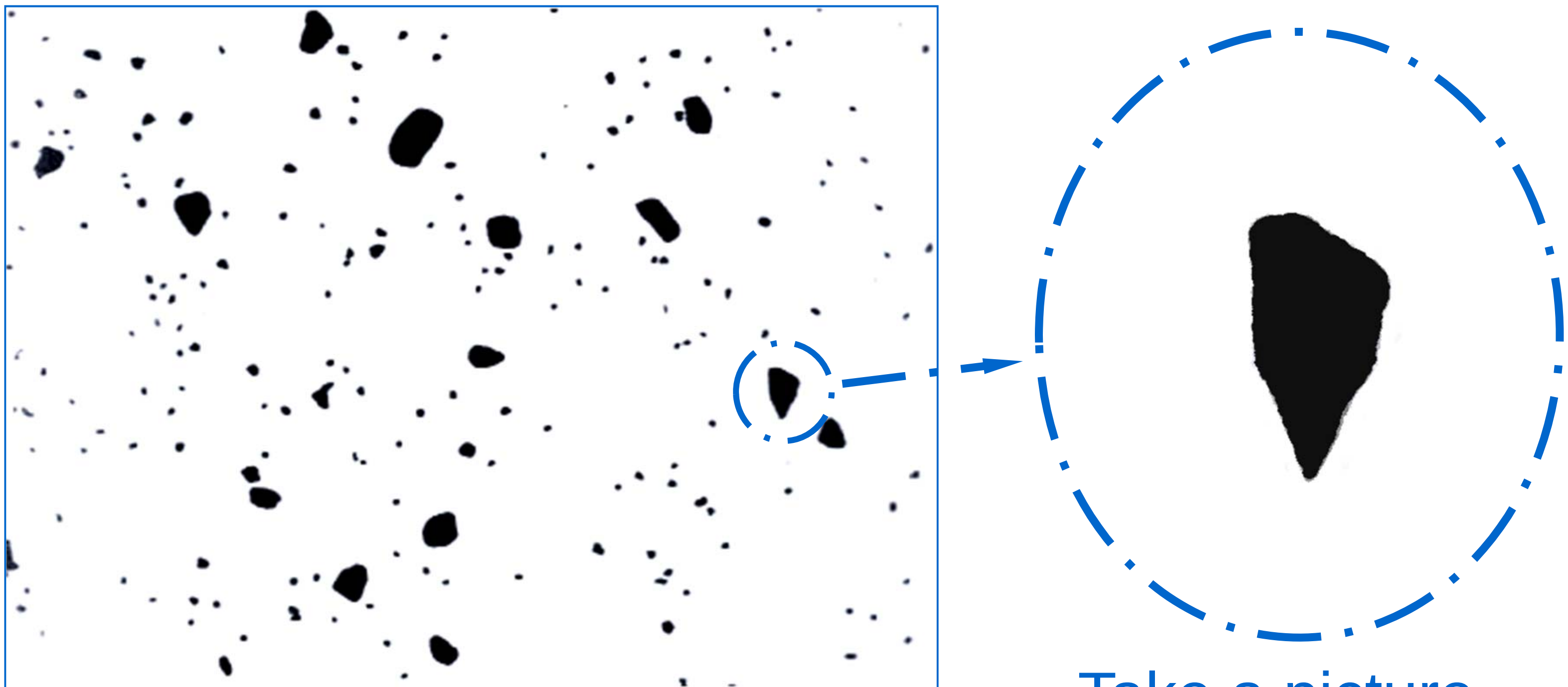

Take a picture,
analyze for size

Static Image Analysis

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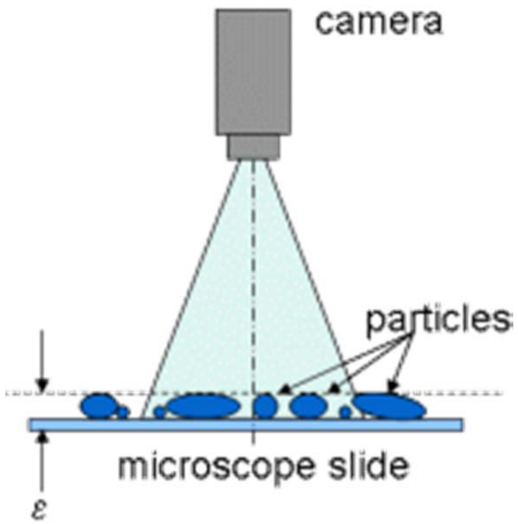
- Particles are dispersed (isolated) on a surface
- Picture are taken from stationary particles
- Camera or surface with particles is shifted, multiple images are taken from different positions, images are processed and evaluated
- High resolution images is possible
- Number of images/particles is limited (because of time limitations)
- Preferred orientation of the particles on the surface (largest 2D)



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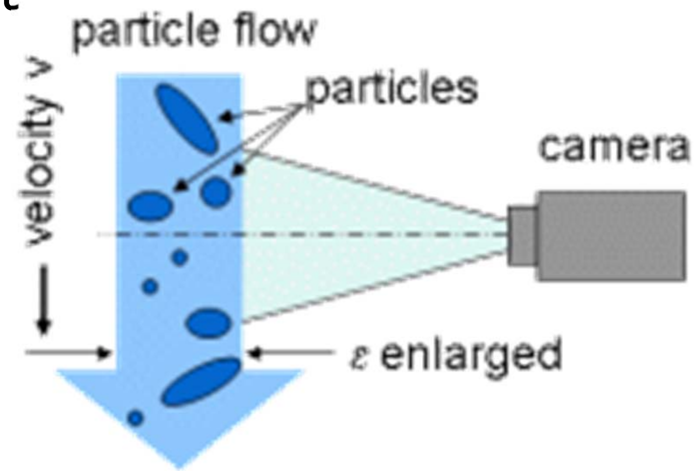
Dynamic Image Analysis

- Particles flow through the measurement volume of the instrument and the field of view of the camera
- Particles are captured during movement, no other moving parts necessary
- Capturing of many particle images in a short time interval
- Limitations because of image rate of the camera(s)
- Image quality is (a bit) worse
- Particles are projected in random orientation (3D)



CAMSIZER

Resolution

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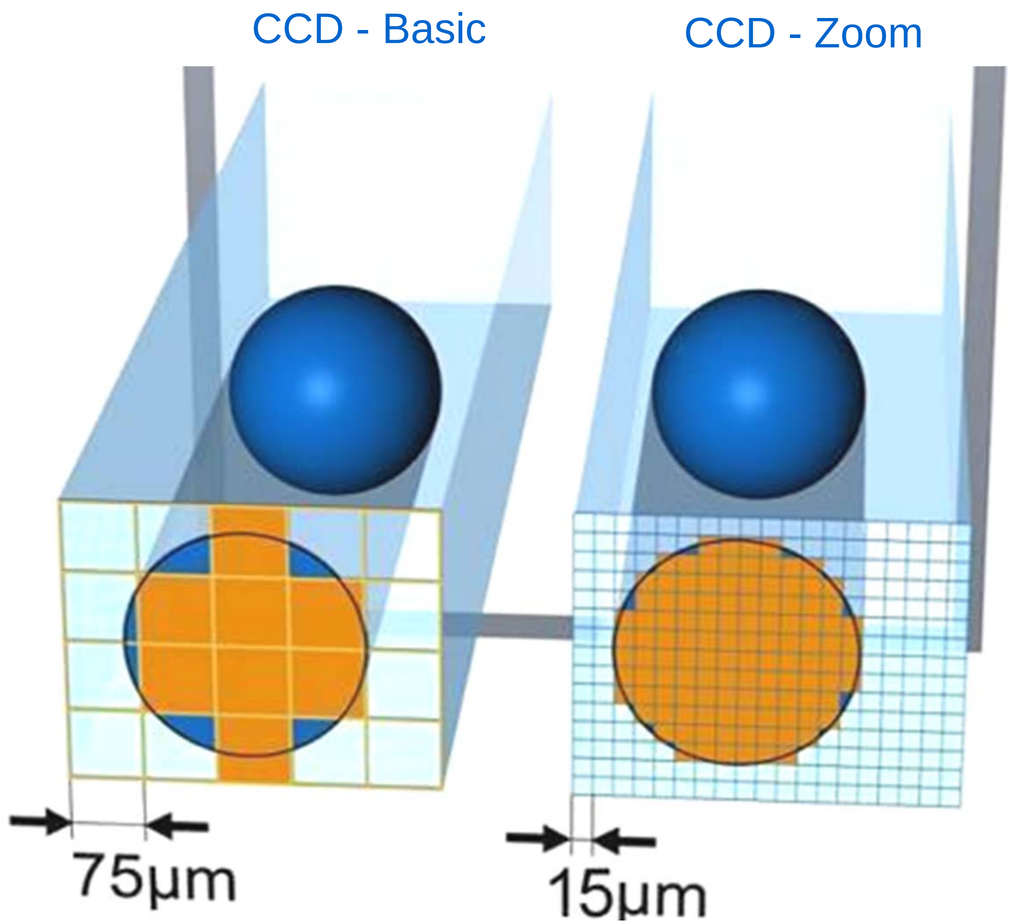
Detection of particles

One pixel is element of a projection when at least half of the pixel is covered.



CAMSIZER

CAMSIZER XT 14µm 1µm

Maximum size

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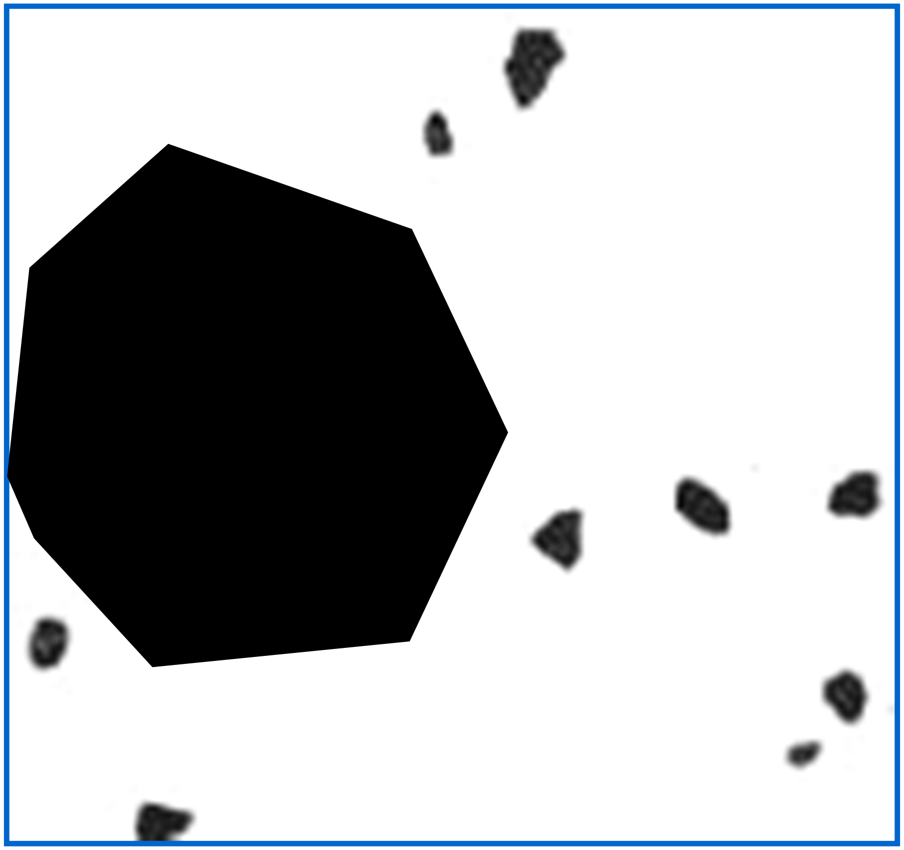
Large particles cannot been measured properly even they fit in the frame.

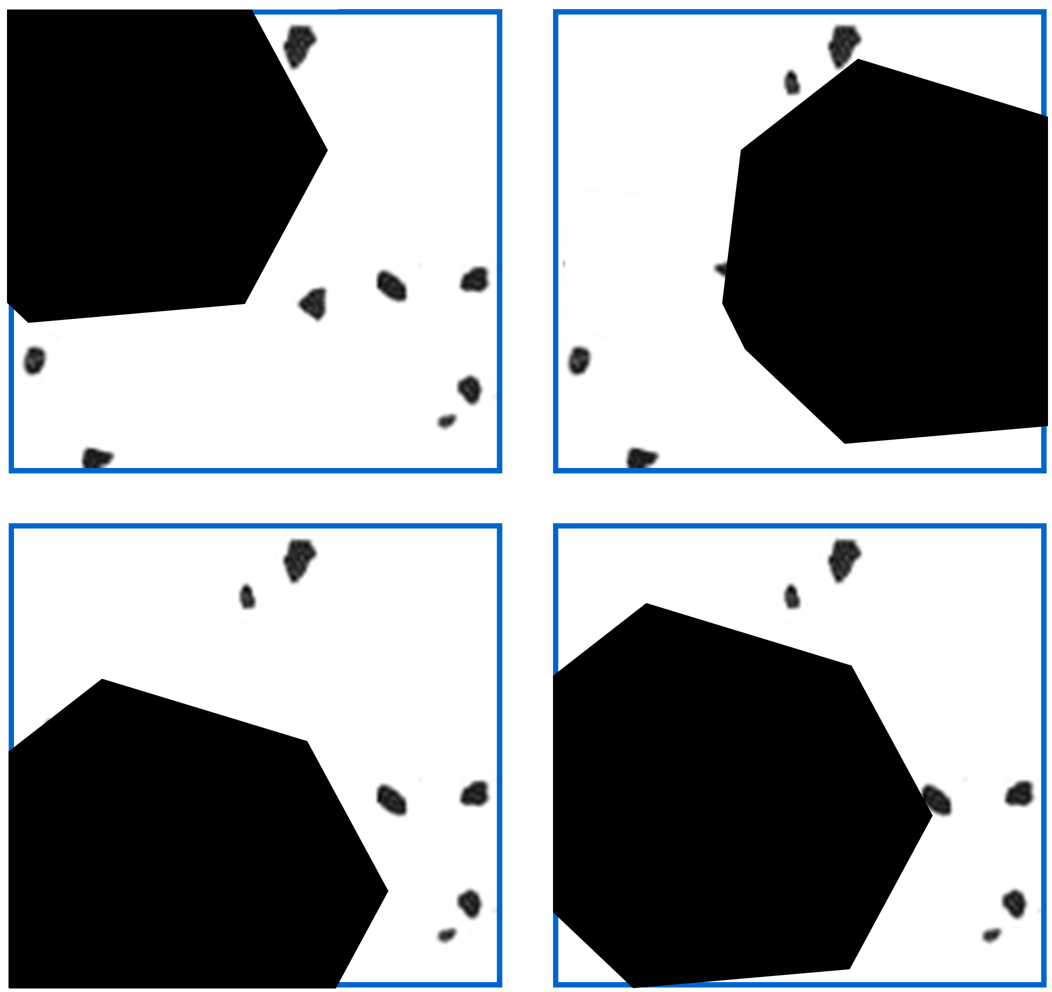


Maximum size

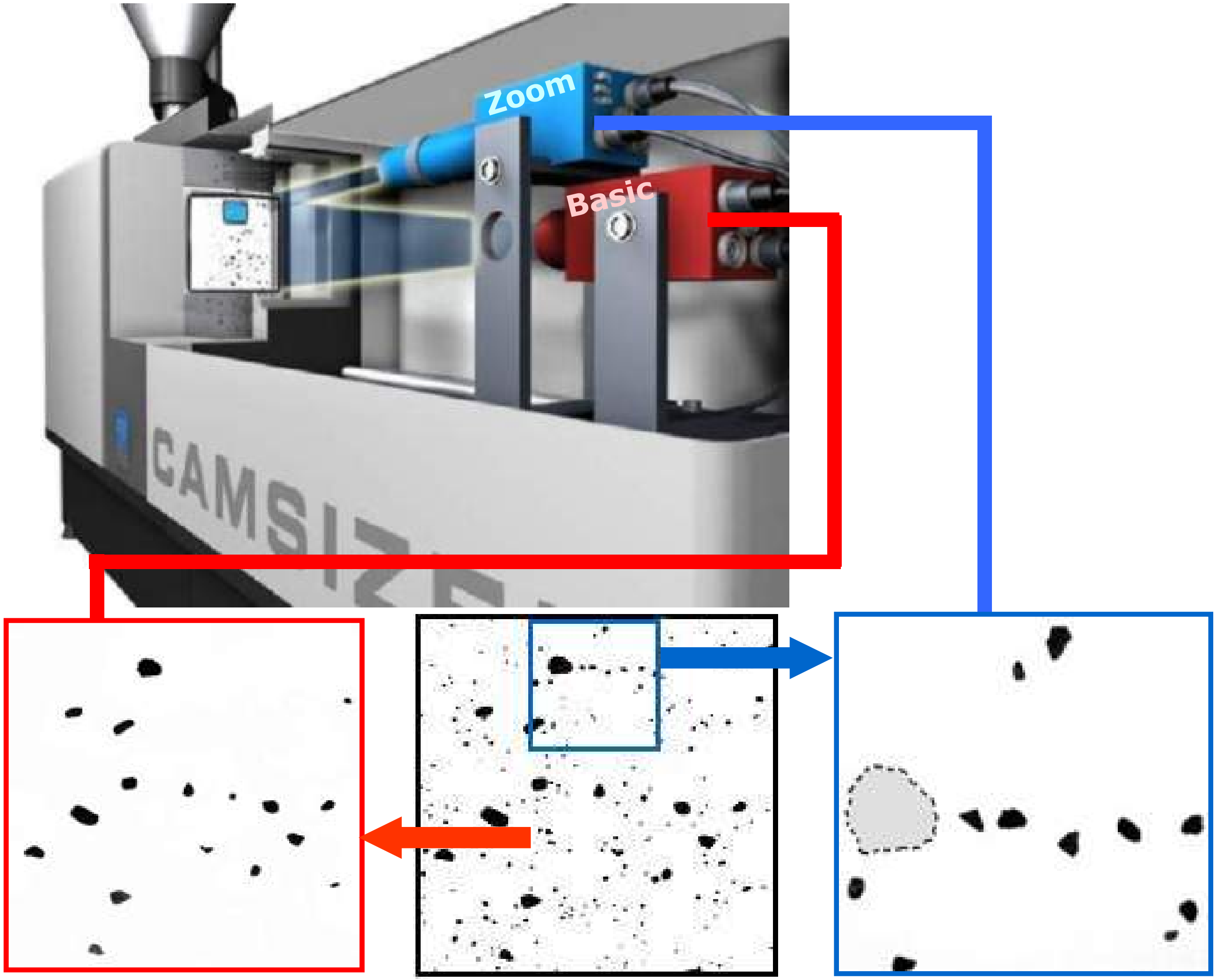
The probability of large particles touching the edge of the frame is higher than for smaller particles.

=> Large particles cannot been measured sufficiently

Upper limit of measurement range



Two-Camera-System



Basic-Camera

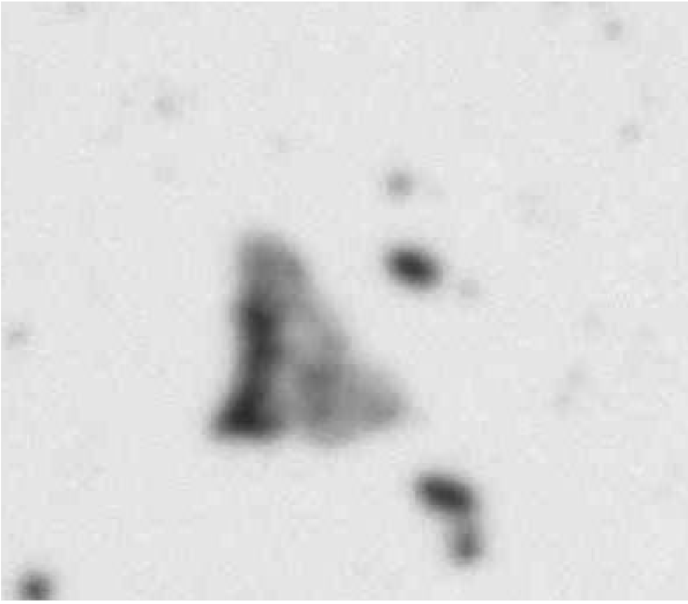
Zoom-Camera

Acquiring Images

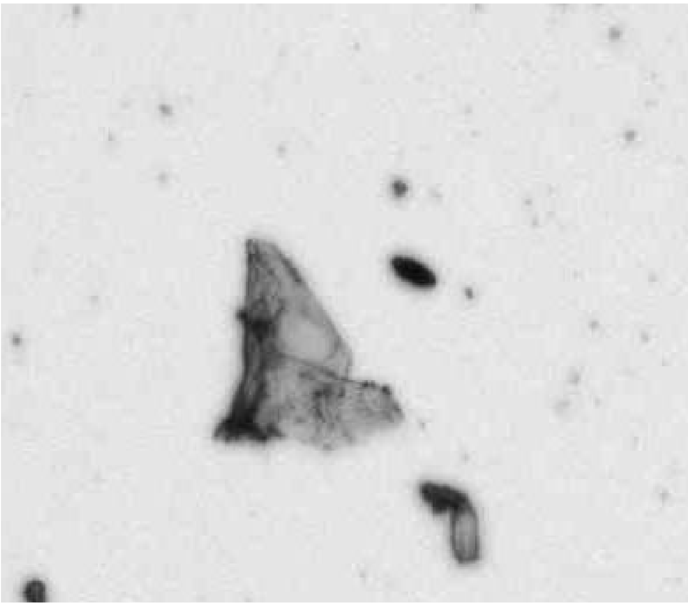
We want a good microscope and nice sharp images.

Pay attention to lighting and focus.

No



Yes



Multilayer Grab for Sharpness

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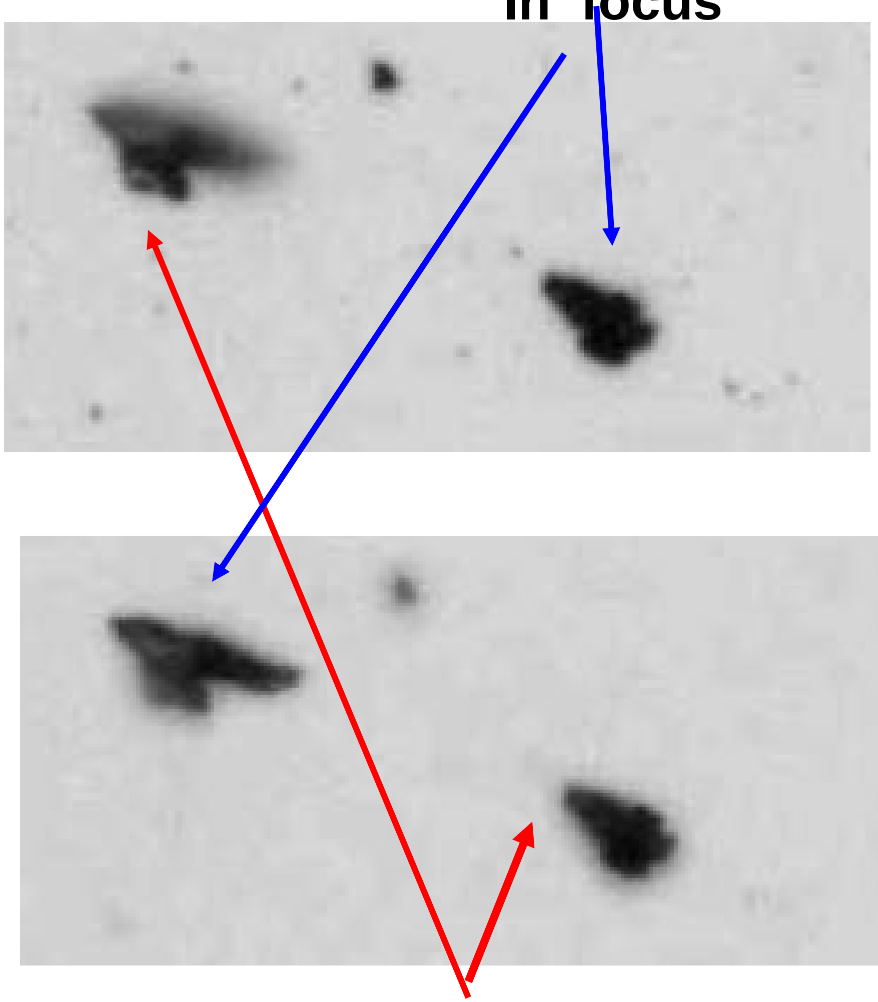
In focus



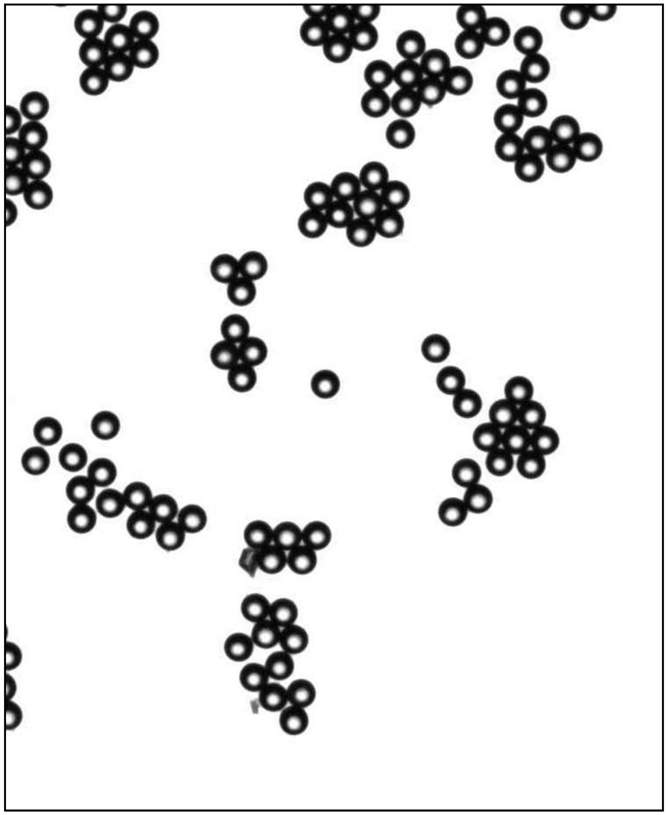
Out of focus

Stack images for sharper final image

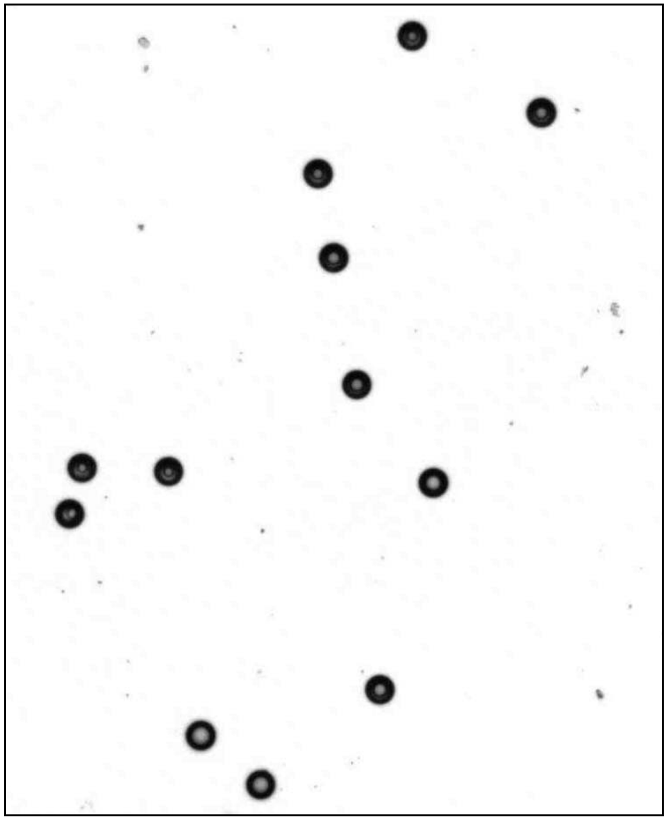
Dispersing a Sample

Want to spread particles out so that they don't touch.

No



Yes



Control feed rate.

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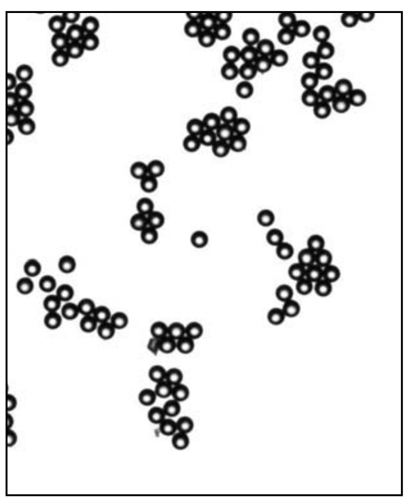
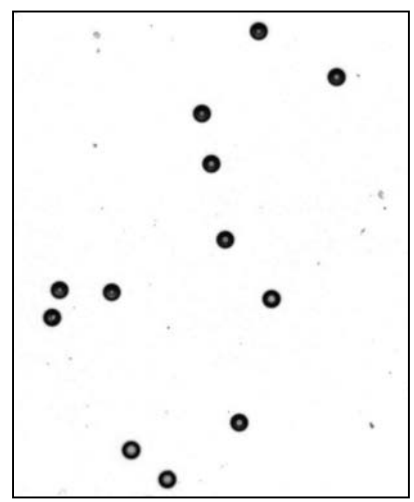
Want to spread particles out so that they don't touch.

Use % of field of view that is covered in order to control feed rate. Try 1% at first.

Feeding Too fast



Good



Major Steps in Image Analysis

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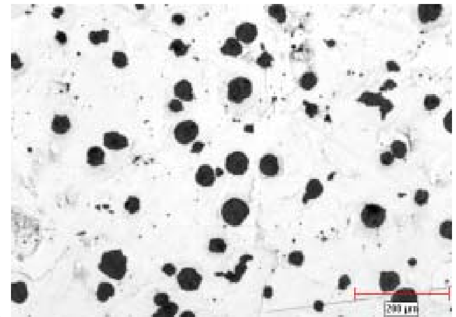
Image Acquisition and enhancement

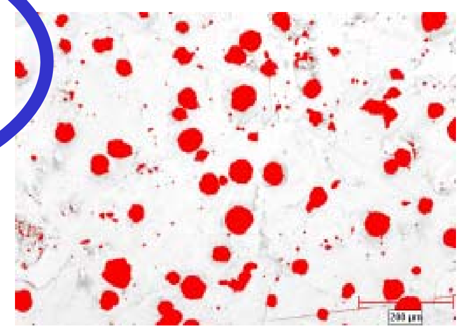
↓

Object/Phase detection

↓

Measurements





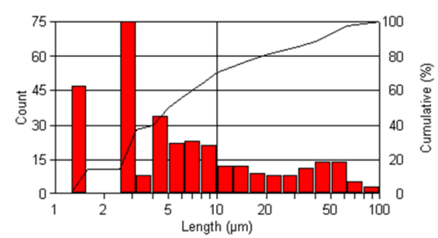
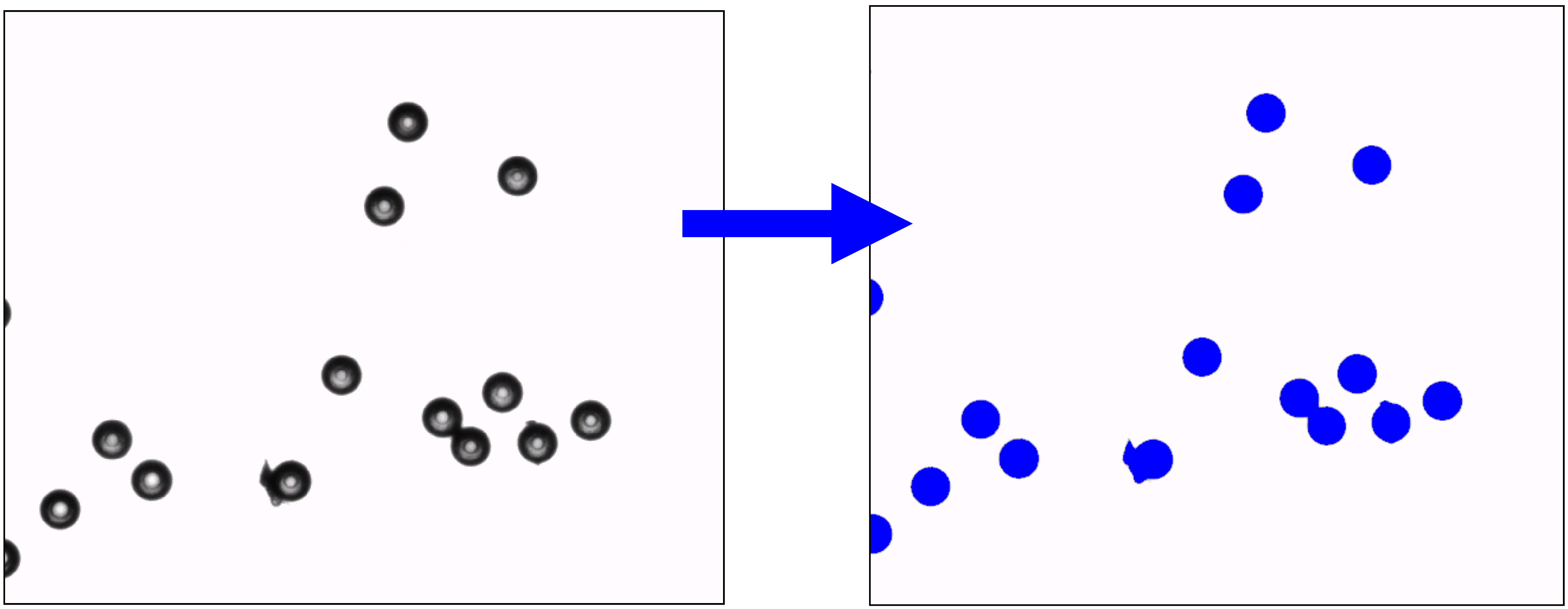


Image Binarization

Turn into binary image (i.e., decide what is a particle and what isn't).



Separation

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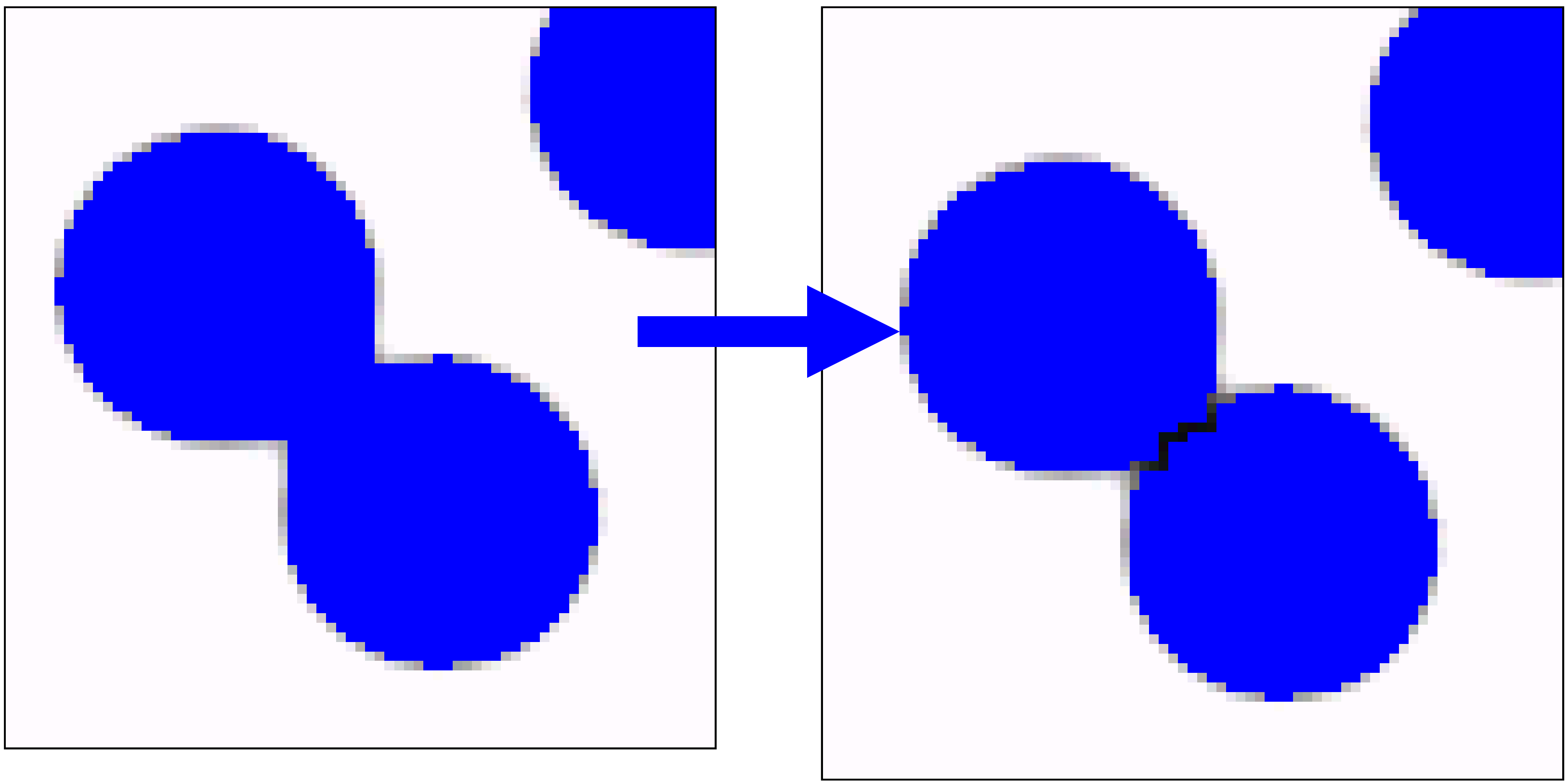
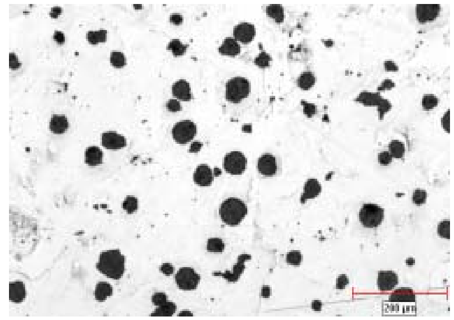


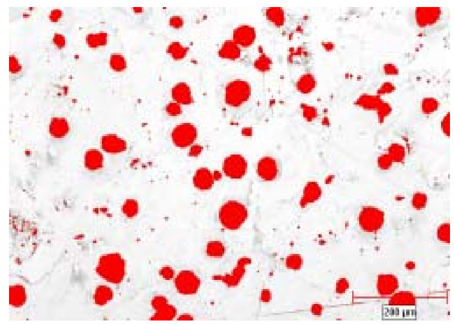
Major Steps in Image Analysis

HORIBA Scientific

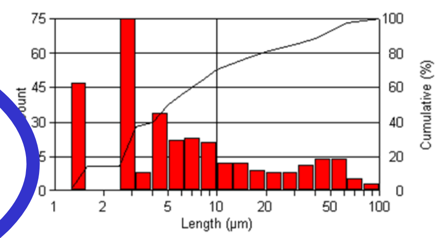
Image Acquisition and enhancement



Object/Phase detection



Measurements



Many Size Measures

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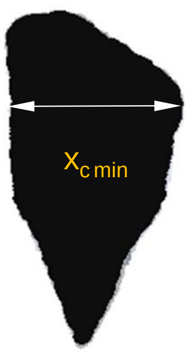
$x_{c\,min}$

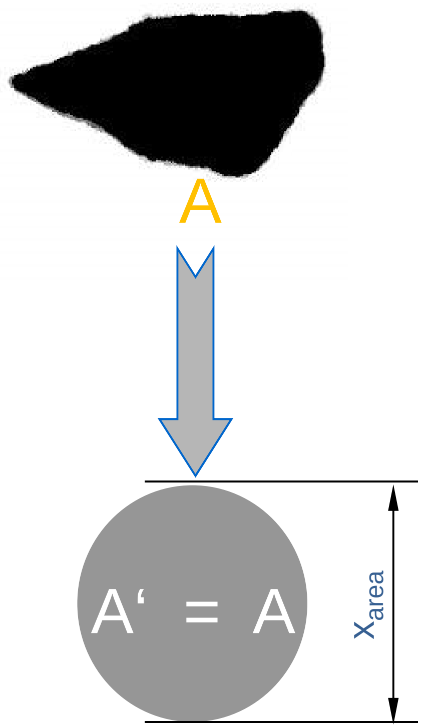
"width"



Width is best suited for comparison with sieves !

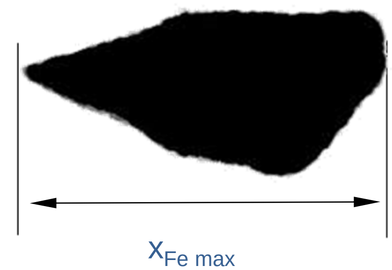
x_{area}

"diameter over projection surface"



$x_{Fe\,max}$

"length"



Shape parameters can be calculated!

Comparison of Size Definitions

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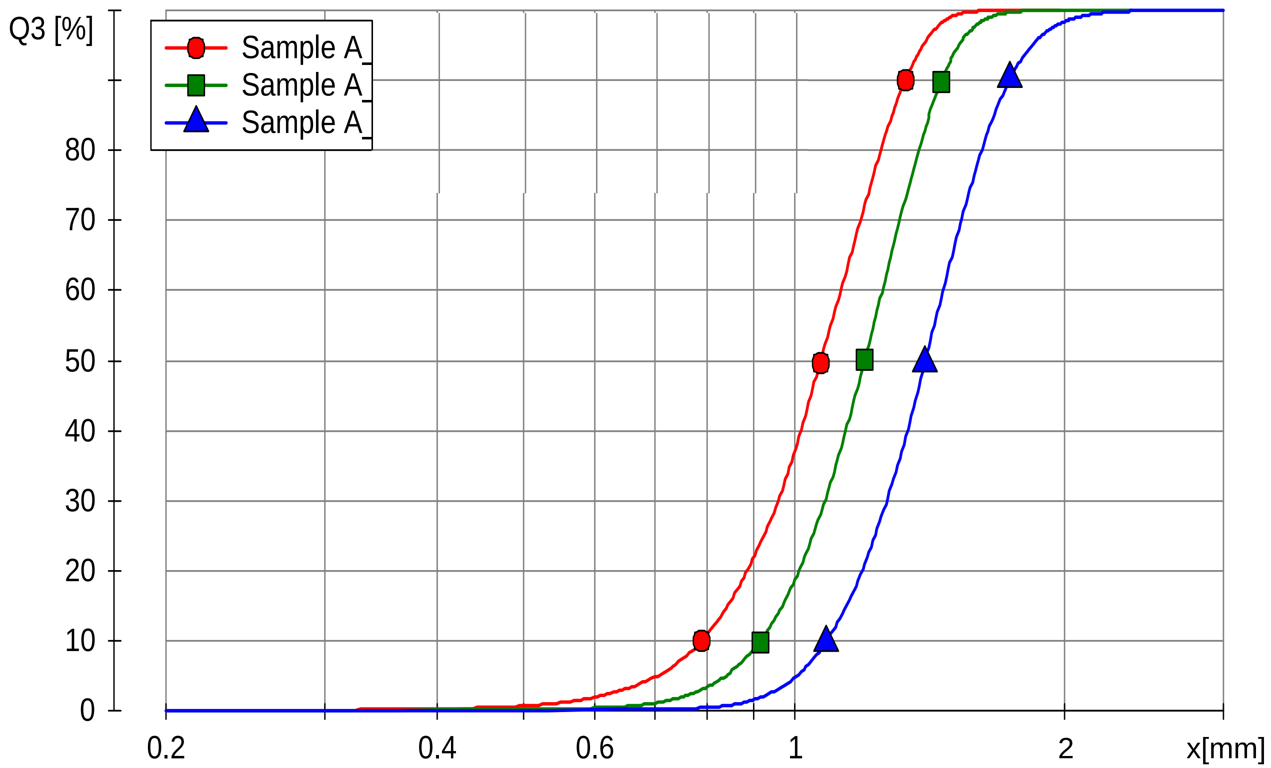
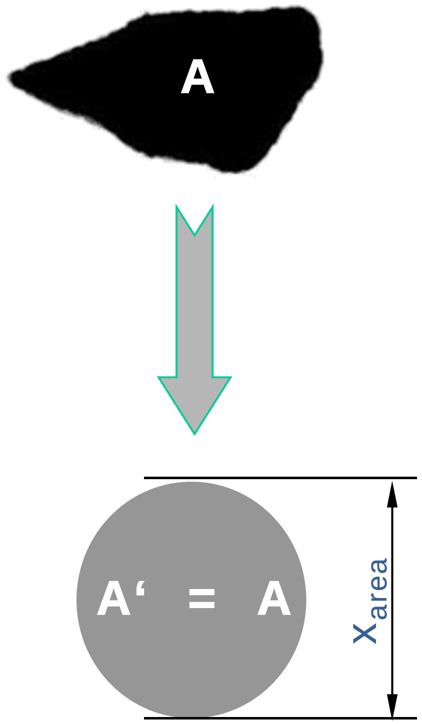
$x_{c\,min} \Leftrightarrow x_{Area} \Leftrightarrow x_{Fe\,max}$



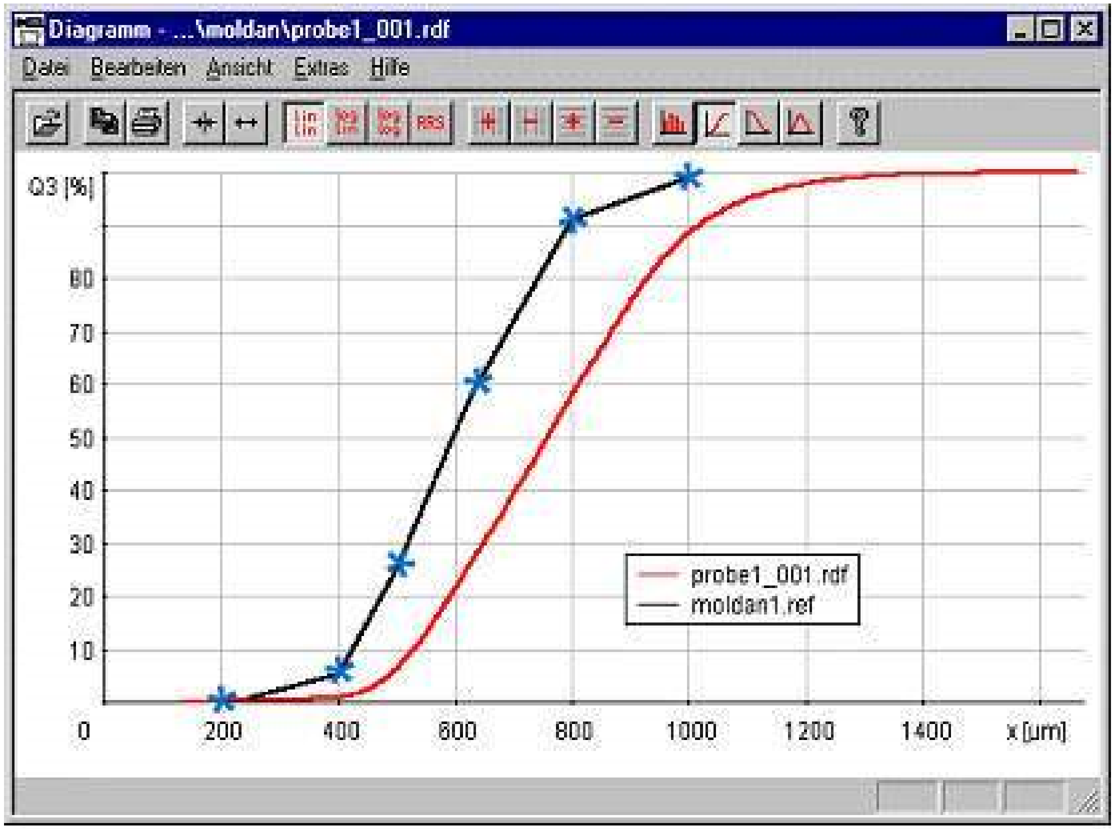
different size definitions ⇔ different results

Digital Image Processing
Area Measurement ⇔ Sieving



x_{area}

"diameter
via projection surface"



comparison

CAMSIZER-measurement x_{area} (**red**)
and sieving * (**blue**)

Digital Image Processing

Measuring of Width ⇔ Sieving

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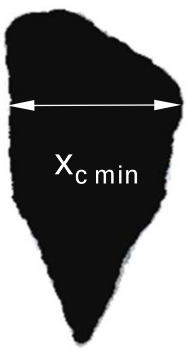
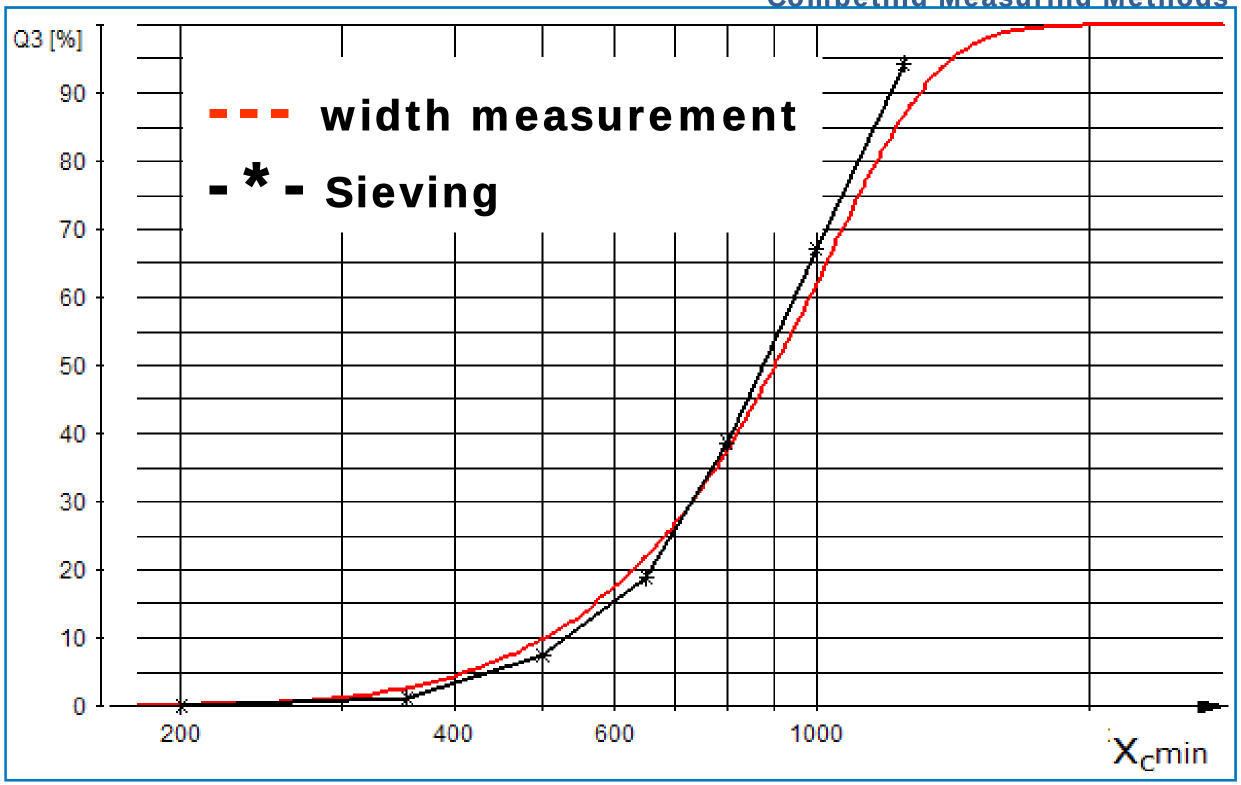
$X_{c\,min}$

"width"



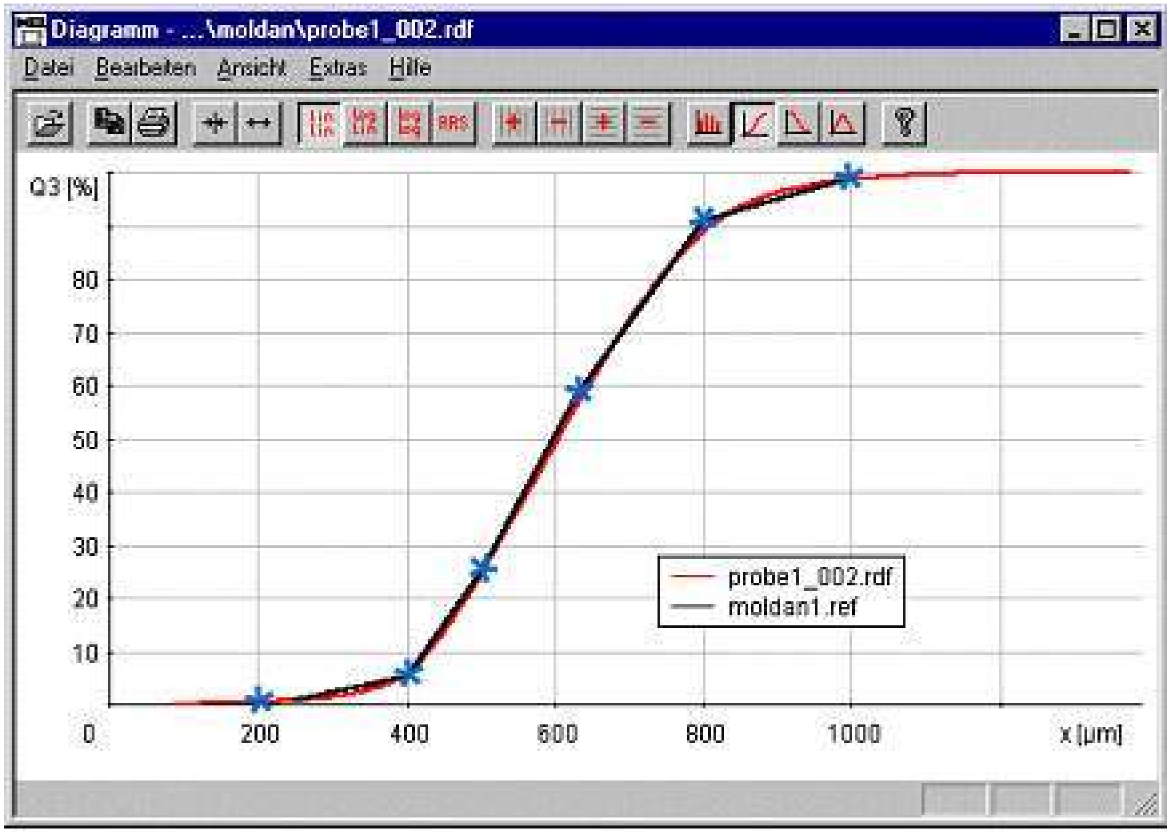
Competing Measuring Methods



comparison

CAMSIZER-measurement **$x_{c\,min}$** (**red**)
and sieving * (**black**)

Fitting of CAMSIZER results to Sieving



fitted result

CAMSIZER-measurement x_{area} (**red**)
to sieving * (**blue**)

Shape: Aspect Ratio

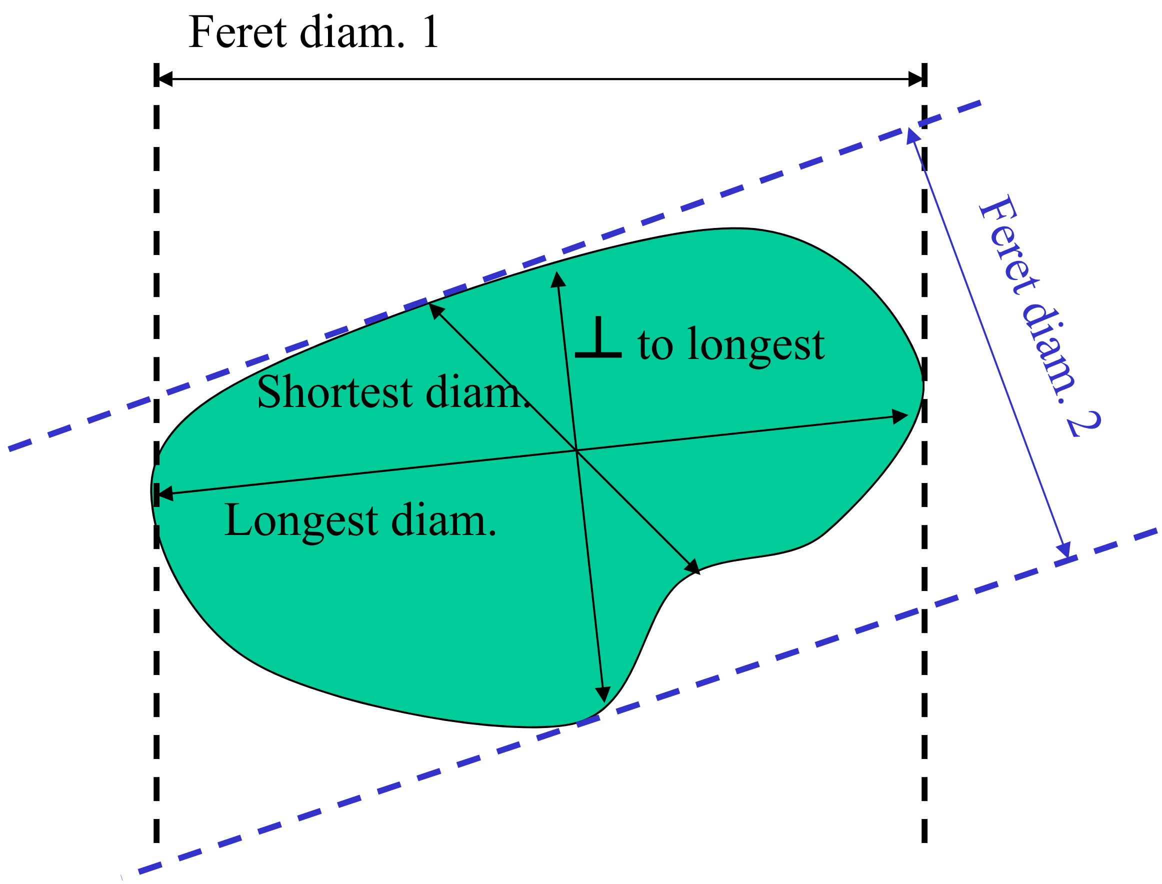
Aspect ratio

$= \frac{\text{shortest diam}}{\text{longest diam}}$

$= \frac{\perp \text{ to longest diam}}{\text{longest diam}}$

$= \frac{\text{shortest Feret diam}}{\text{longest Feret diam}}$

= three different numbers!



More Shape Descriptors

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Roundness

A shape measure that quantifies the "roundness" of an object's edges:

$$\frac{4 \times \text{Area}}{(\pi \times \text{L} \times \text{L})}$$

Compactness

Ratio of area over convex perimeter:

$$\frac{4\pi \text{ A}}{\text{Convex perimeter}^2}$$

Roughness

A shape measure that quantifies the jaggedness of an object's edges:

$$\frac{\text{Convex perimeter}}{\text{Perimeter}}$$

Fractal Dimension

Numerical characterization of irregular contours through fractal geometry.

$$P = P_e \delta^{1-D}$$

D is the Fractal Dimension, d is the unit length of the scale used for the measurement and P is the perimeter of the object (1<D<2).

Aspect Ratio

Ratio of length over width.

$$\frac{\text{Length of longest feret}}{\text{Length of shortest feret}} = \frac{\text{Length}}{\text{Width}}$$

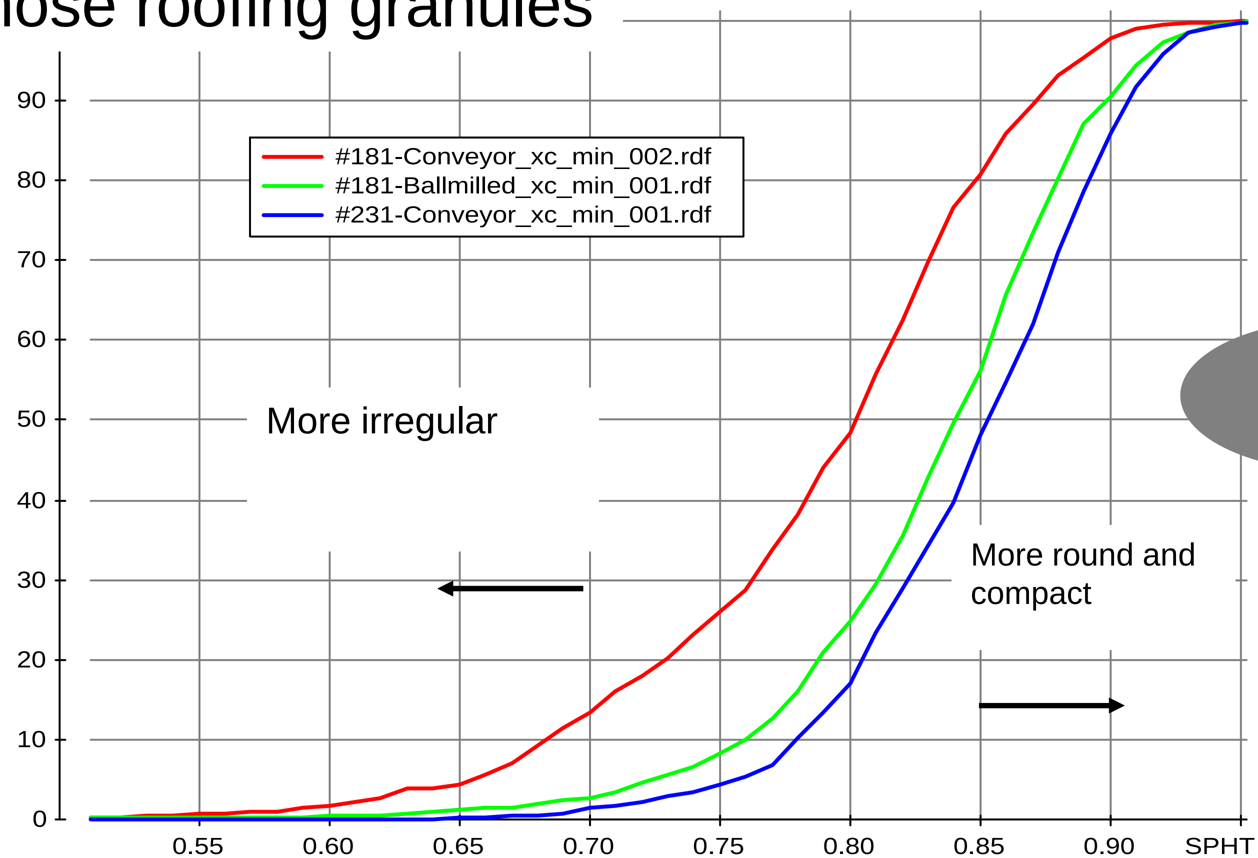
Sphericity

Estimate of the sphericity of an object:

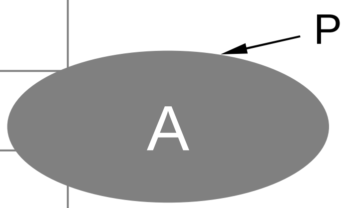
$$\frac{4\pi \text{ A}}{\text{p}^2}$$

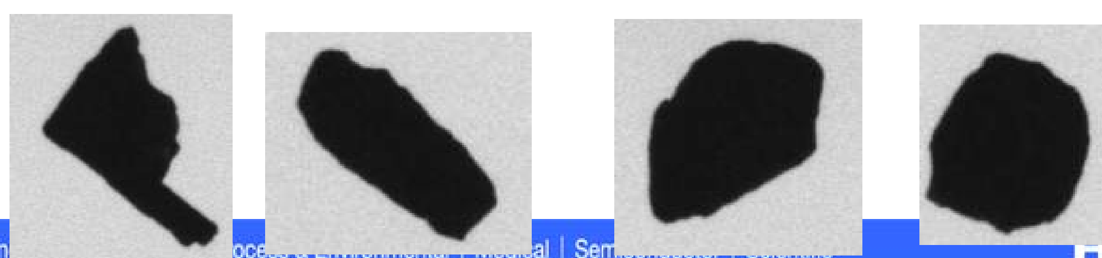
Shape analysis

Back to those roofing granules

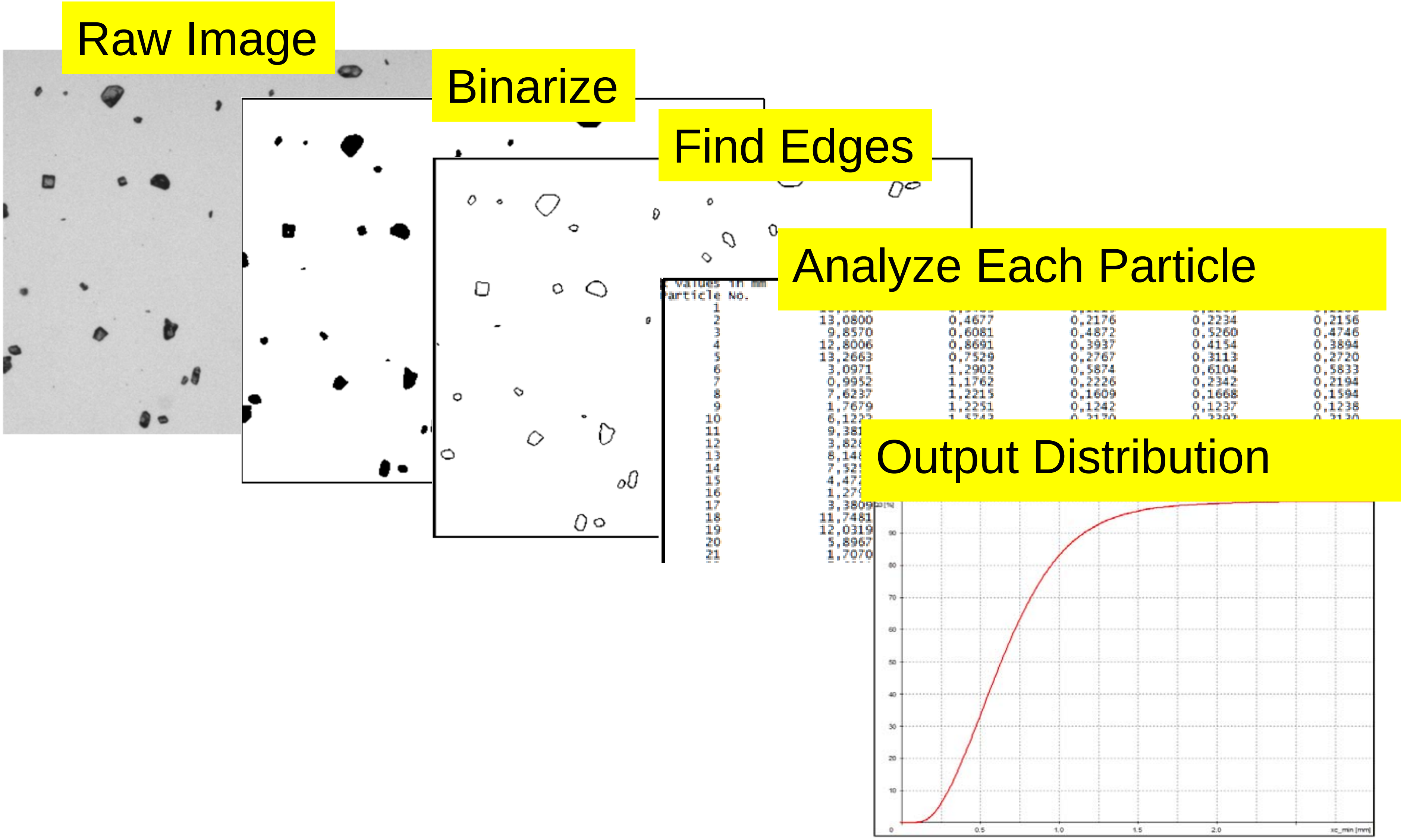


$$\frac{4\pi A}{P^2}$$





Data Evaluation



Effect of Number of Particles Counted

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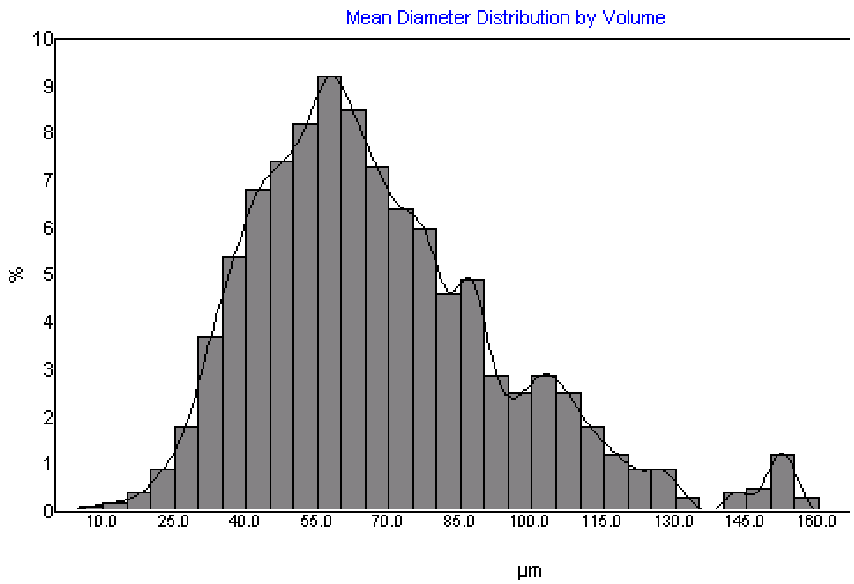
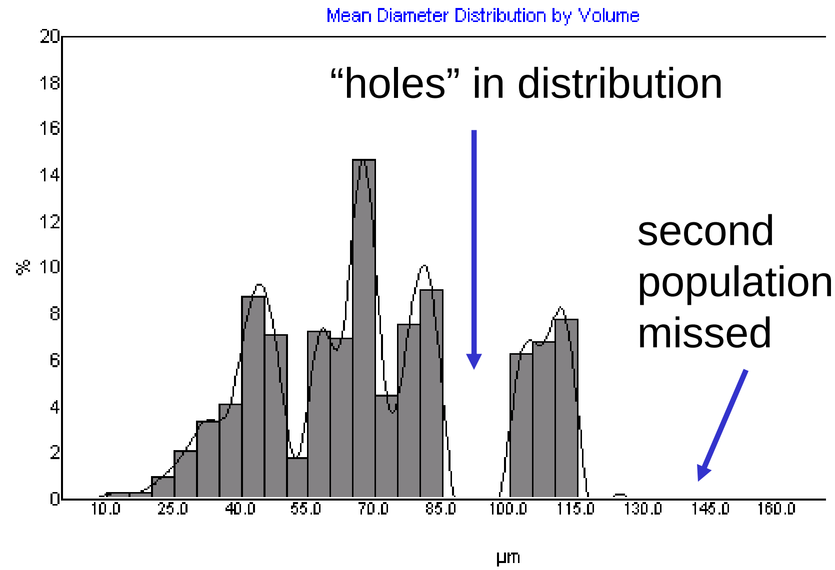
20,000 particles



Statistics - Silica Catalyst

Mean:	27.477	D[4,3]:	68.088	Min, Ma
STD:	22.129	D[3,2]:	57.598	Confider
RSD:	80.539%	D[v,0.1]:	37.870	Confider
D[n,0.1]:	4.697	D[v,0.5]:	63.467	Optics:
Median:	23.824	D[v,0.9]:	104.351	Scanne
D[n,0.9]:	57.814			

200 particles



Statistics - Silica Catalyst 200

Mean:	31.805	D[4,3]:	69.060	Min, M
STD:	21.927	D[3,2]:	58.947	Confid
RSD:	68.942%	D[v,0.1]:	39.418	Confid
D[n,0.1]:	7.021	D[v,0.5]:	68.941	Optics
Median:	29.488	D[v,0.9]:	105.901	Scanr
D[n,0.9]:	63.734			

But d10, d50 &d90 may appear similar

More particles for more accuracy.

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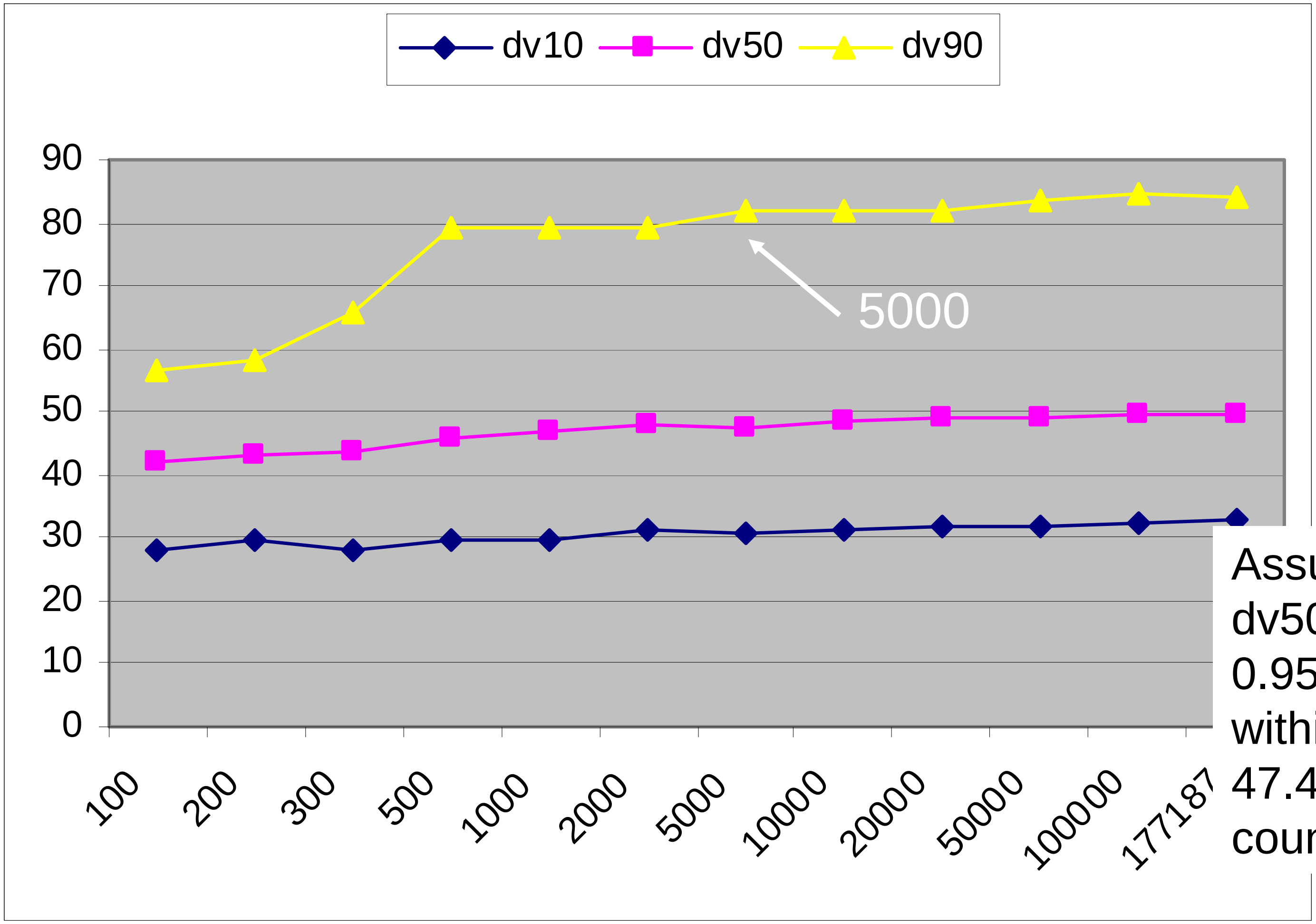
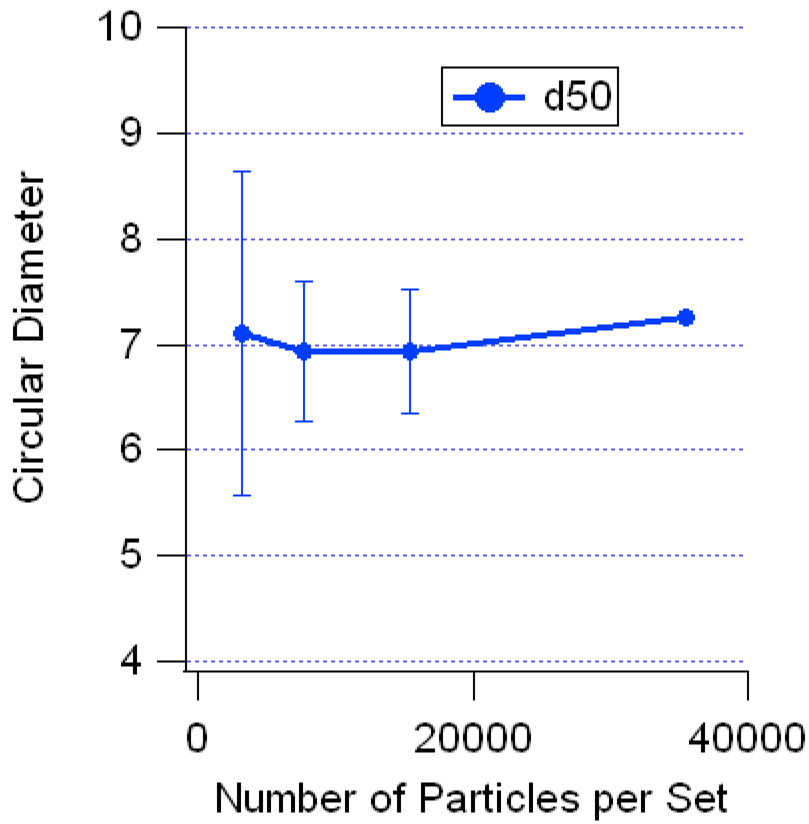


Assume 49.833 is "correct" dv50,
0.95 x 49.833 = 47.34 is within 95%,
47.463 achieved by 5000 counts!

Use this to control precision of your data (and not spend extra time on precision you don't need.

Divide Large Data Set into Smaller Sets



- Error bars are one standard deviation from repeated measurements of the same number of particles from different parts of the sample.
- The error bars get smaller as you evaluate more particles.

<USP> 776: Standard Deviation

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$$\sqrt{s^2\left(\frac{n}{\chi_a^2}\right)} < \sigma < \sqrt{s^2\left(\frac{n}{\chi_b^2}\right)}$$

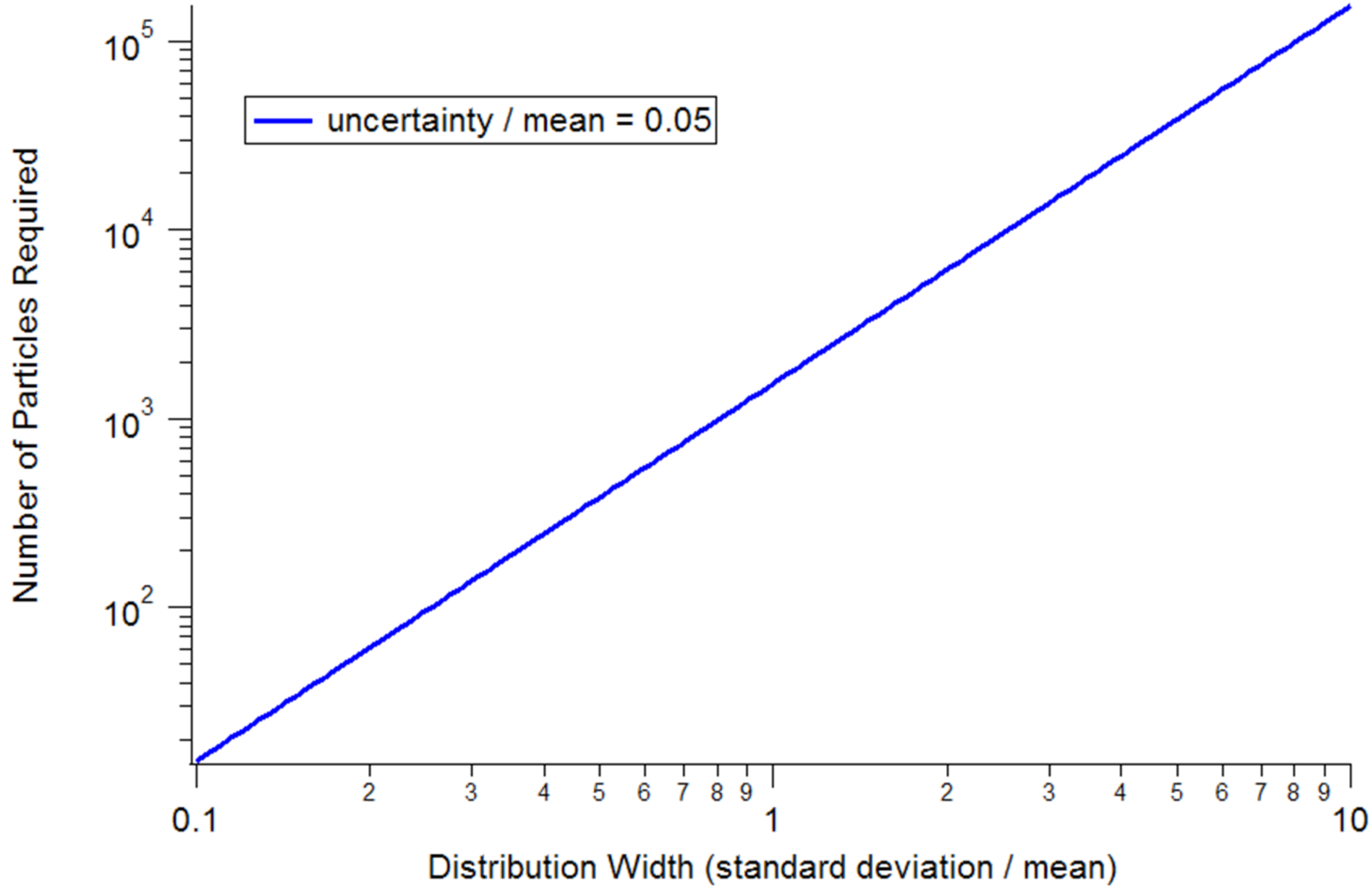
χ=Moment of chi squared distribution (see a statistics book)
s=estimated standard deviation of distribution (width)
n=number of particles measured

These limits are asymmetric around the standard deviation.

Implies normal particle size distribution, greater than 30 particles, and known standard deviation.

How Many Particles?

HORIBA Scientific



Some materials have a distribution such that SD/Mean ~ 1.
To obtain reliable mean values, measure ~1500 particles.
To obtain more details about the distribution, (10x?) more particles need to be measured.

Accuracy

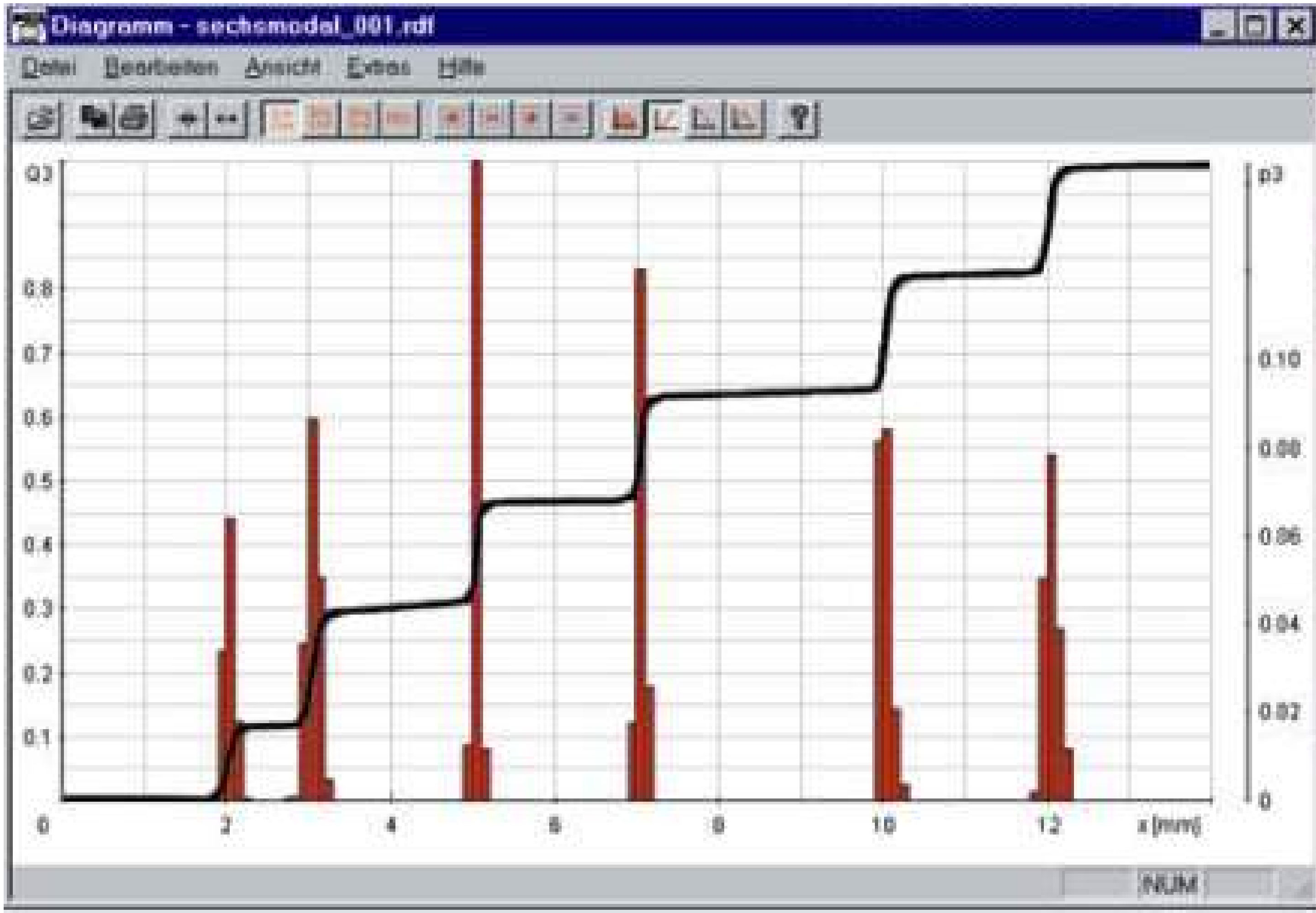
- Is that the "real size"?

- Image analysis uses actual pictures to extract size.
- Calibration with a reticle.

Resolution

HORIBA
Scientific

sults



mixture of six sizes of grinding balls

Why dynamic image analysis

HORIBA Scientific

- Robust measurement….the interaction between the instrument and the particle is optical, so there is no wear and change in calibration.
- High resolution size distribution results
- Fast

Also, these are all reasons to use Dynamic Image Analysis instead of sieves.

The HORIBA PSA300

- Turnkey System
  - More time getting results and less time engineering
- Automated
  - Faster
  - Less operator labor
  - Less operator bias
- Powerful Software Features
  - Image Enhancement
  - Particle separation
- Separate Disperser Option
  - More flexible sample preparation

The CAMSIZER

- Measurement of very broad particle distributions
- Direct particle definition
  - by width (analogue to sieving)
  - by length
  - or projection surface
- Two camera system for more accuracy/wider range
- Easy operation
- Fail-safe, robust
- Ideal for particle shape analyses
- Measurement of density, counting of particles

Static or Dynamic Image Analysis?

HORIBA Scientific

- **Dynamic**
  - Broad size distributions (since it is easier to obtain data from a lot of particles)
  - Samples that flow easily (since they must be dropped in front of camera)
  - Powders, pellets, granules

- **Static**
  - Samples that are more difficult to disperse (there are more methods for dispersing the samples)
  - Samples that are more delicate
  - Pastes, sticky particles, suspensions

Conclusions

HORIBA Scientific

- Image Analysis is good for
  - Replacing Sieves
  - Size
  - Shape
  - Supplementing other techniques
- Watch out for
  - Sample preparation
  - Image quality
  - Measure enough particles

Questions?

www.horiba.com/us/particle

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