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Particle Analysis

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Size and Shape of Particles from Dynamic Image Analysis

October 30, 2018

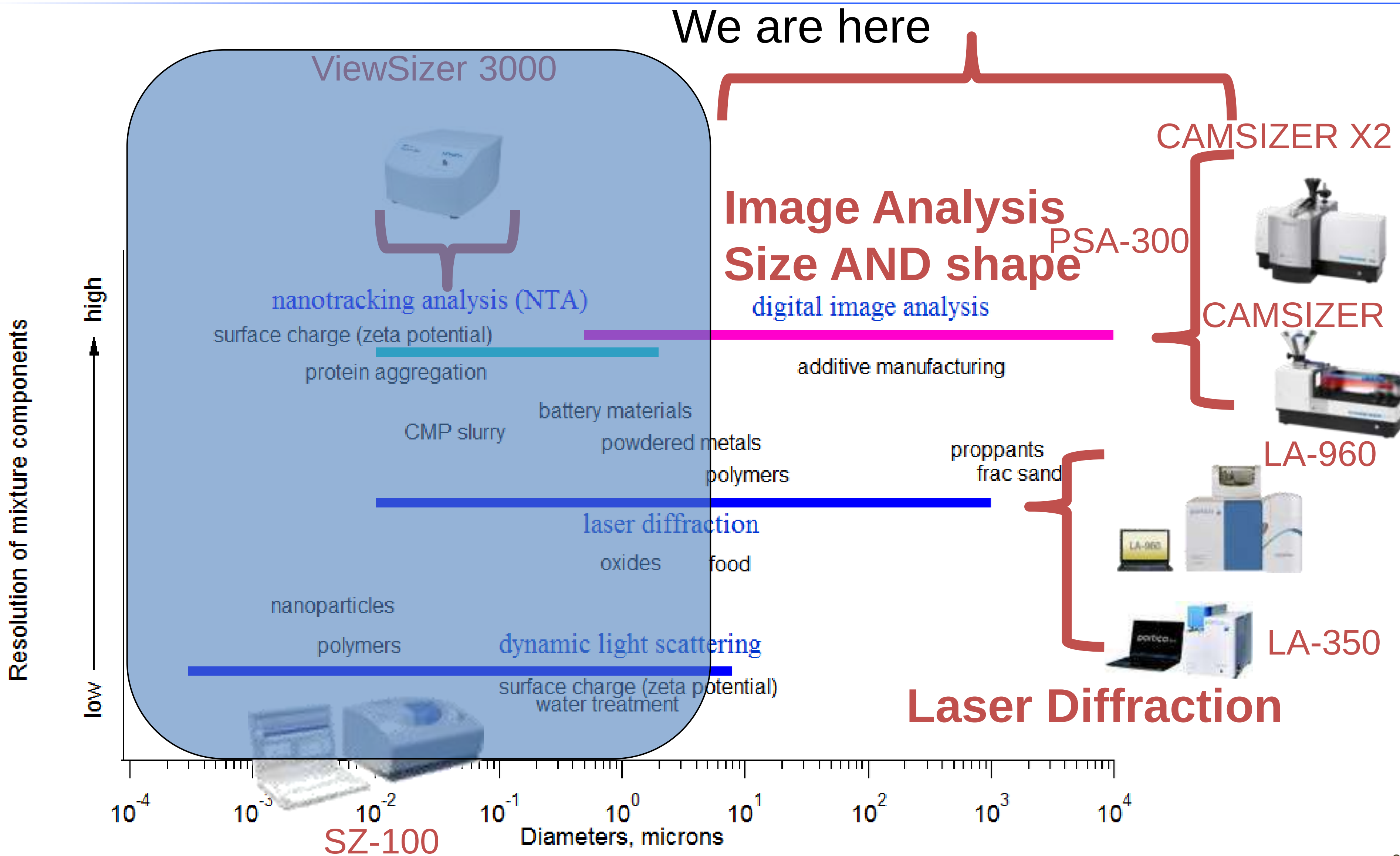
Intro

Orientation

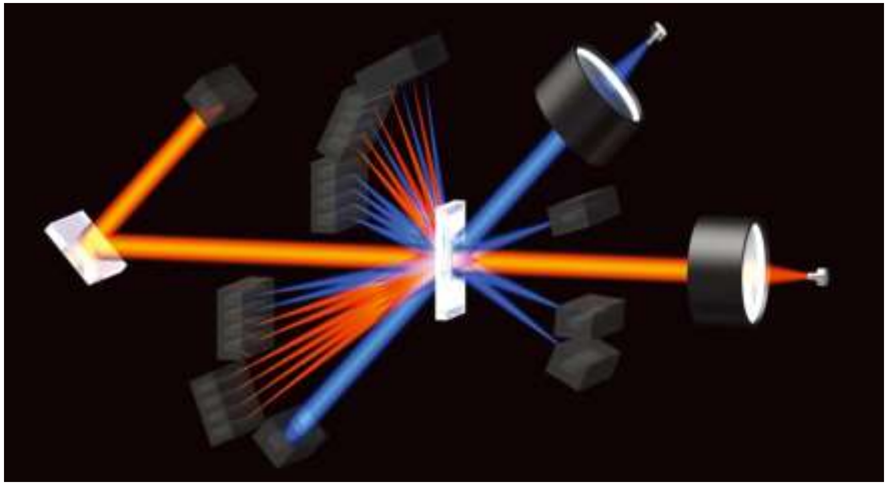
What is image analysis

Why?

Analysis Techniques



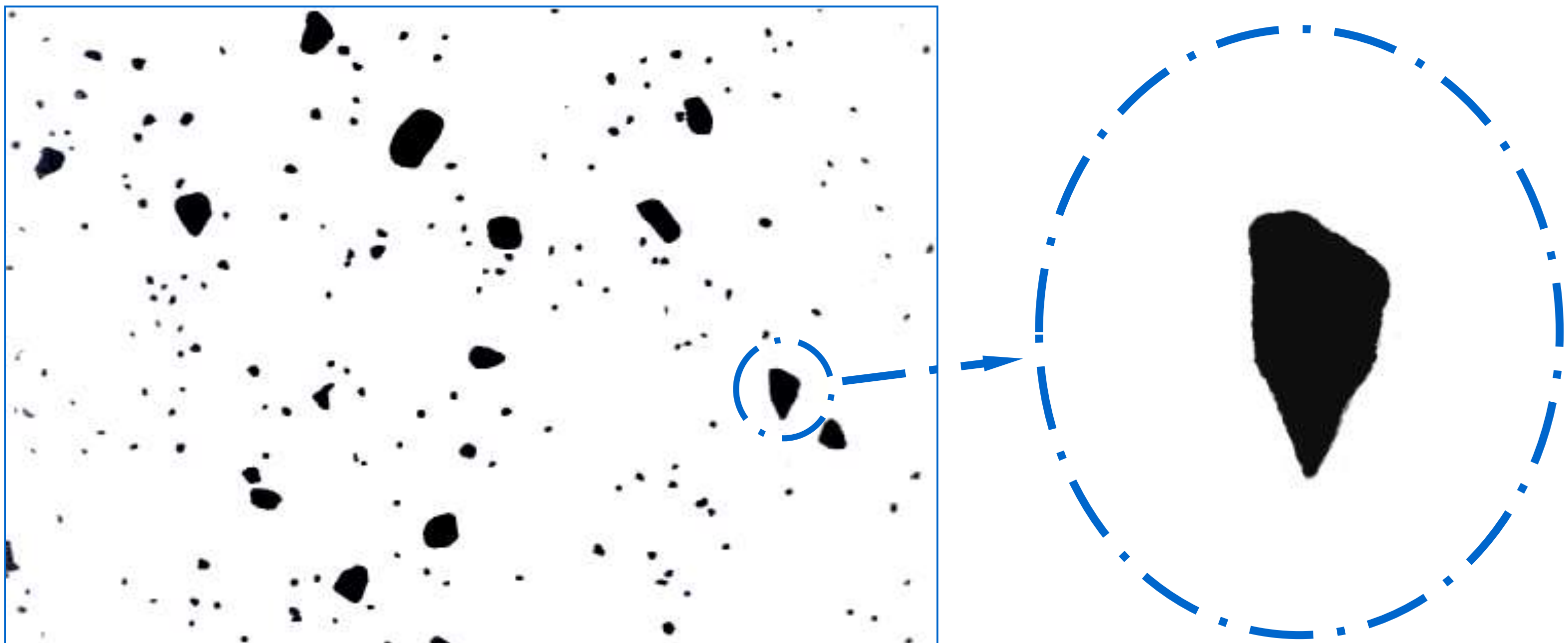
Laser Diffraction



- Converts scattered light to particle size distribution
- Quick, repeatable
- Powders, suspensions
- Most common technique

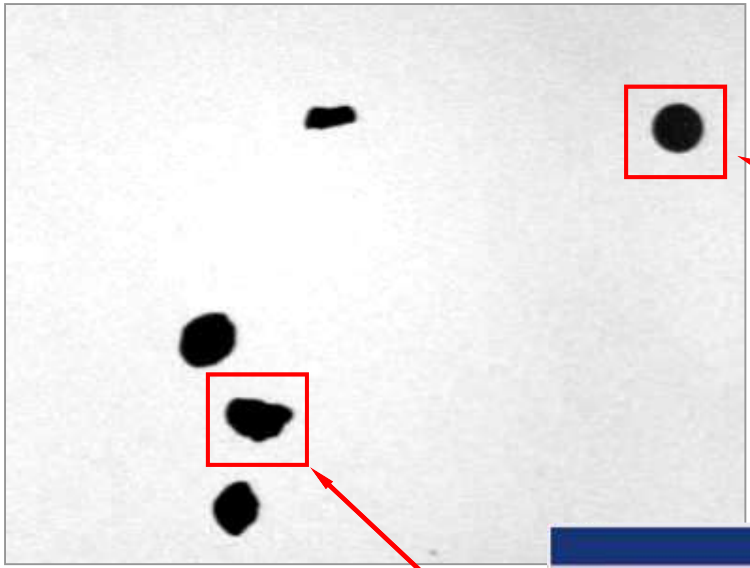
- Fast, gets to very small size, but no shape information

What is image analysis?



Take a picture,
analyze for
size and **shape**

Size and shape from pictures



x-values in mm

Nr=0 , d=2.148 ,

xFe_min=2.148 , xFe=2.203 , xFe_max=2.241 ,
xMa_min=2.134 , xMa=2.199 , xMa_max=2.241 ,
xc_min=2.148 , xc=2.200 , xc_max=2.241 ,

Symm=0.9827 , b/l=0.9480 ,
SPHT=0.9818 ,
Convex=0.9997 ,

OK

x-values in mm

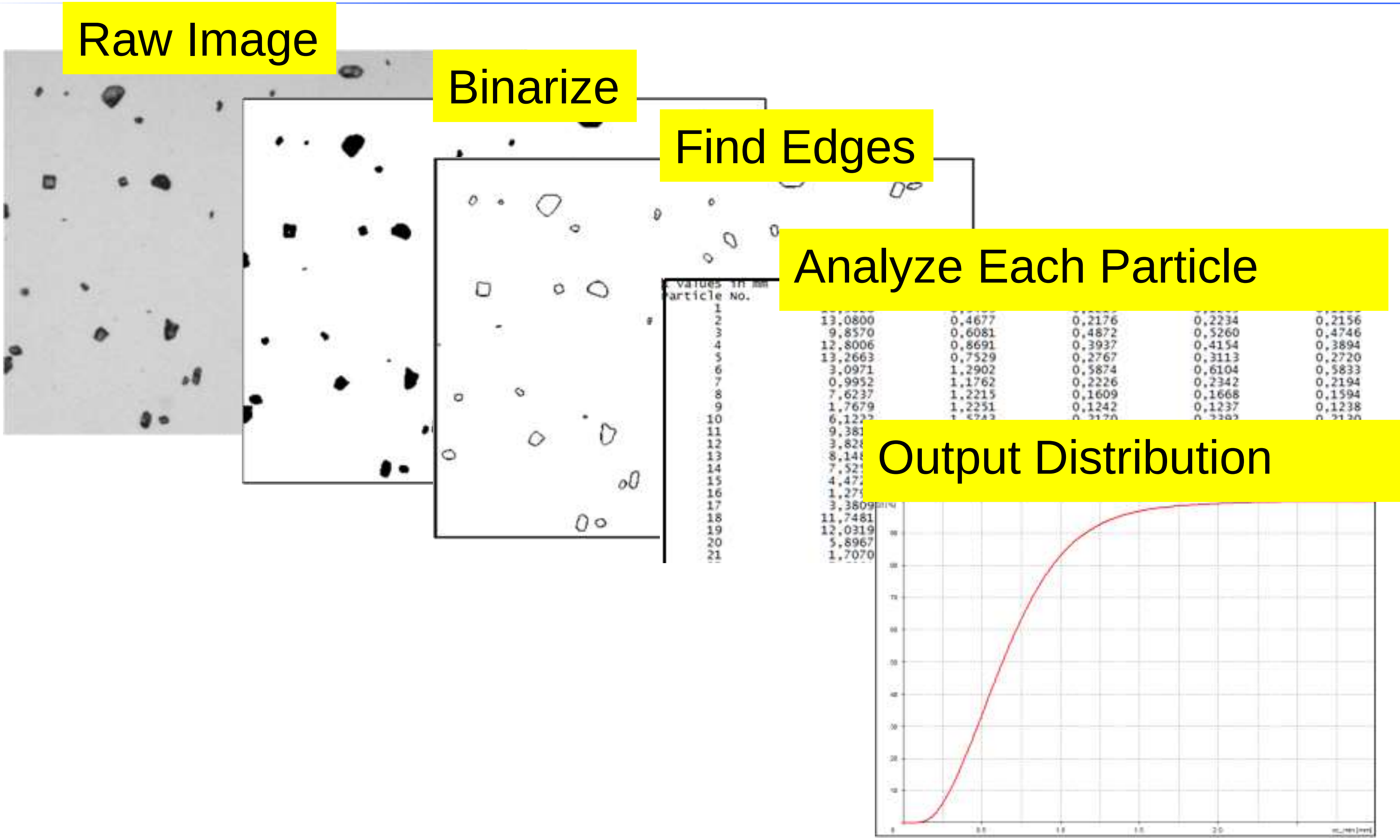
Nr=0 , d=1.966 ,

xFe_min=1.970 , xFe=2.546 , xFe_max=3.063 ,
xMa_min=1.889 , xMa=2.419 , xMa_max=3.047 ,
xc_min=1.966 , xc=2.419 , xc_max=3.060 ,

Symm=0.9289 , b/l=0.6361 ,
SPHT=0.9066 ,
Convex=0.9995 ,

OK

Data Evaluation



Why image analysis?

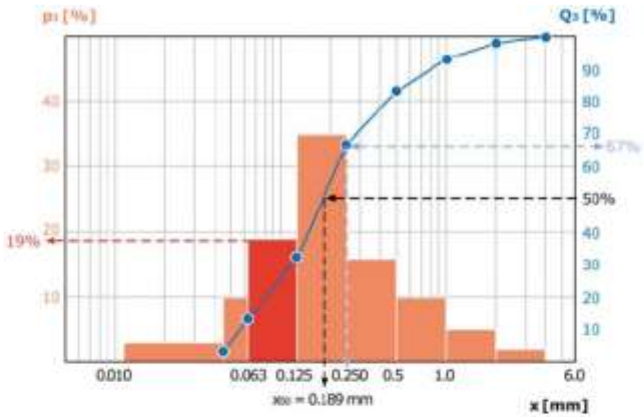
- Replace sieves (really!)
- Verify/supplement laser diffraction results (orthogonal technique).
- Need shape information, for example due to importance of powder flow

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

Why image analysis?

Replace Sieves

- **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**



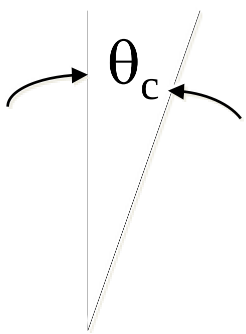
Size class [mm]			p_3 [%]	Q_3 [%]
	<	0.045	3.0	3.0
0.045	-	0.063	10.0	13.0
0.063	**-**	**0.125**	**19.0**	**32.0**
0.125	-	0.250	35.0	67.0
0.250	-	0.500	16.0	83.0
0.500	-	1.000	10.0	93.0
1.000	-	2.000	5.0	98.0
2.000	-	4.000	2.0	100.0
> 4.000			0.0	100.0

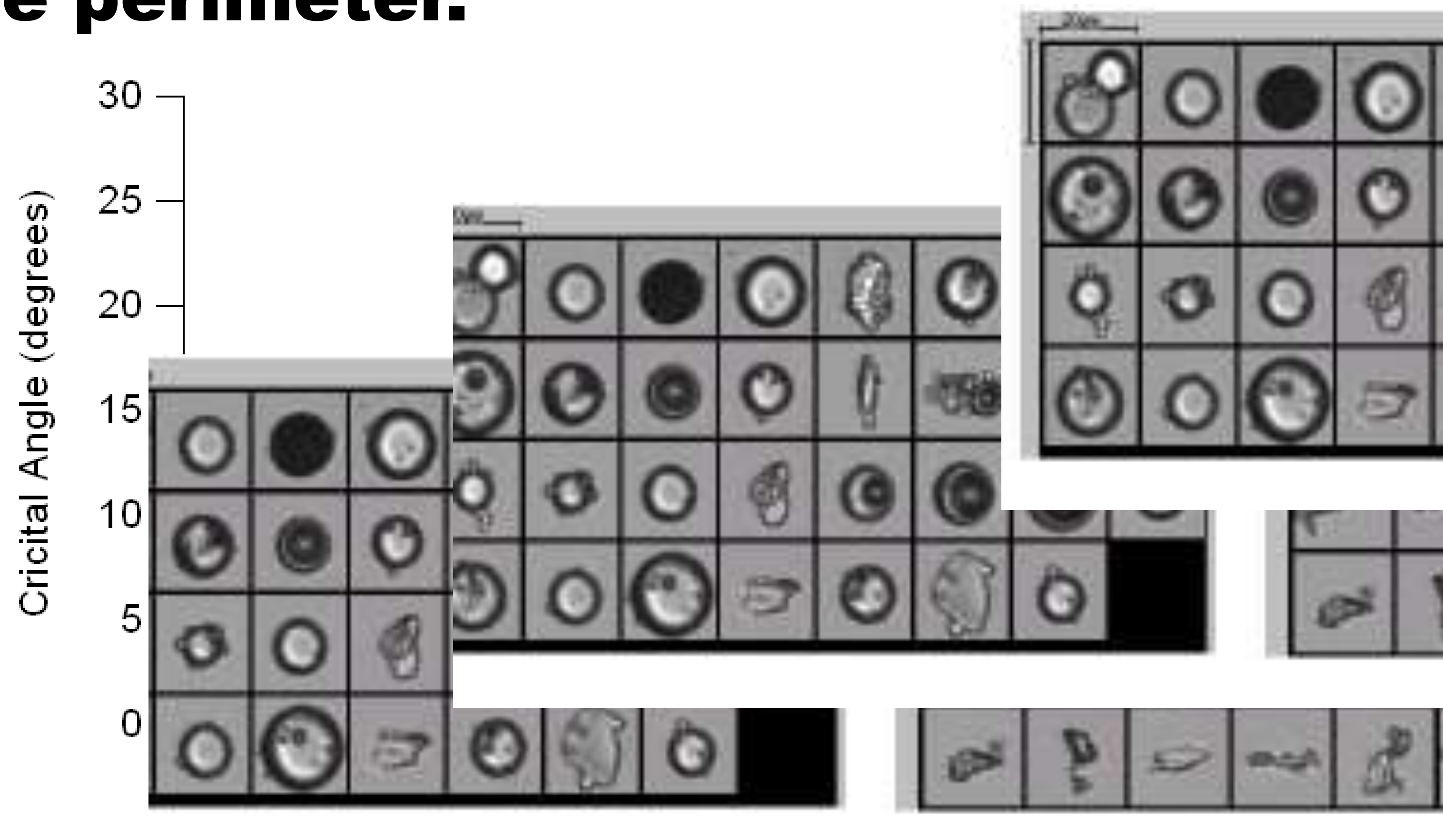
x_{50} = 0.189 mm

More information available through www.retsch.com

Why: effect of shape on flow

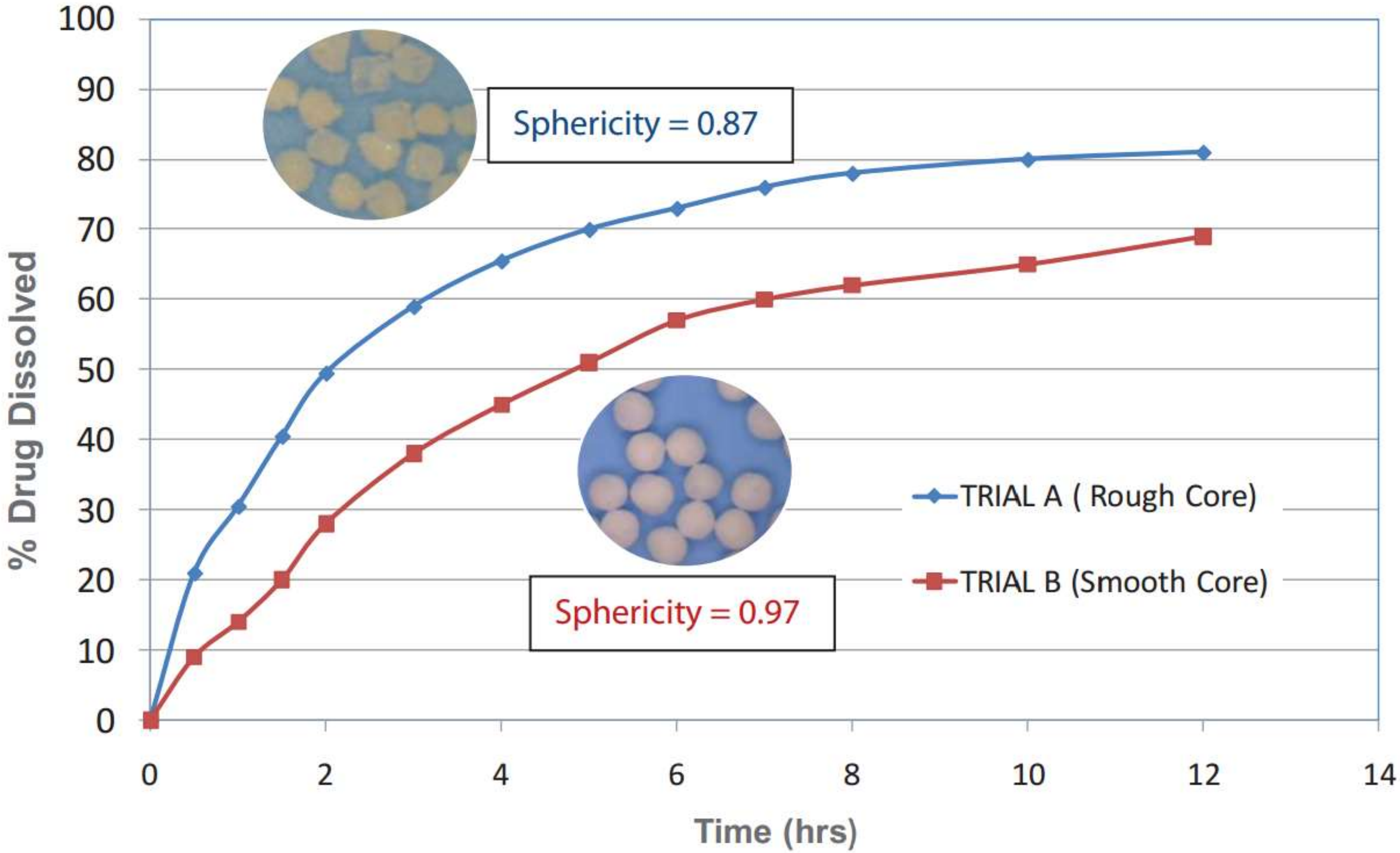
- **Yes, I assumed density doesn't matter.**
- **Roundness is a measure based on particle perimeter.**



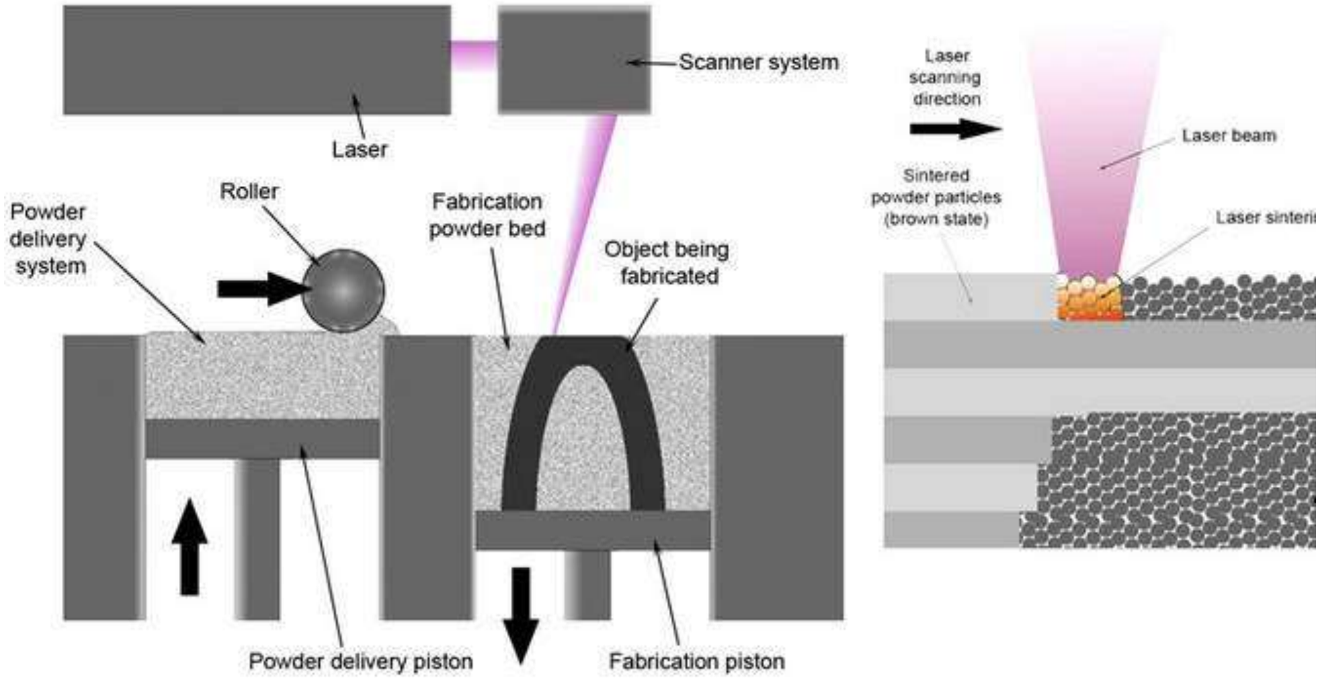


Why: multiparticulate drugs

Shape affects drug release profile

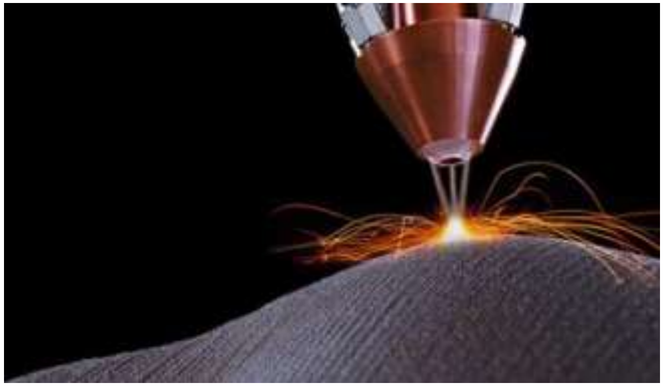


Why: 3D printing



3D printing with metal / additive manufacturing

Size affects quality, shape affects flow.



The image

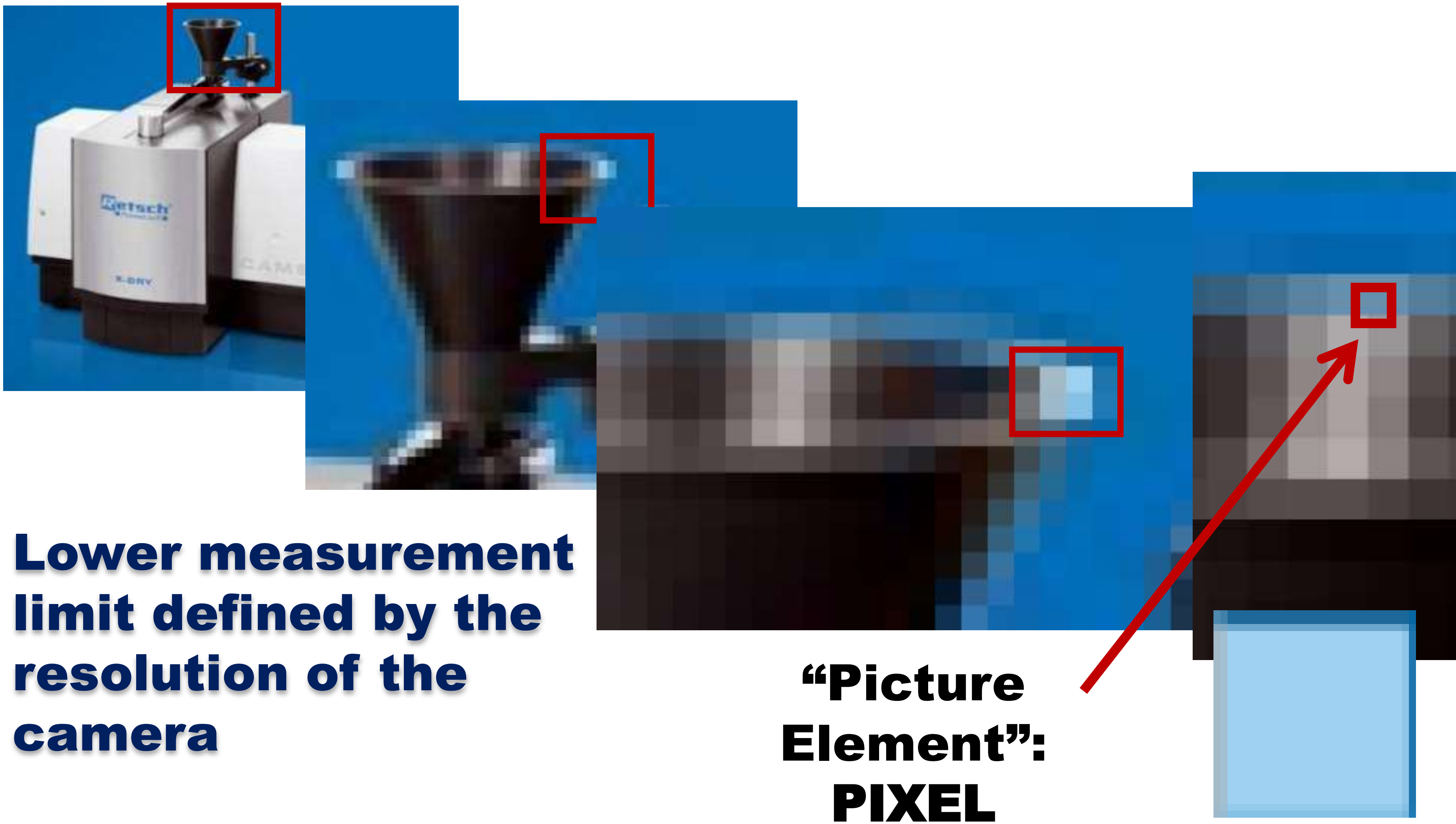
Resolution

Max size/field of view

Two cameras

The image: resolution

Pictures of digital cameras:



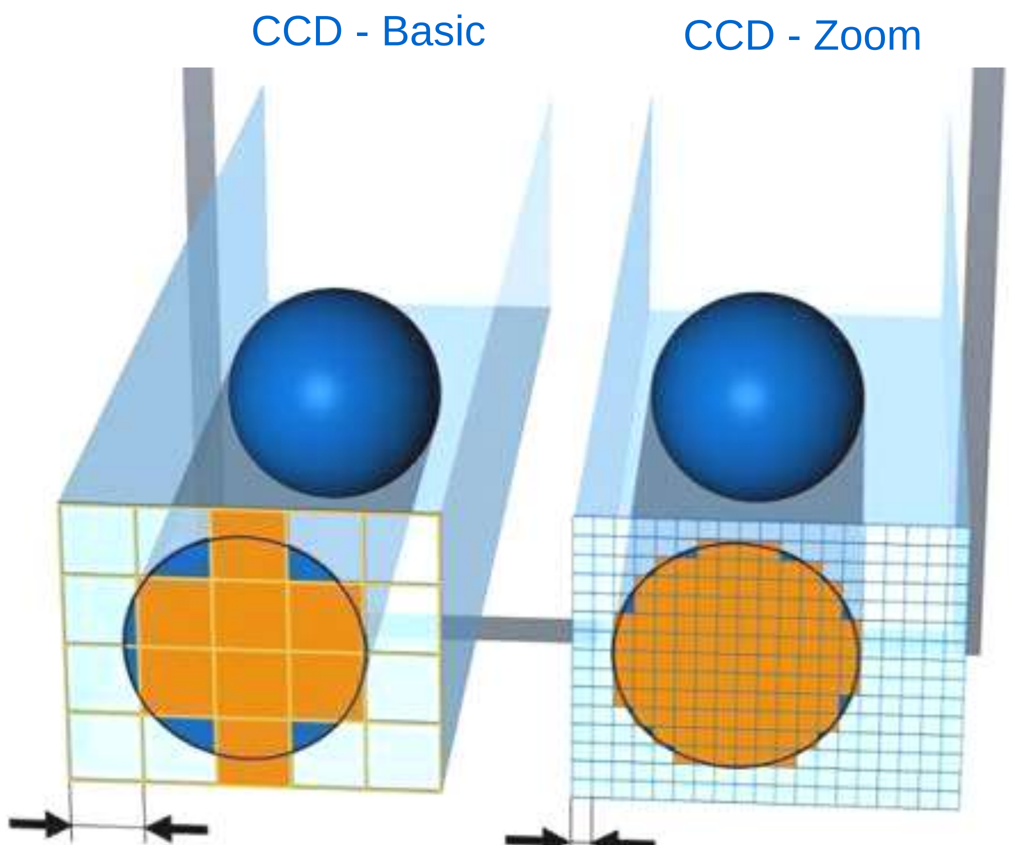
Lower measurement limit defined by the resolution of the camera

"Picture Element": PIXEL

The image: resolution

Detection of particles

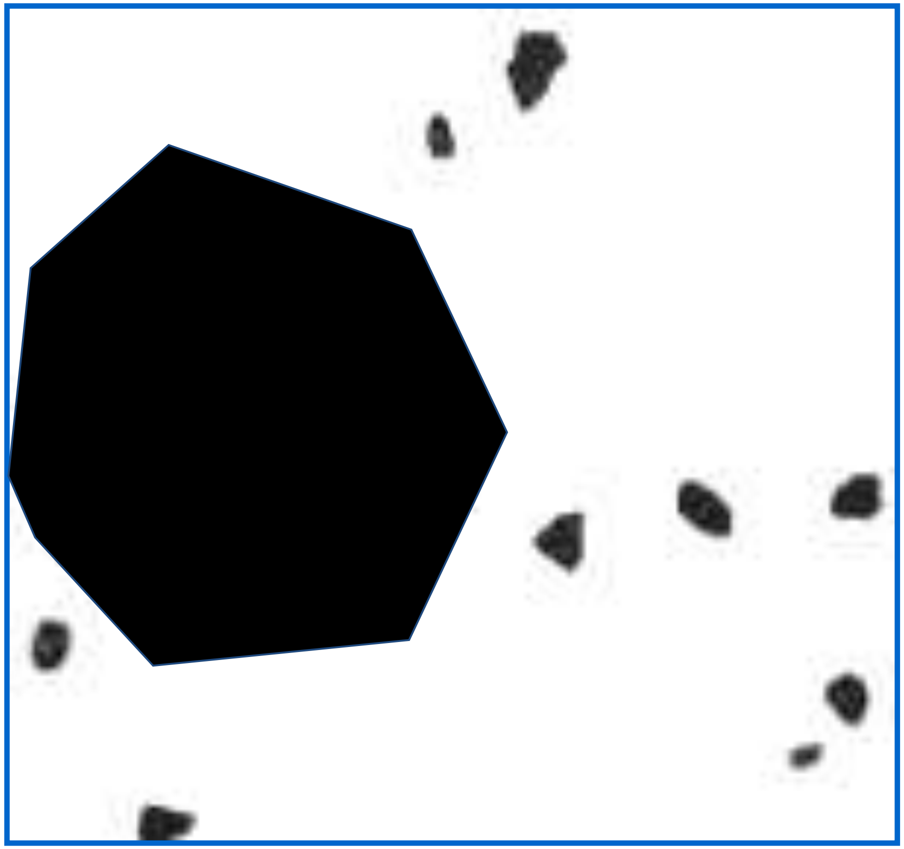
One pixel is element of a projection when at least half of the pixel is covered.



	CCD - Basic	CCD - Zoom
CAMSIZER	75µm	15µm
CAMSIZER XT	10µm	0.8µm

The image: maximum size

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

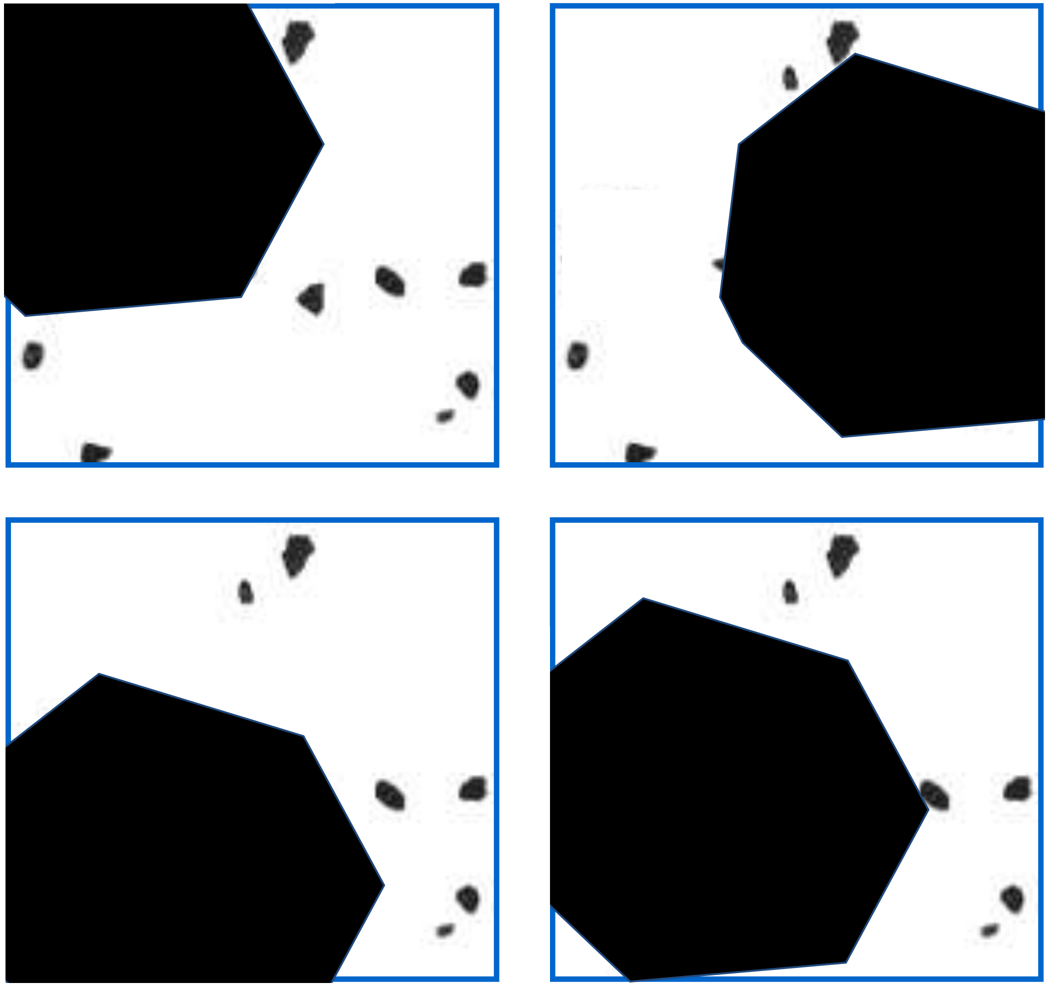


The image: maximum size

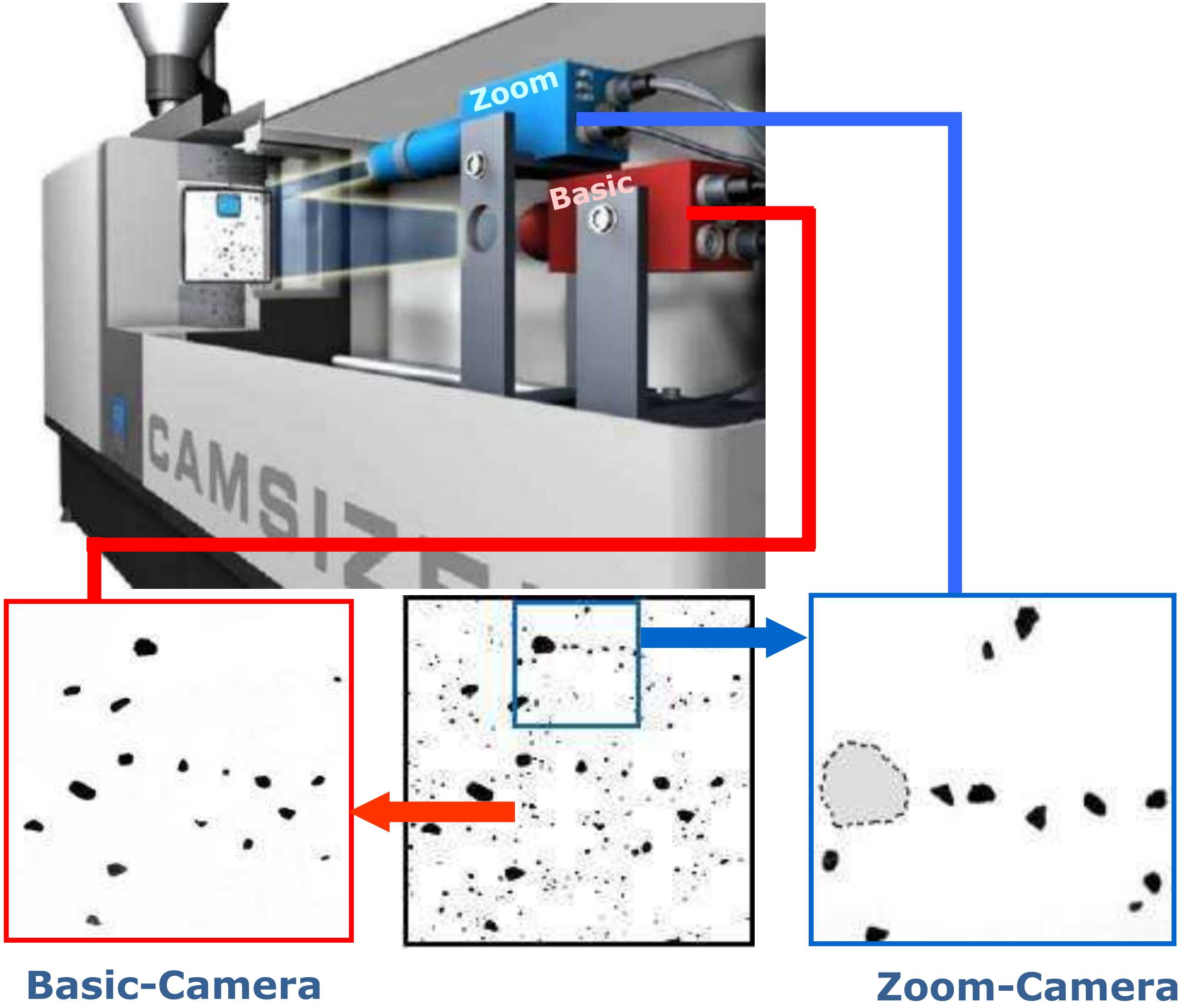
The probability of large particles touching the edge of the frame is higher than for smaller particles.

=> Large particles cannot been measured sufficiently

→ Upper limit of measurement range

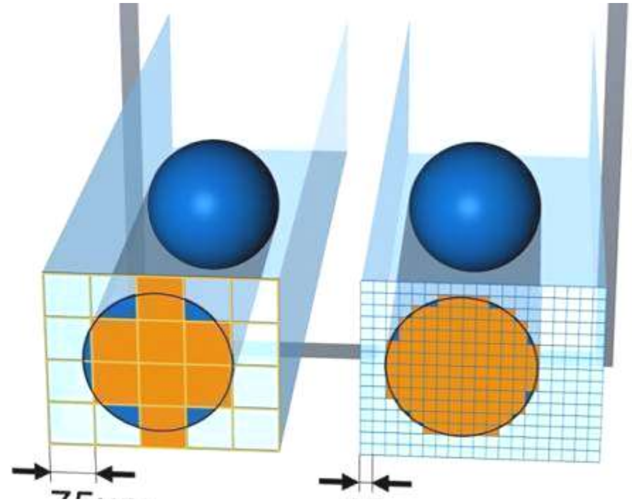


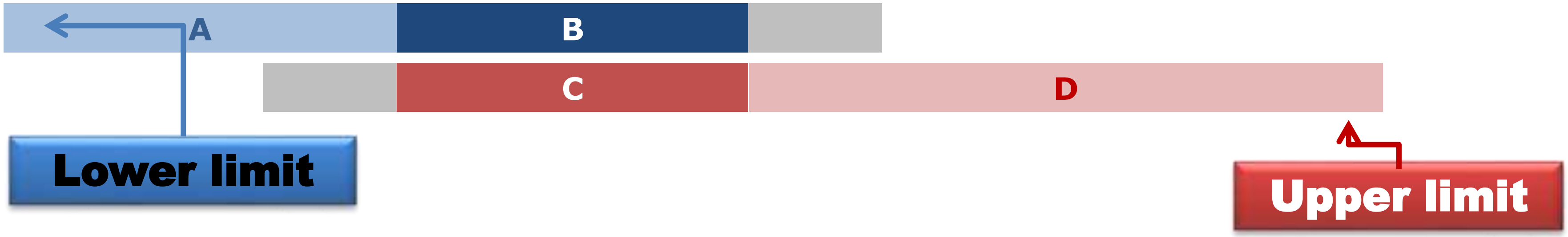
The image: two camera system



The image: two camera system

	CCD - Basic	CCD - Zoom
Pixel Size		
CAMSIZER	66 µm	10 µm
Size Range	130 µm to 30 mm	20 µm to 1.25 mm





The sample

Dispersing

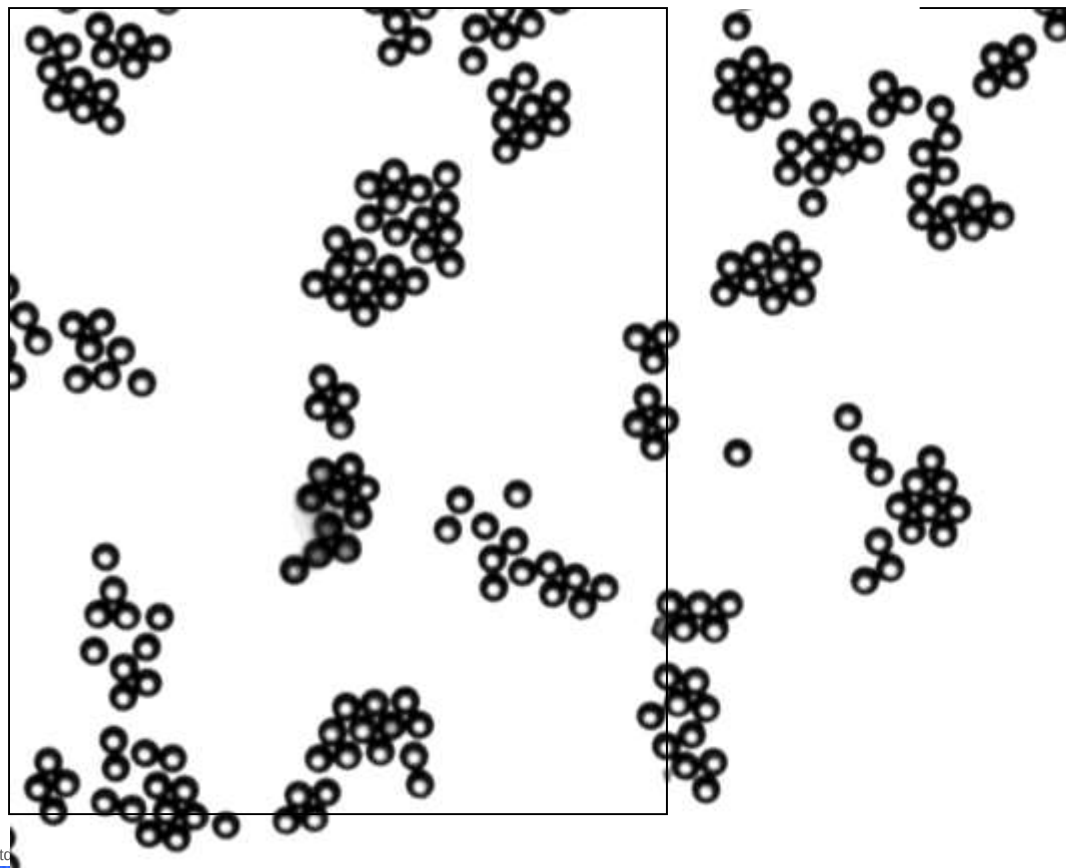
Static vs. dynamic

Dispersing options

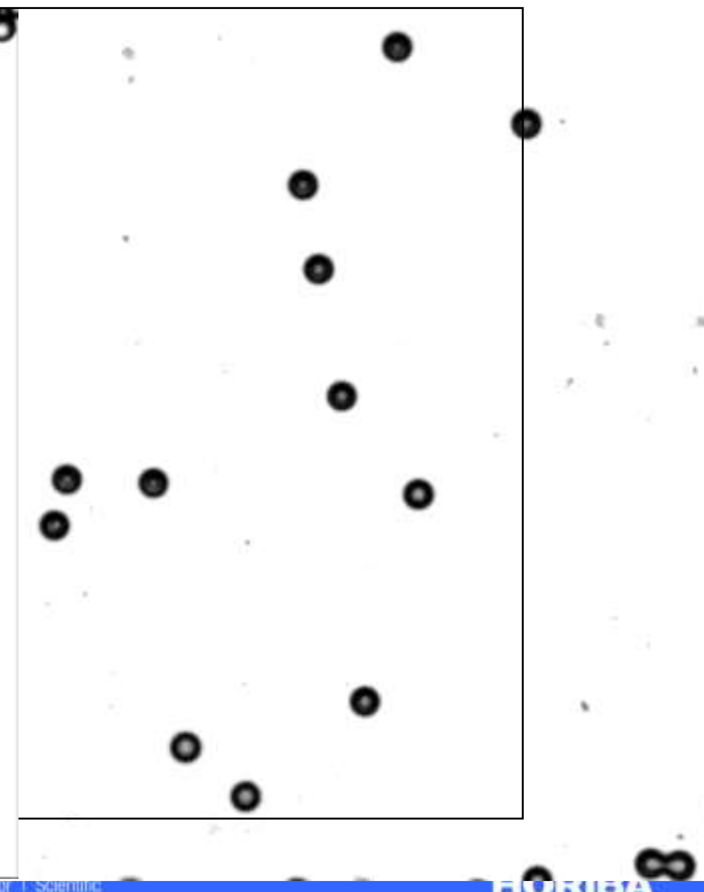
Dispersing a sample

For static image analysis, want to spread particles out so that they don't touch.

No

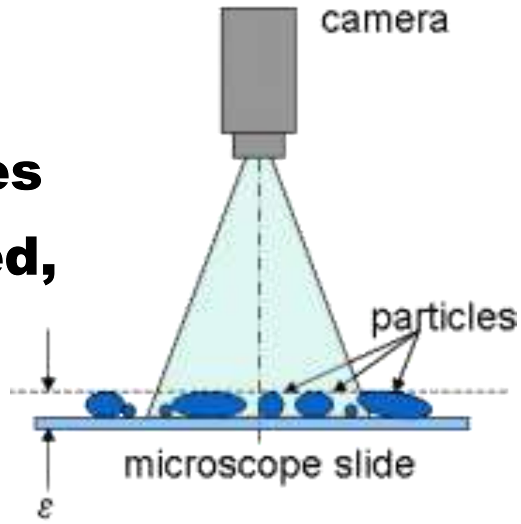


Yes



Static image analysis

- 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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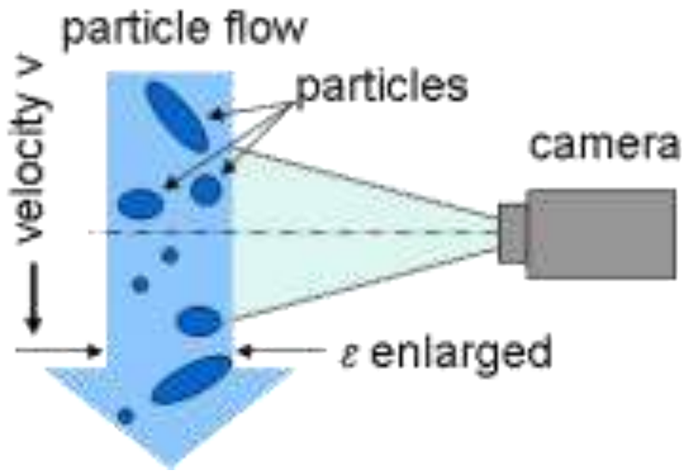
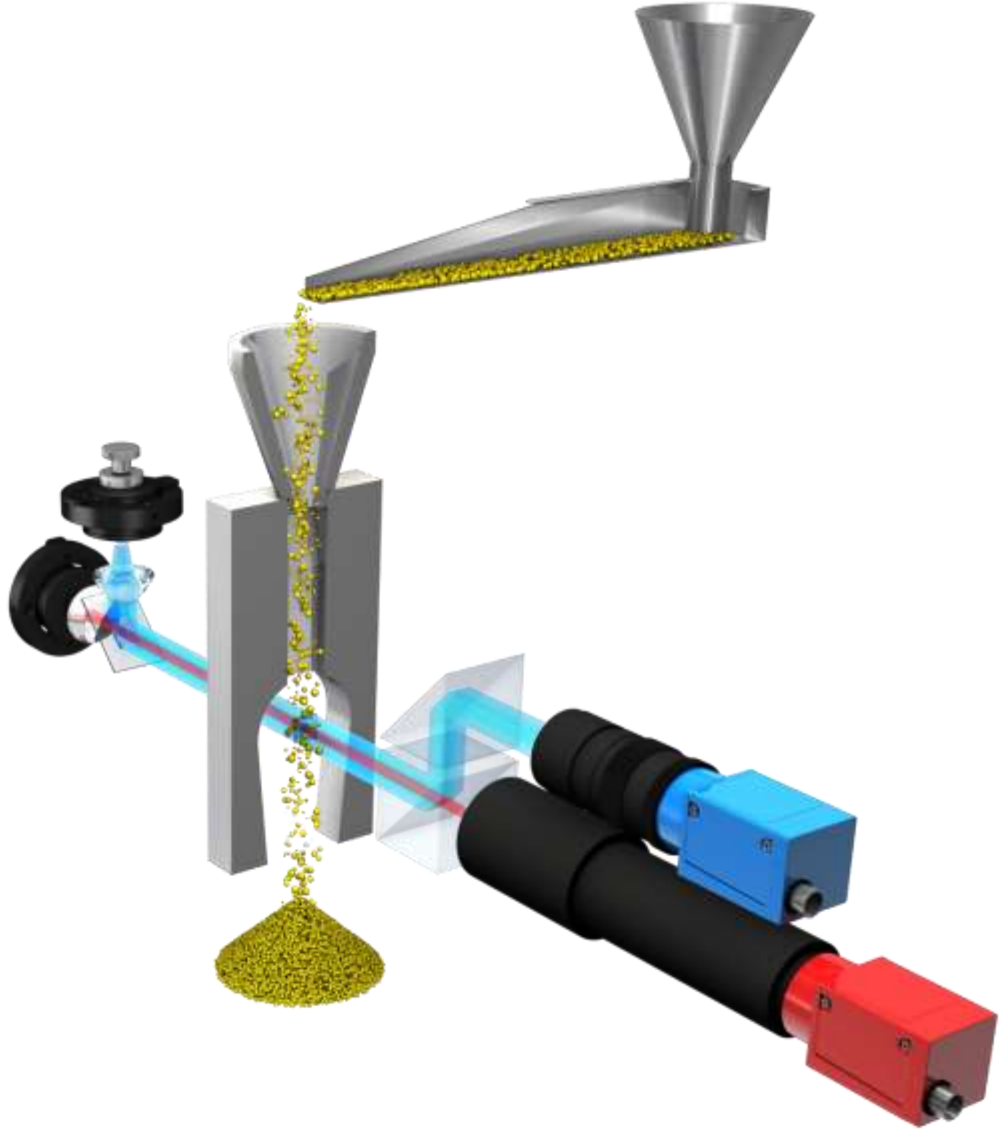
Dynamic image analysis

- 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

Free flowing powder

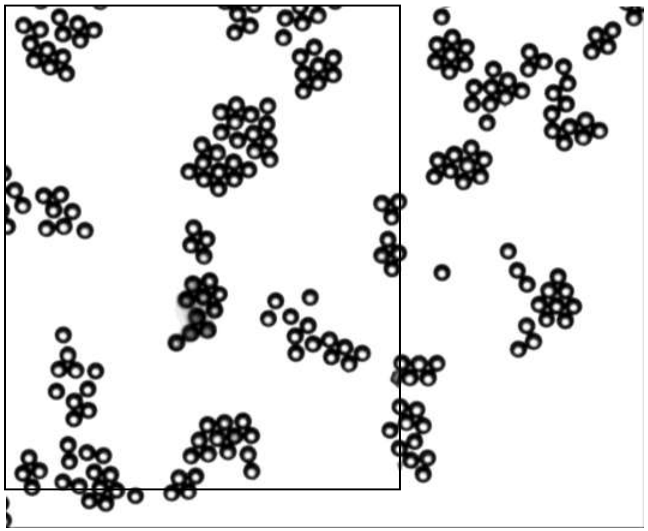


Control feed rate

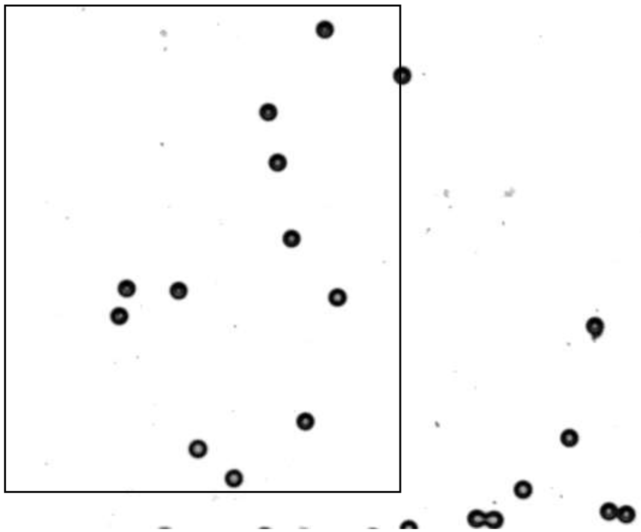
Want to spread particles out so that they don't touch.

Use % of field of view that is covered in order to automatically control feed rate.

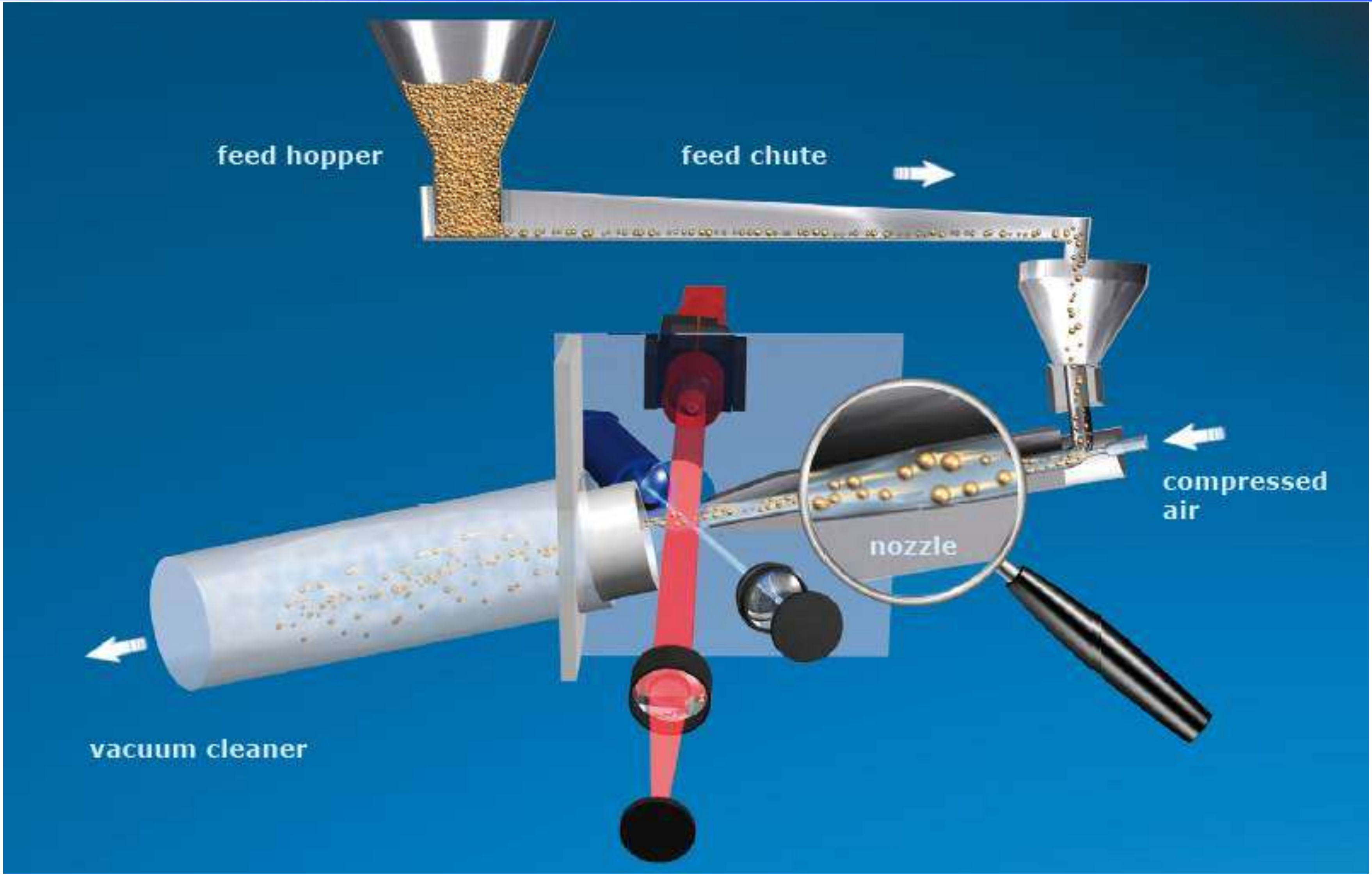
Feeding too fast



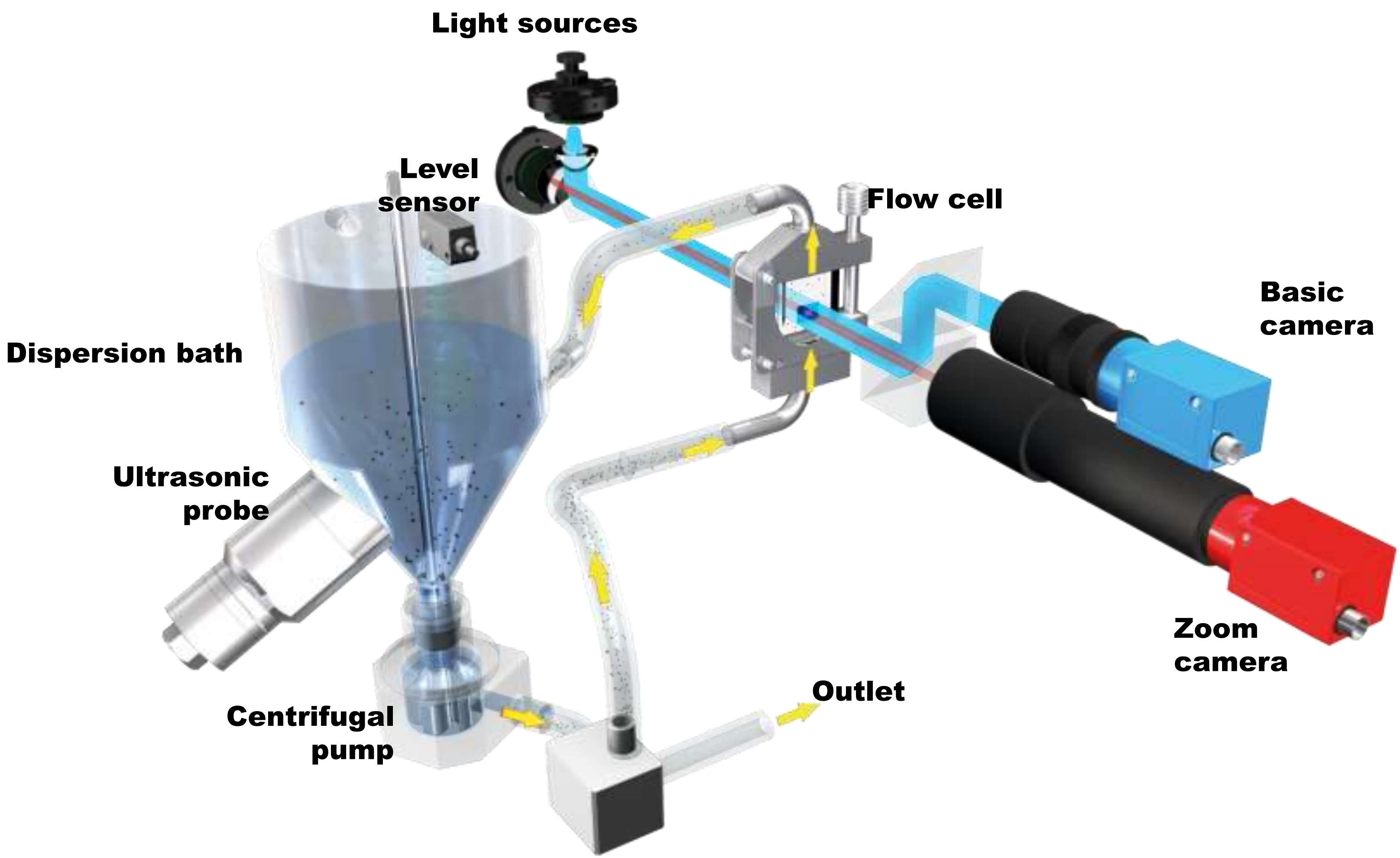
Good



Air dispersion with X-Jet



Or examine wet dispersion…



The data

Size measures

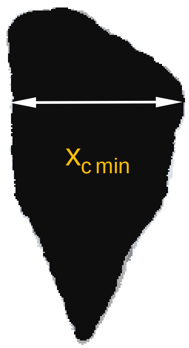
Matching sieves

Shape

Many size measures

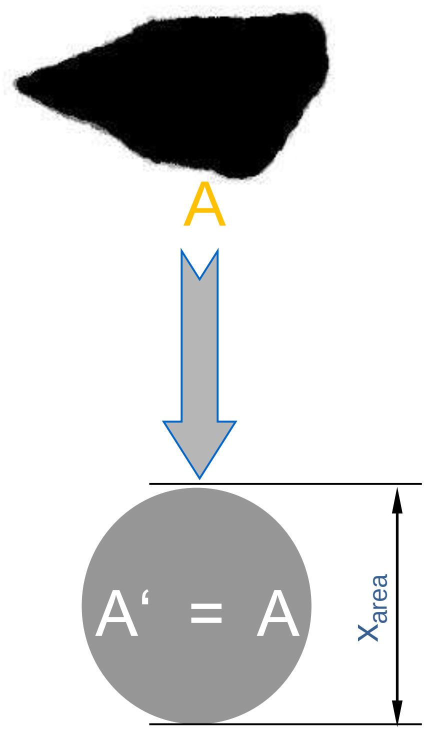
$x_{c\,min}$

"width"



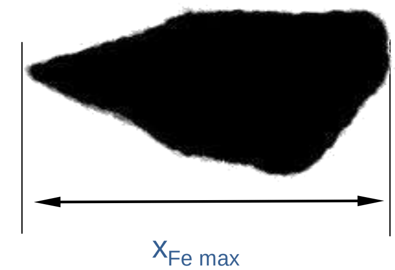
x_{area}

"diameter over projection surface"



$x_{Fe\,max}$

"length"

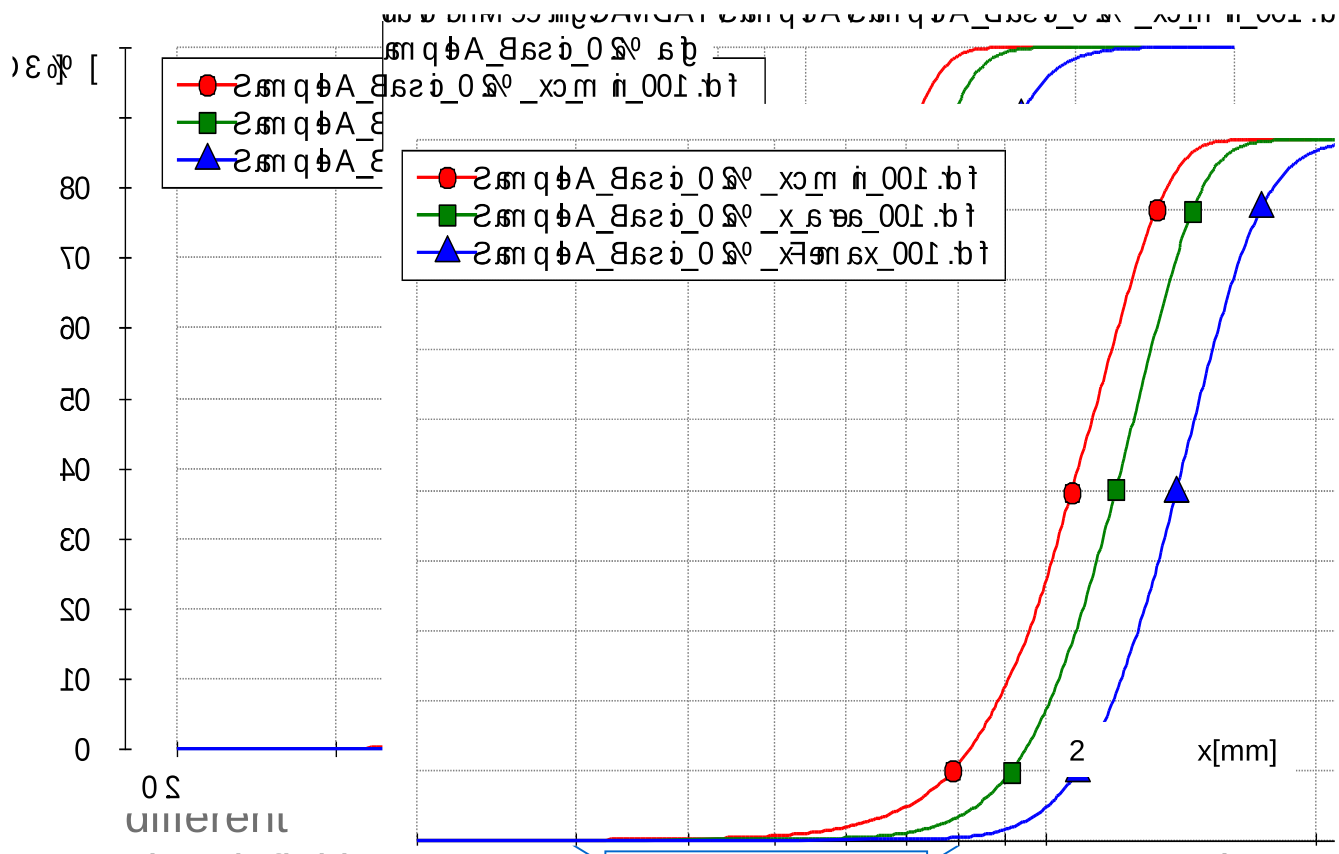


Width is best suited for comparison with sieves !

Shape parameters can be calculated!

Comparison of size definitions

$X_{c\,min}$ ⇔ X_{Area} ⇔ $X_{Fe\,max}$

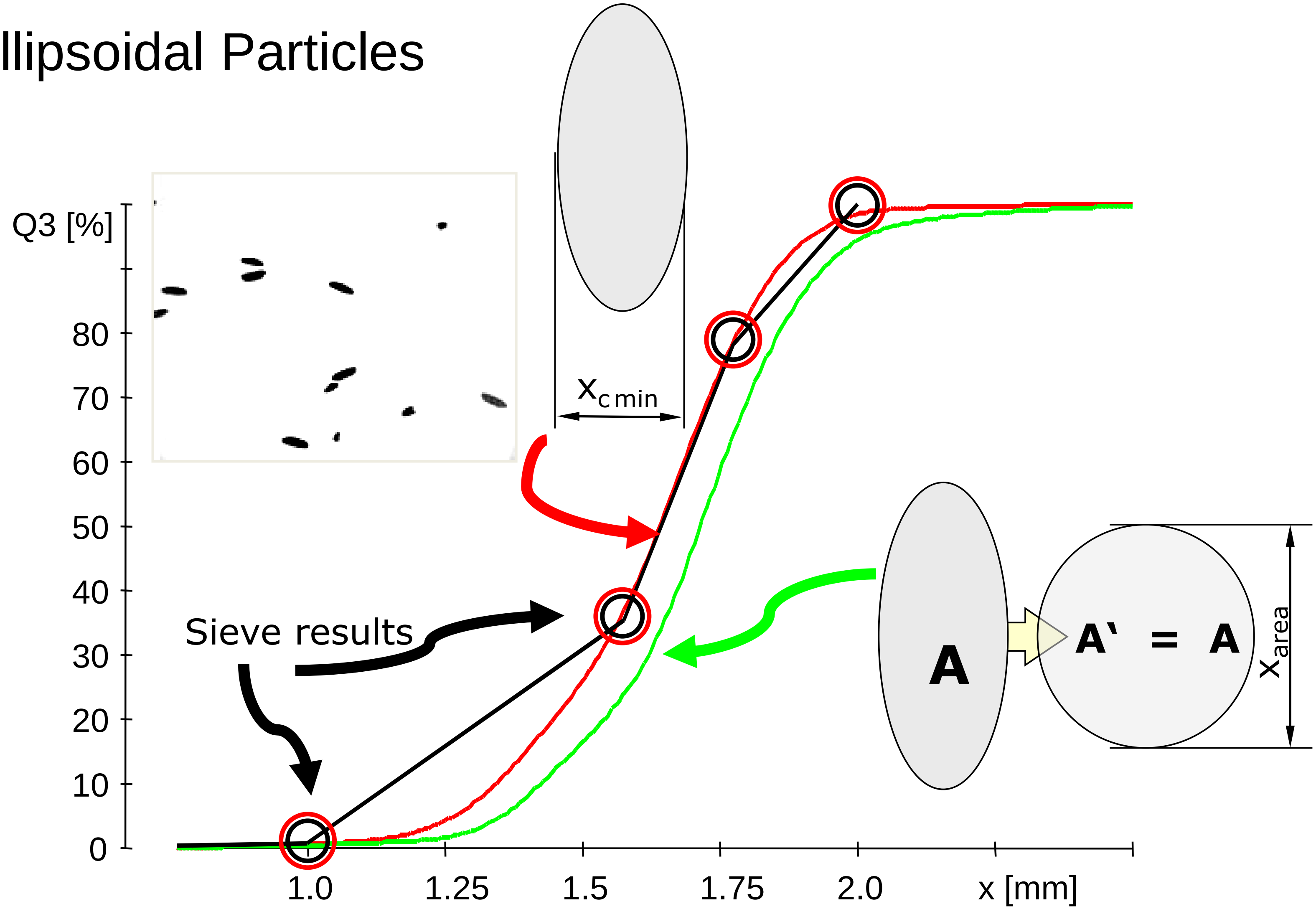


different size definitions

results

Sieving vs. image analysis

Ellipsoidal Particles

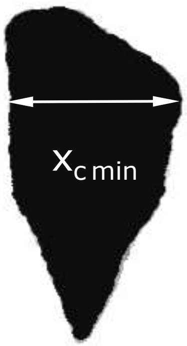


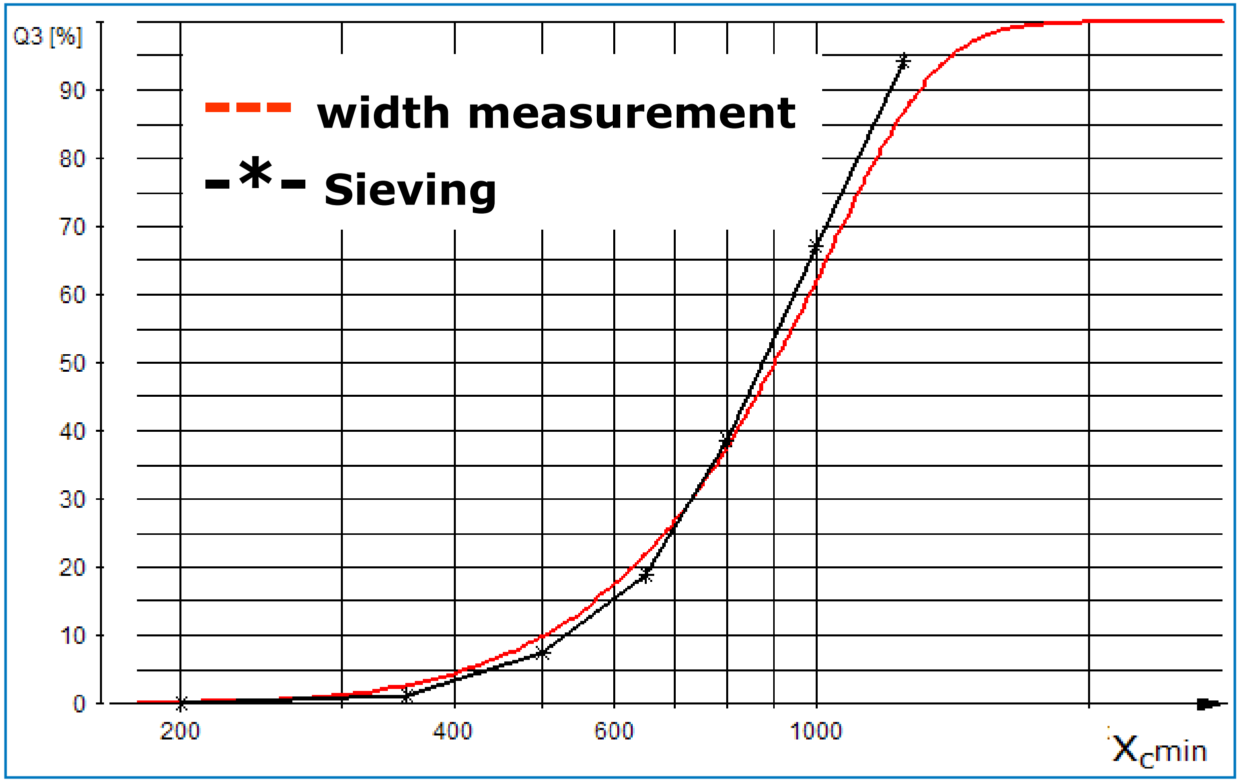
Digital image processing

Width Measurement ⬄ Sieving

Competing Measuring Methods

$x_{c\,min}$
"width"

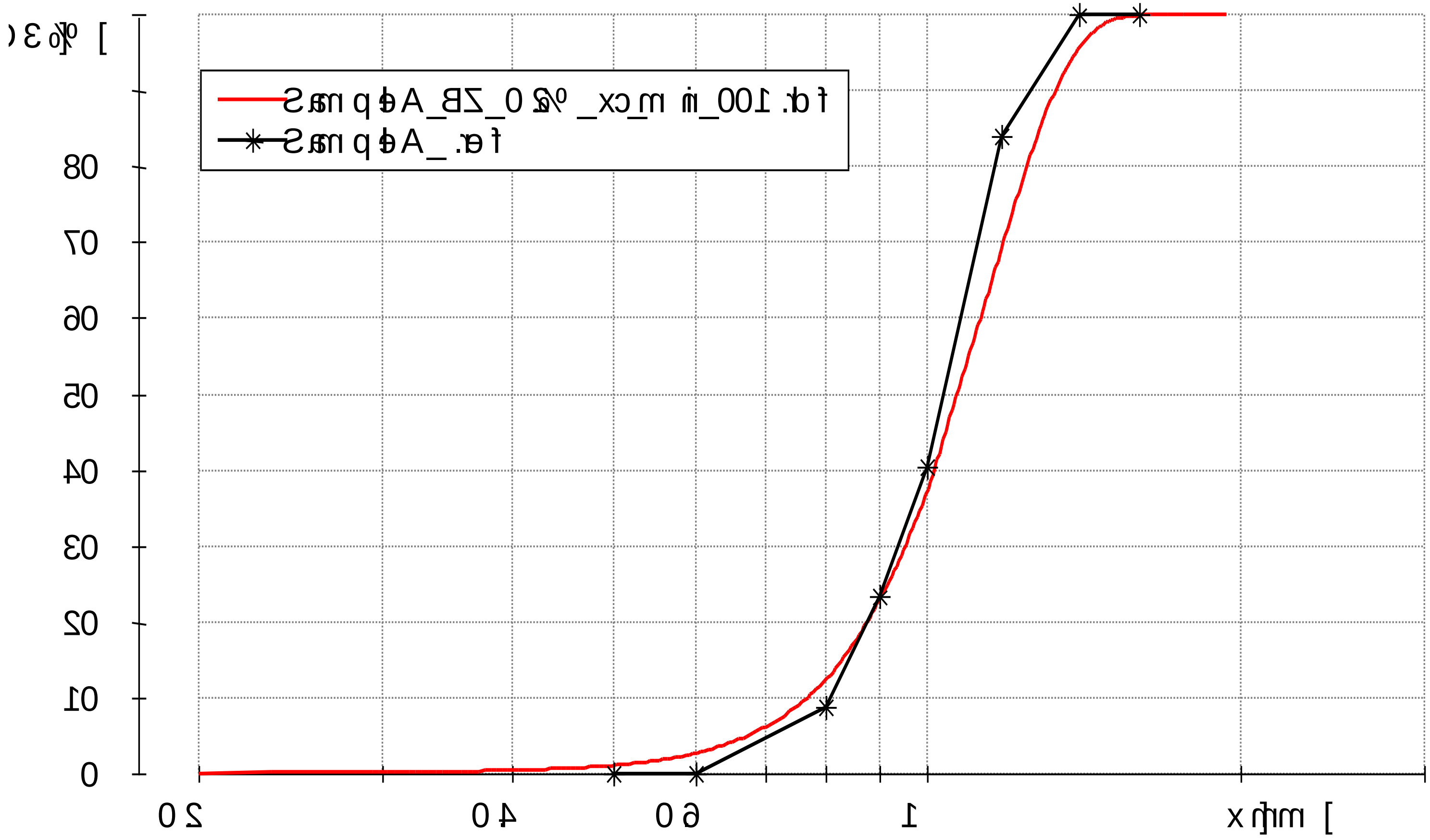




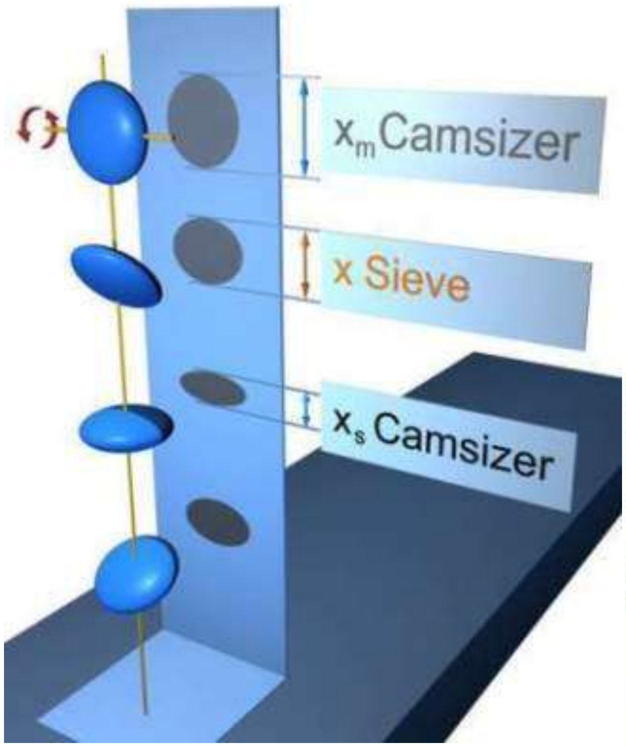
comparison

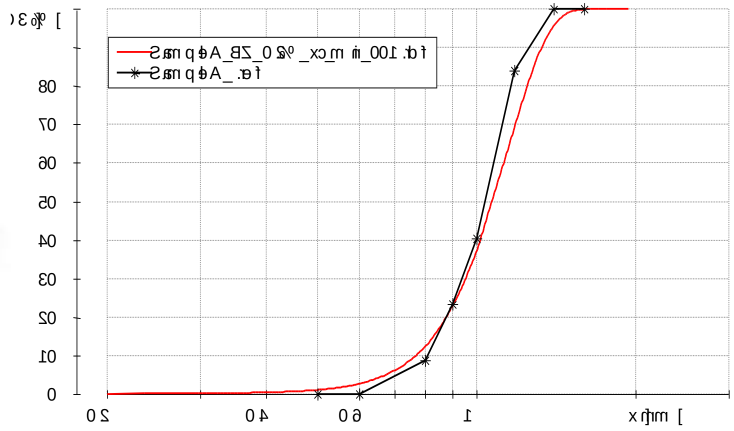
CAMSIZER-measurement **$x_{c\,min}$** (**red**)
and sieving * (**black**)

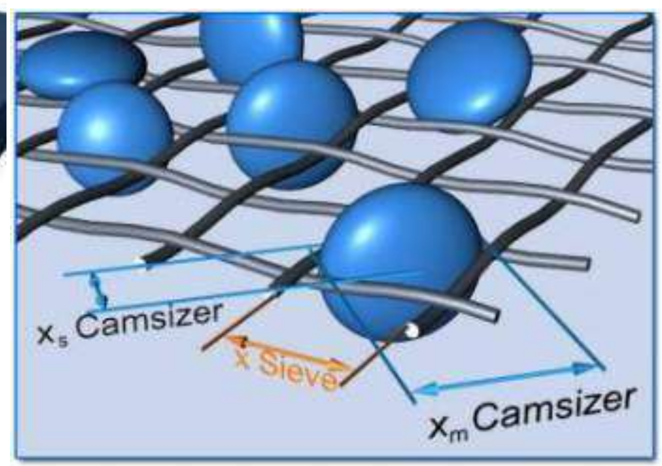
Lenticular particles

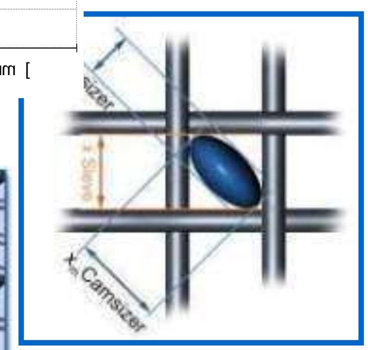


Lenticular particles

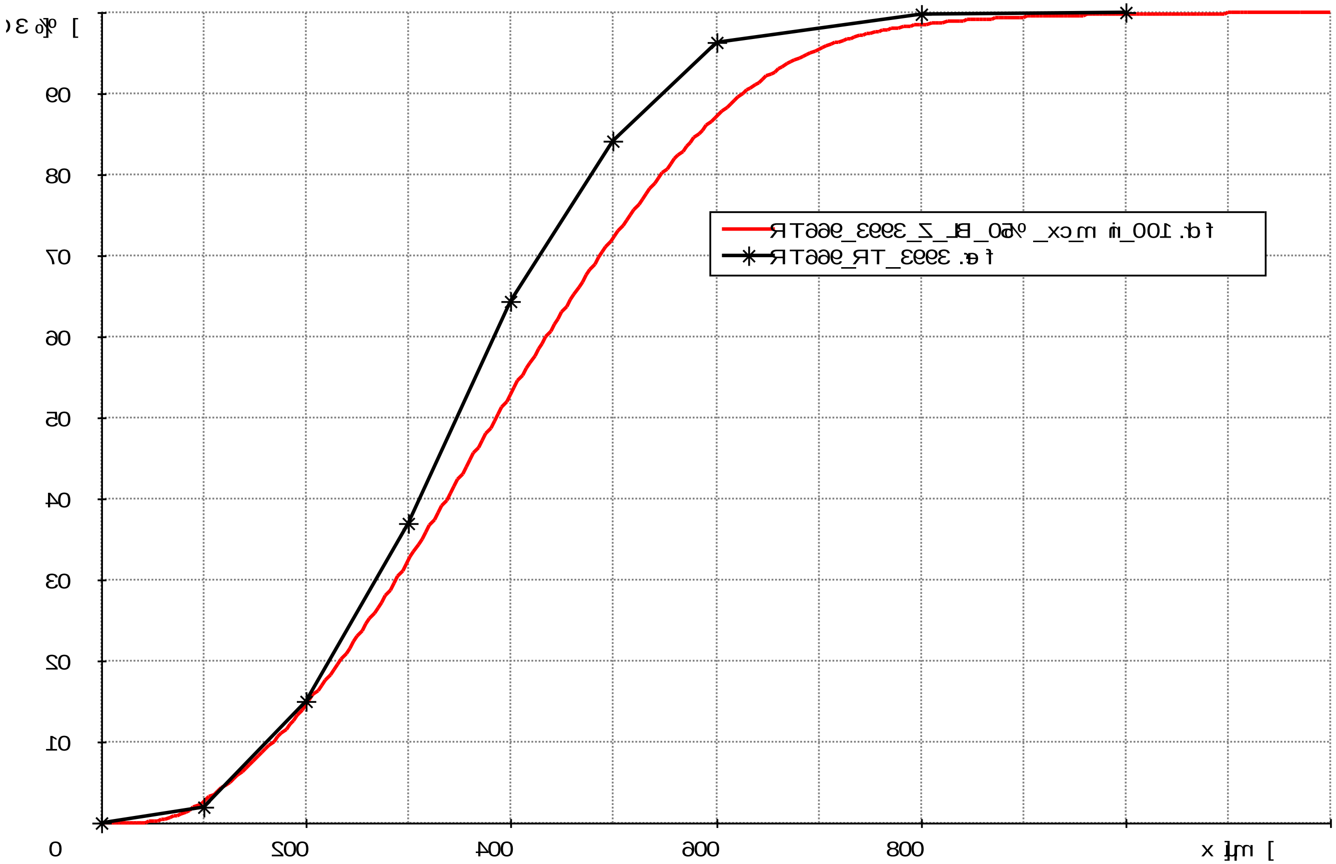




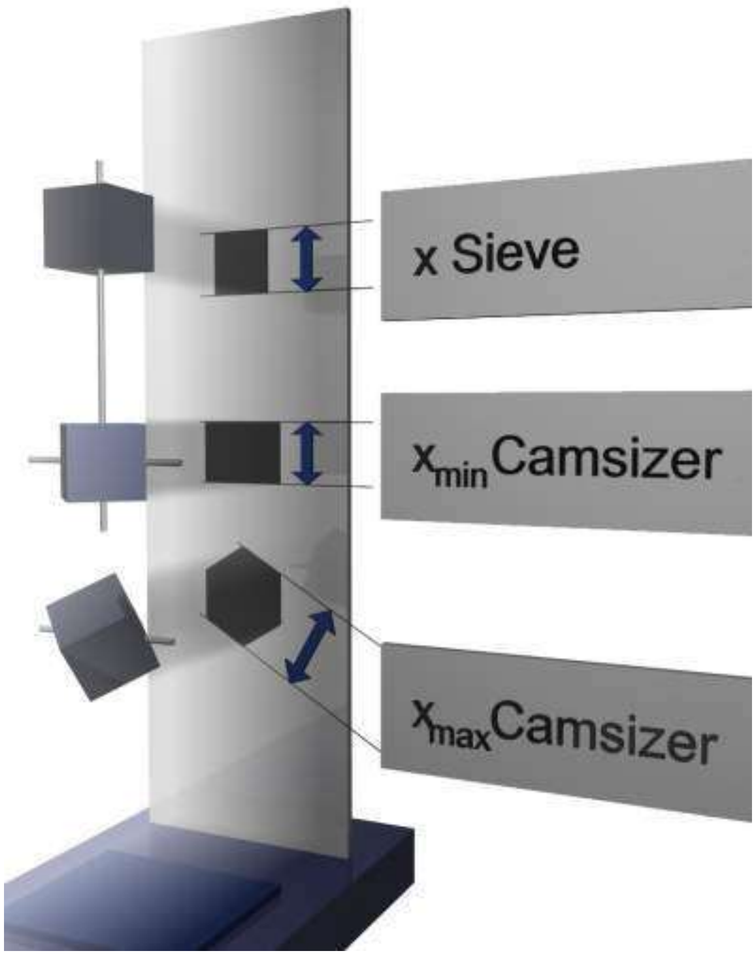


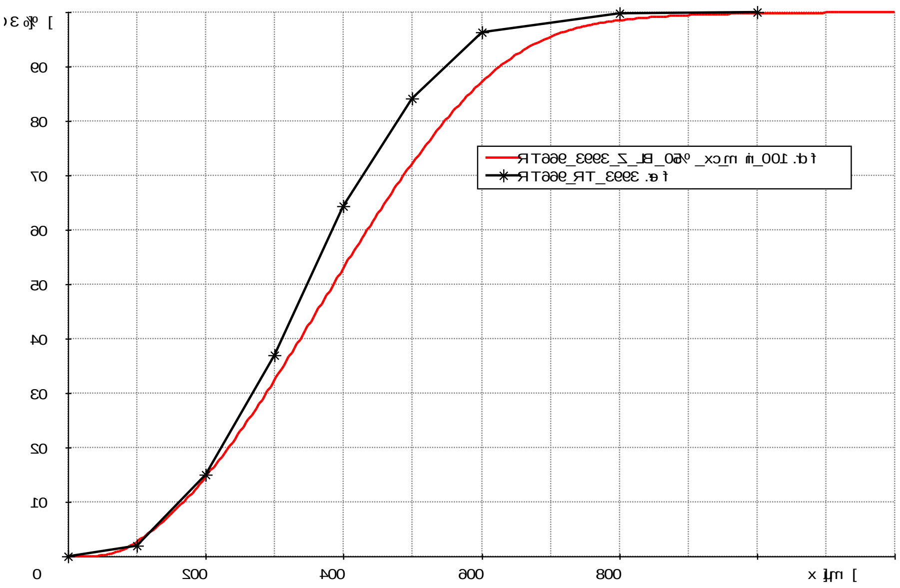


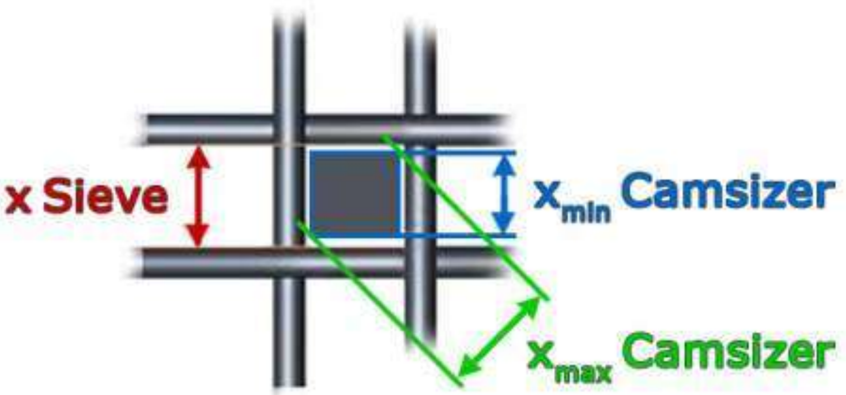
Angular particles (coal, sugar)



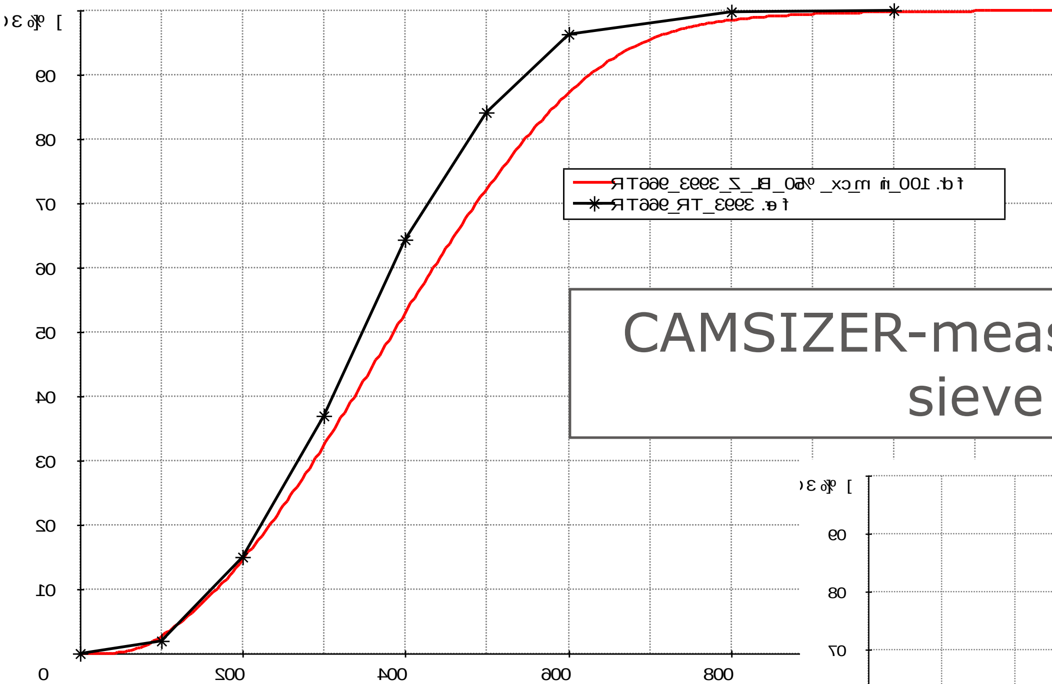
Cube/ angular particles





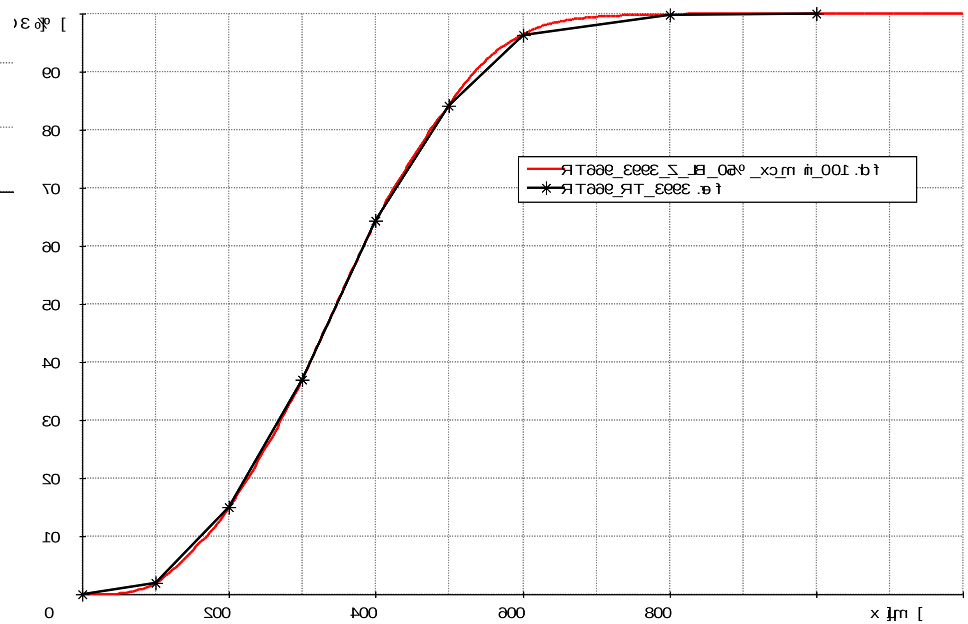


Digital imaging <-> sieving



angular particles without fitting

CAMSIZER-measurement $x_{c\ min}$ (**red**)
sieve analysis * (**black**)



Need only set up one correlation for each process/product.

Shape: aspect ratio

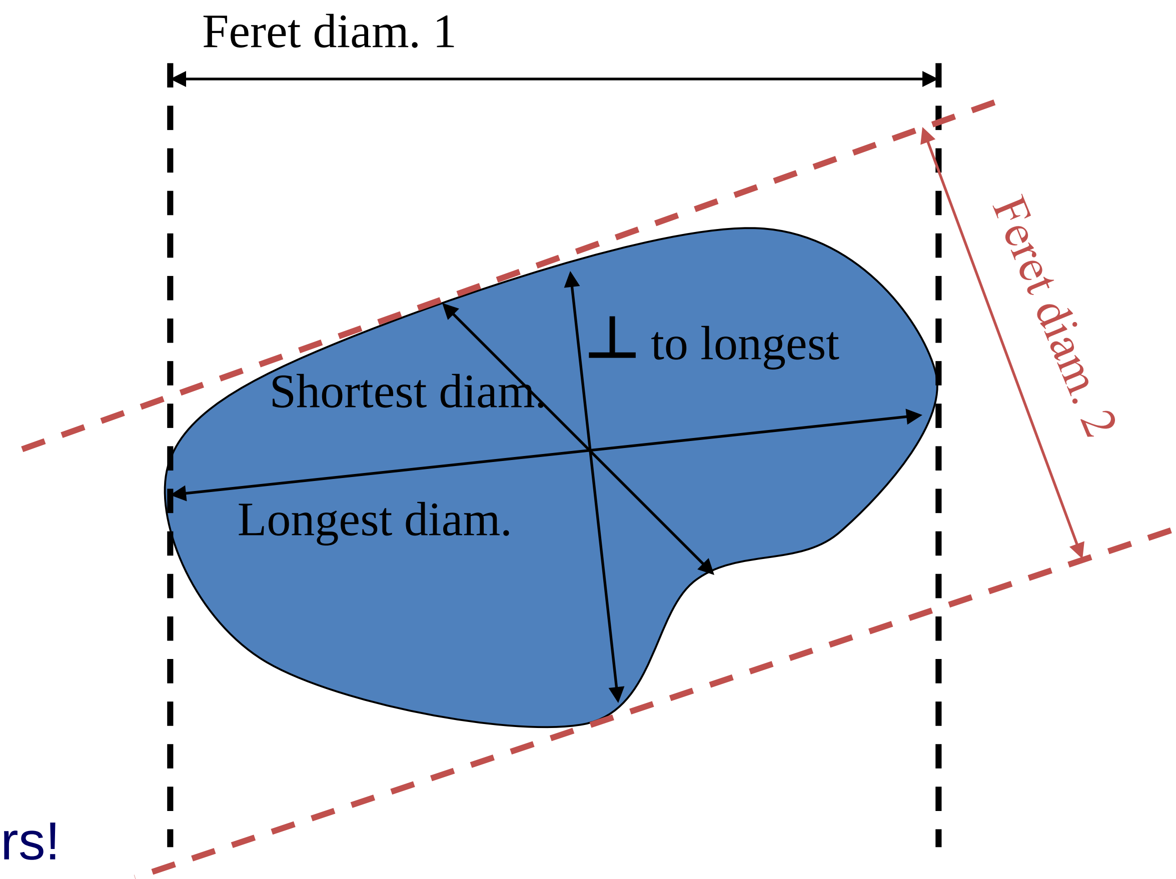
Aspect ratio

= shortest diam / longest diam

= ⊥ to longest diam / longest diam

= shortest Feret diam / longest Feret diam

= three different numbers!



More shape descriptors

Roundness

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

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

Roughness

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

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

Aspect Ratio

Ratio of length over width.

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

Compactness

Ratio of area over convex perimeter:

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

Fractal Dimension

Numerical characterization of irregular contours through fractal geometry.

$$P = P_{\varepsilon}\delta^{1-D}$$

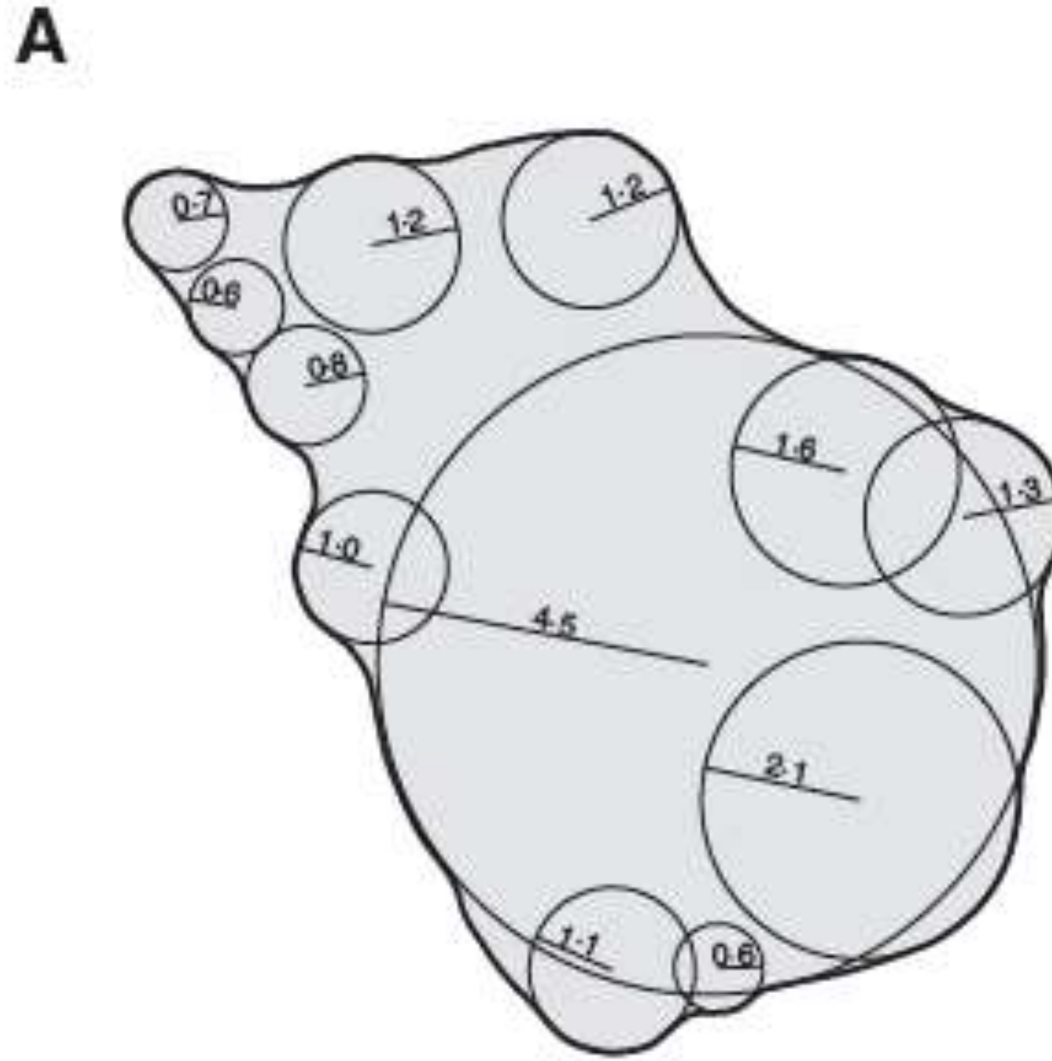
D is the Fractal Dimension, d is the unit length of the scale used for the measurement and P is the perimeter of the object ($1<D<2$).

Sphericity

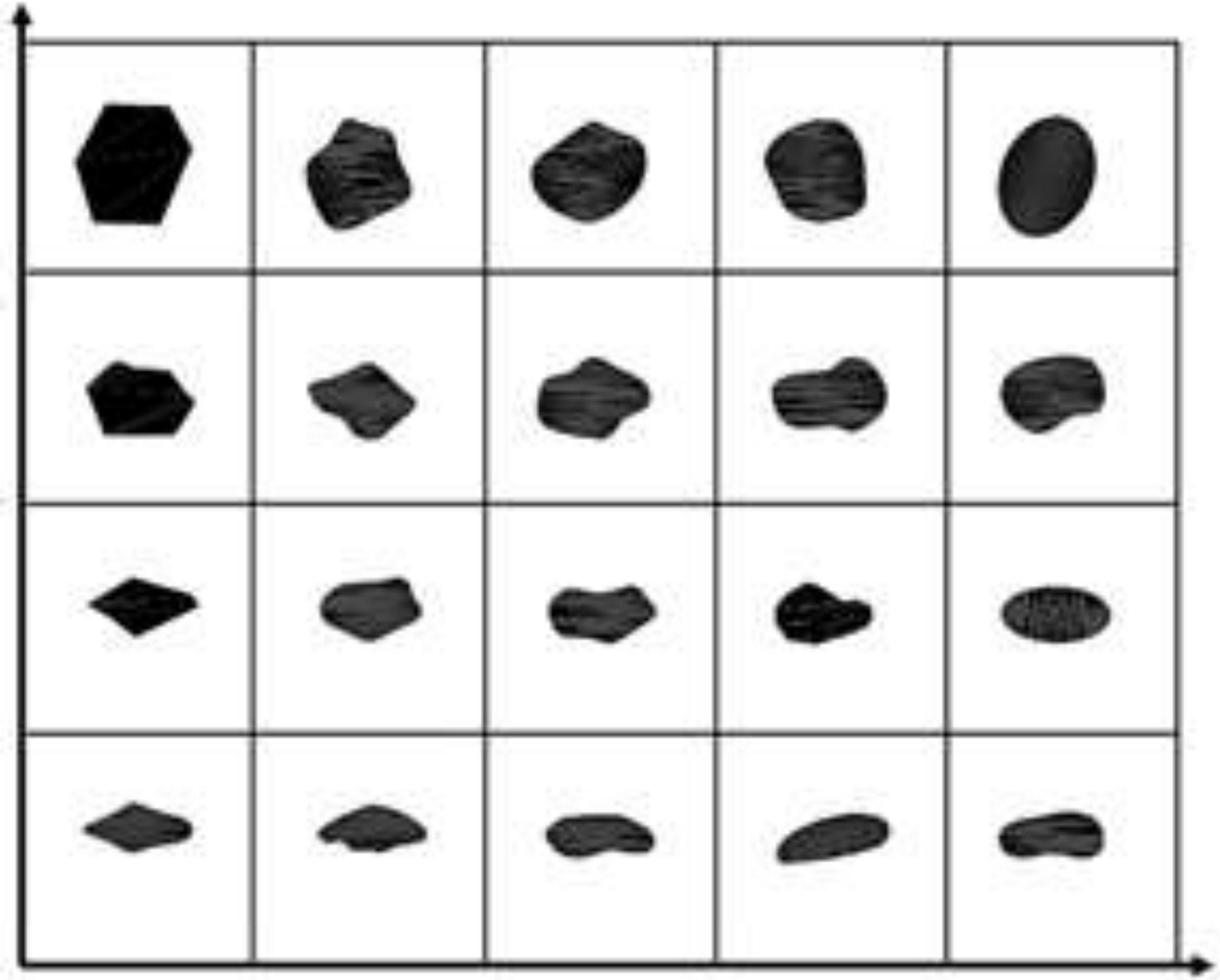
Estimate of the sphericity of an object:

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

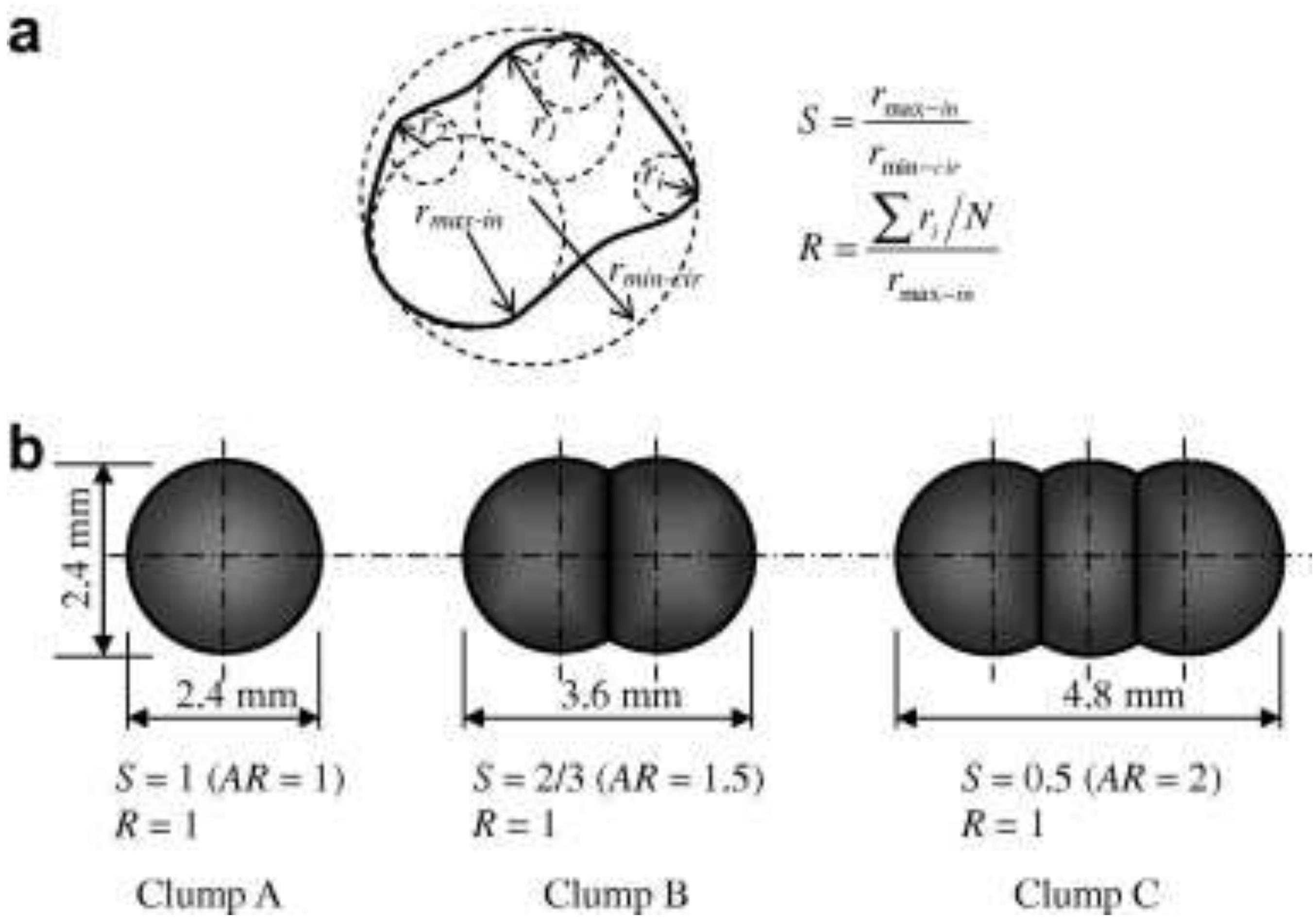
Advanced Shape Descriptors



Krumbein roundness

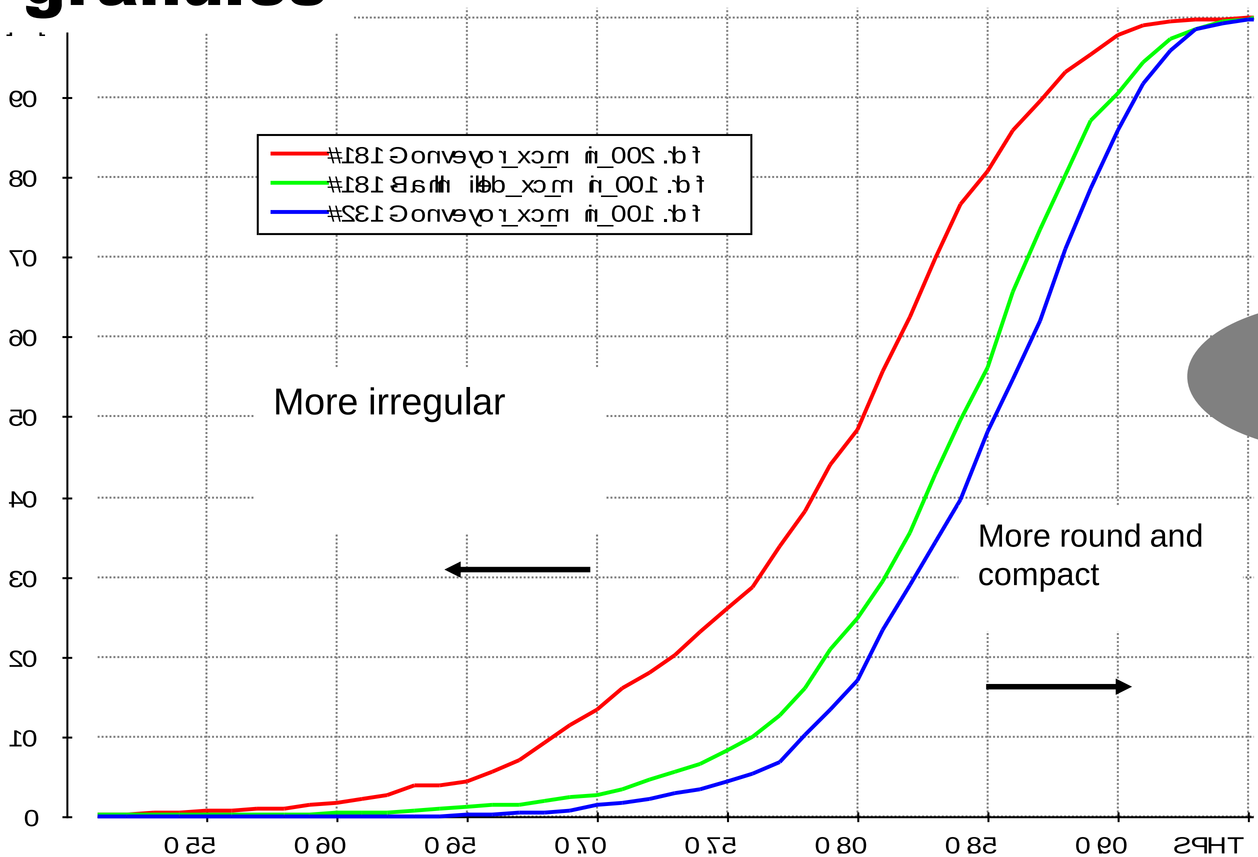


Clumps are "round"

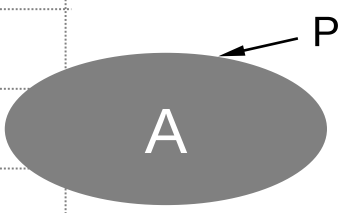


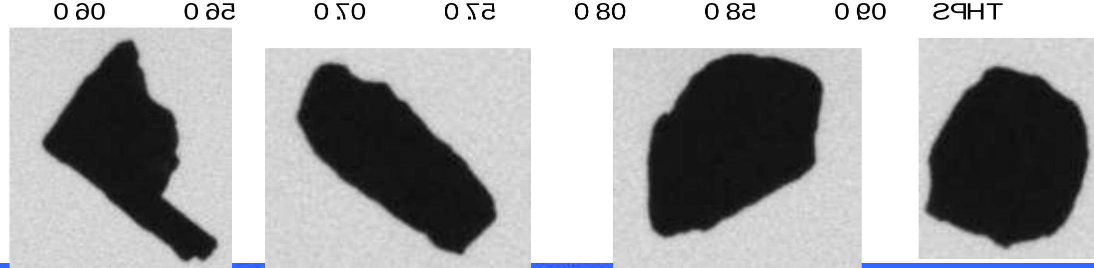
Shape analysis

Roofing granules

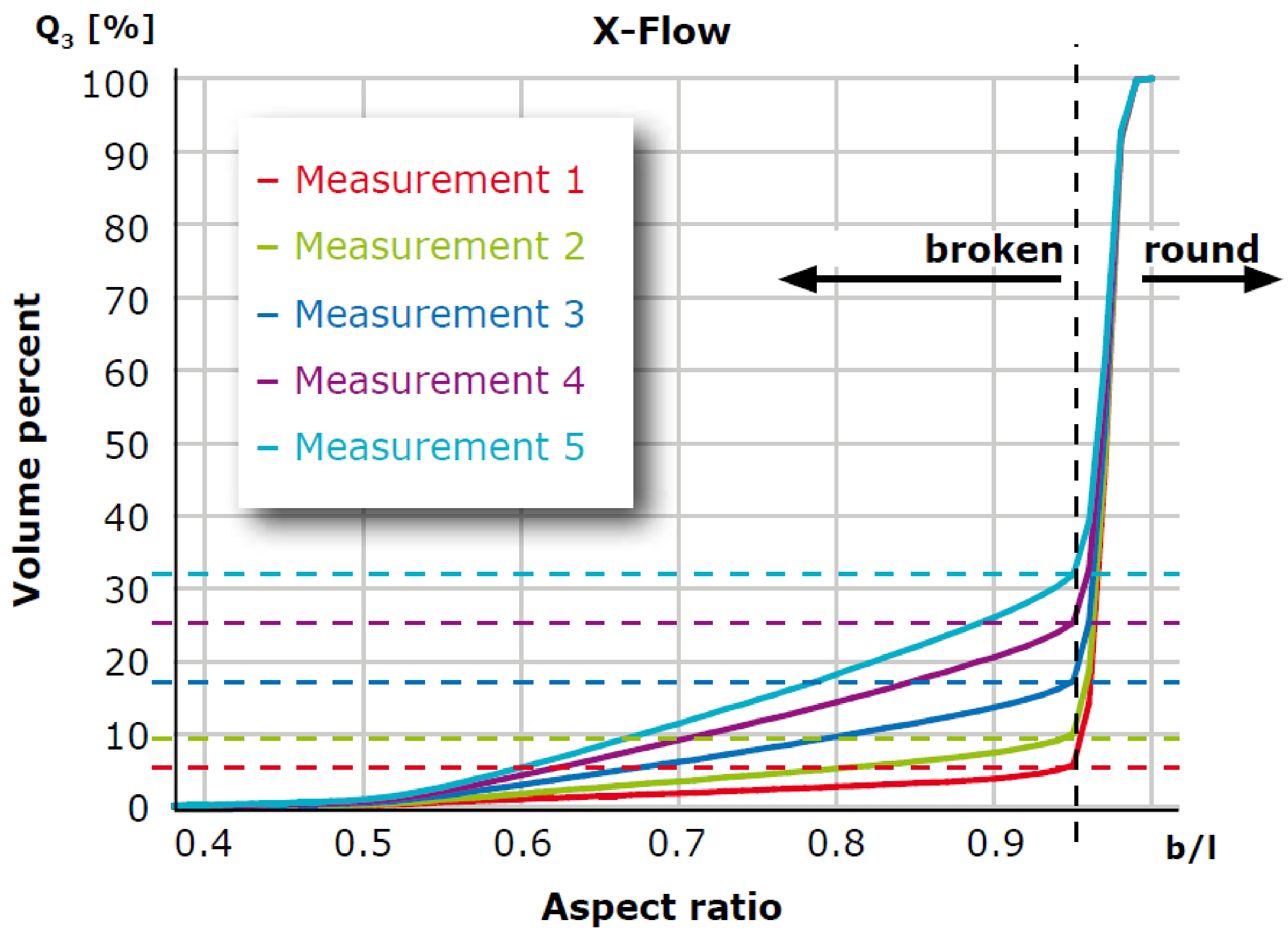


$$\frac{4\pi A}{P^2}$$





Mixture of types by shape



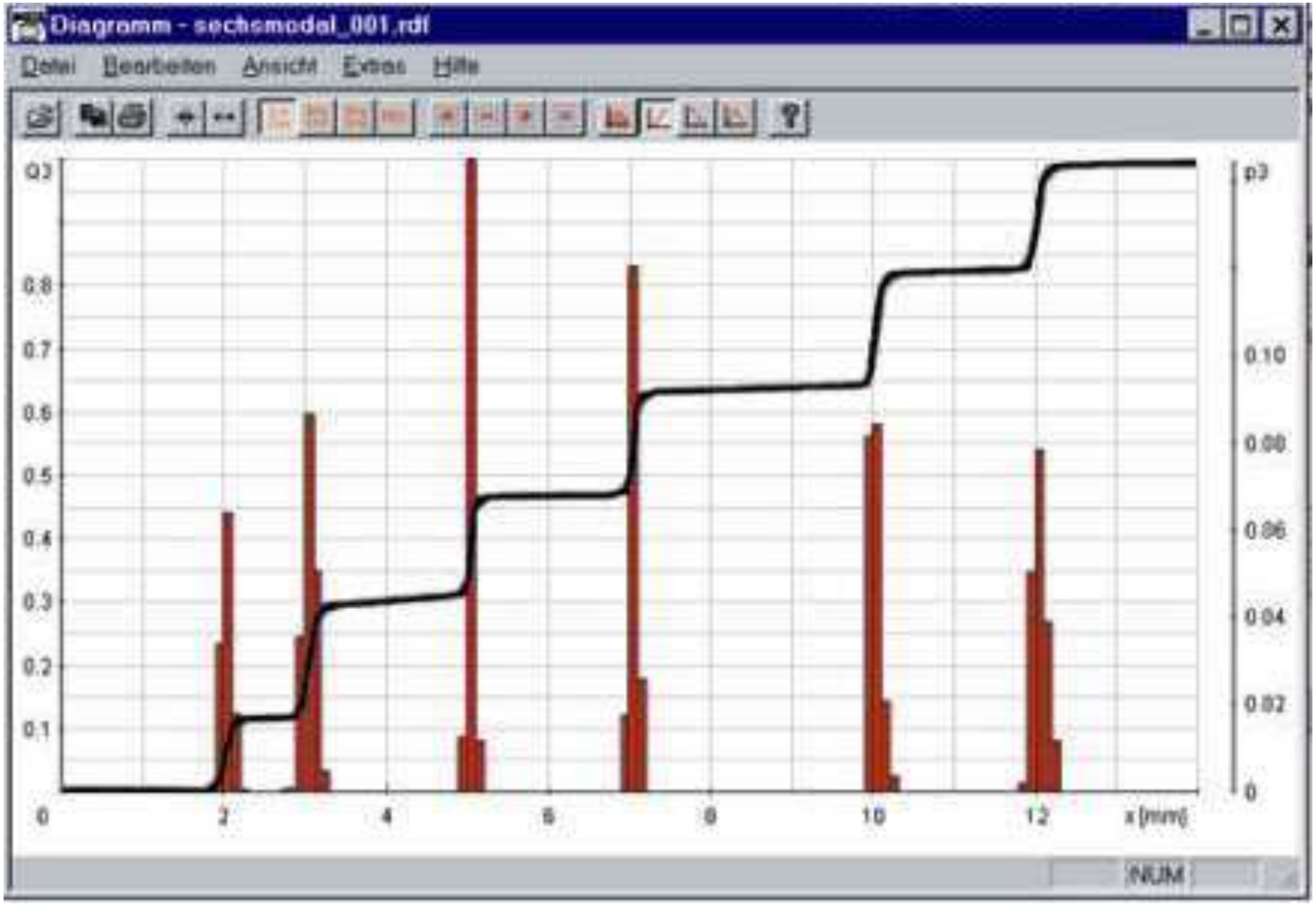
Data Quality

Resolution

Accuracy

How many particles?

Resolution



mixture of six sizes of grinding balls

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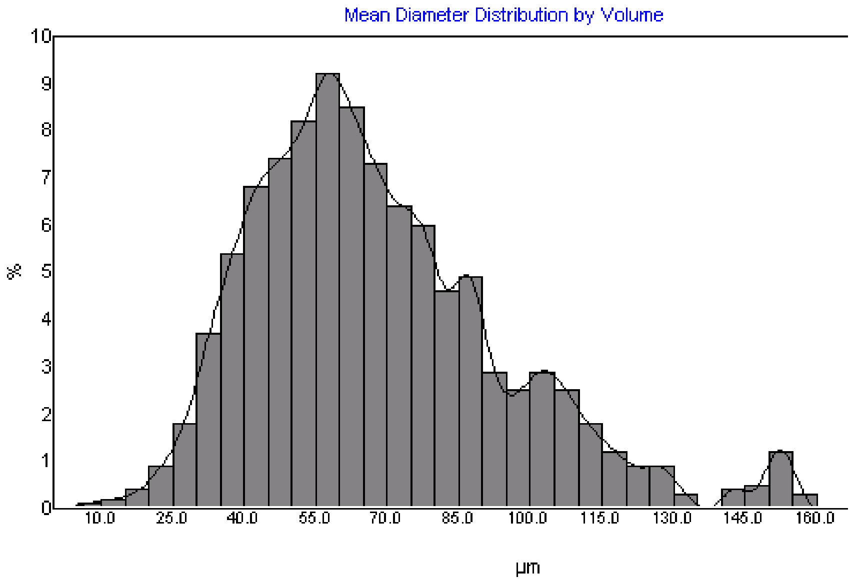
Accuracy

- **Is that the "real size"?**
- **Image analysis uses actual pictures to extract size.**
- **Calibration with a reticle.**

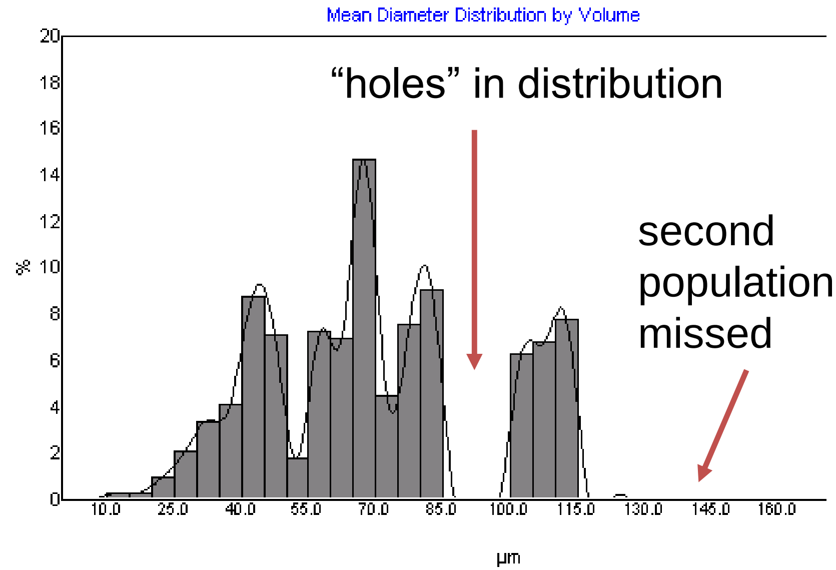
Effect of number of particles

20,000 particles	200 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			



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

<USP> 776: Mean

$$u = z\left(\frac{s}{\sqrt{n}}\right) \qquad n = \left(\frac{sz}{u}\right)^2$$

u=uncertainty
z=confidence coefficient (often ~2, see a statistics book)
s=standard deviation of distribution (width)
n=number of particles measured

Example: for uncertainty of ± 5 µm with st dev= 20 µm
Must measure 61 particles

$$n = \left(\frac{sz}{u}\right)^2 = \left(\frac{(20)(1.96)}{5}\right)^2 \approx 62$$

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

<USP> 776: Standard Deviation

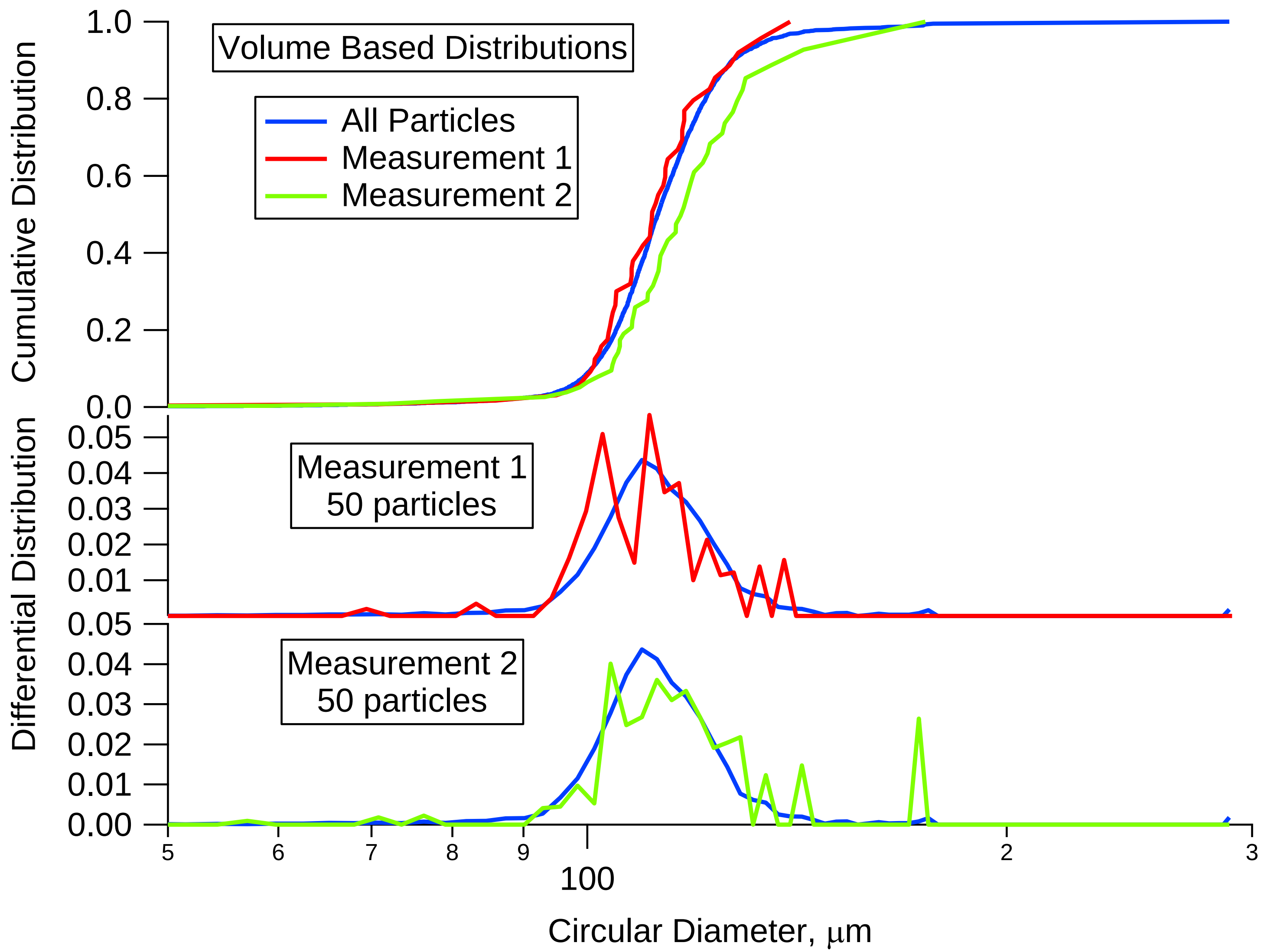
$$\sqrt{s^2\left(\frac{n}{\chi_a^2}\right)} < \sigma < \sqrt{s^2\left(\frac{n}{\chi_b^2}\right)}$$

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

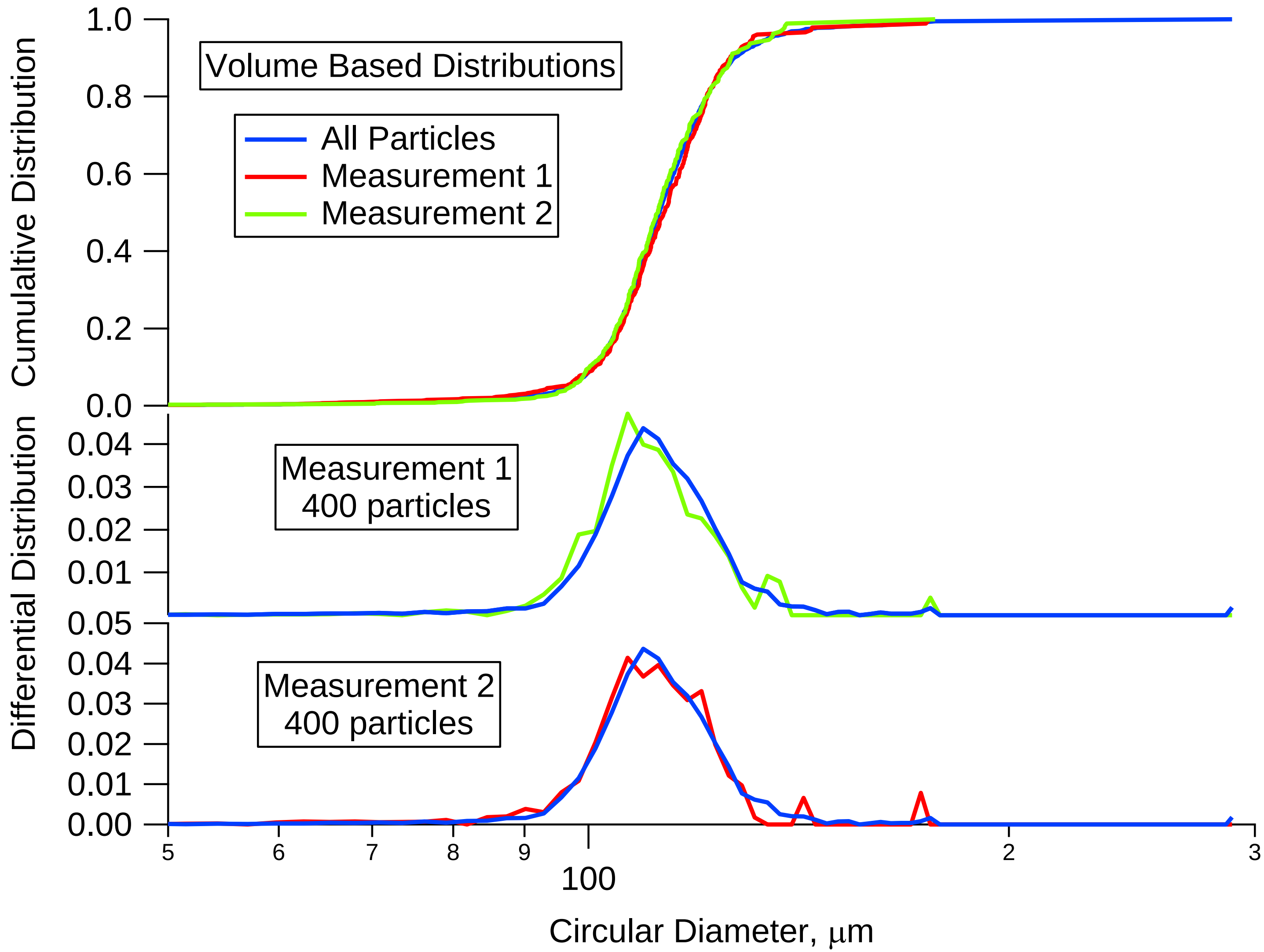
These limits are asymmetric around the standard deviation.

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

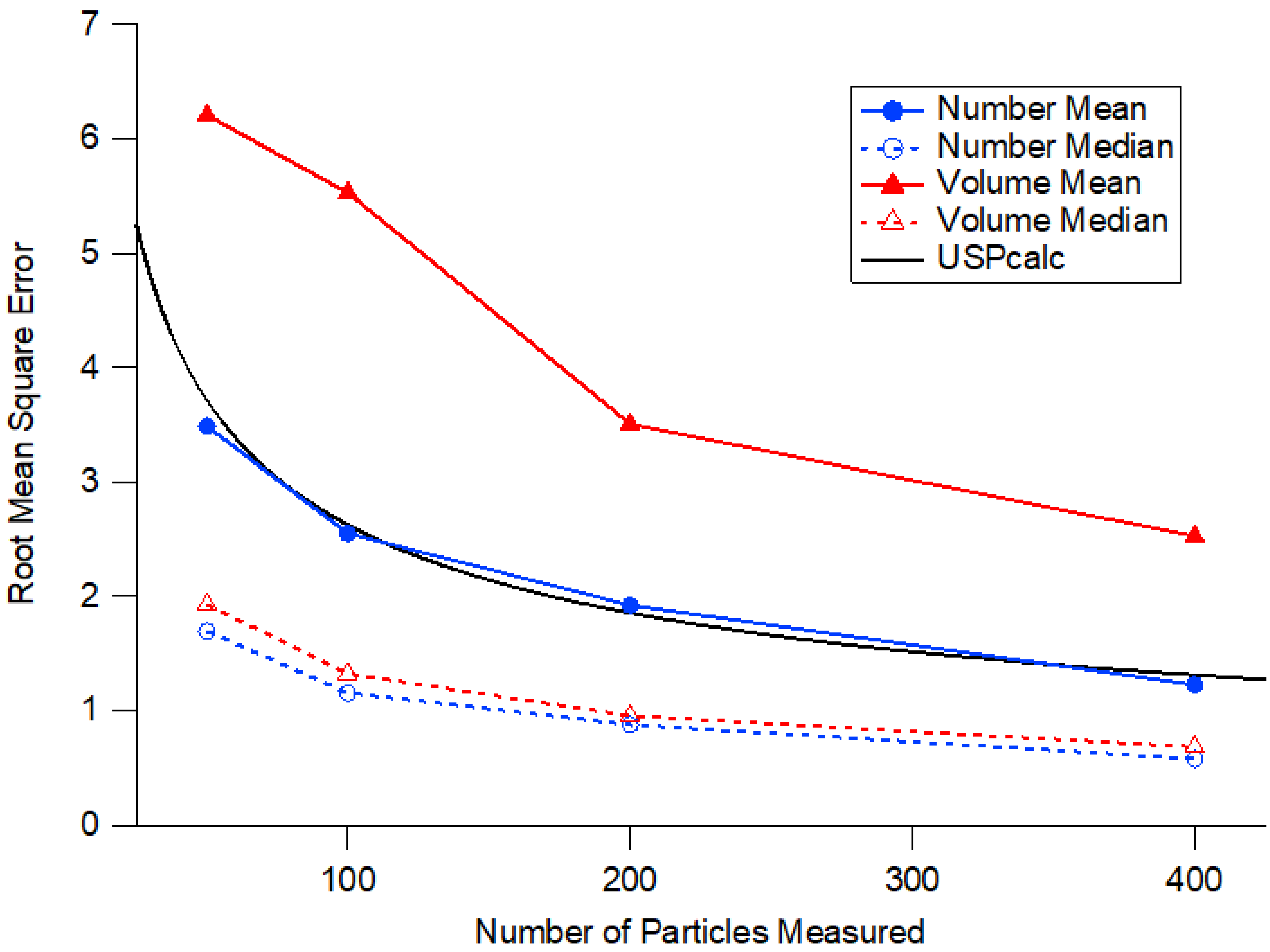
50 particles



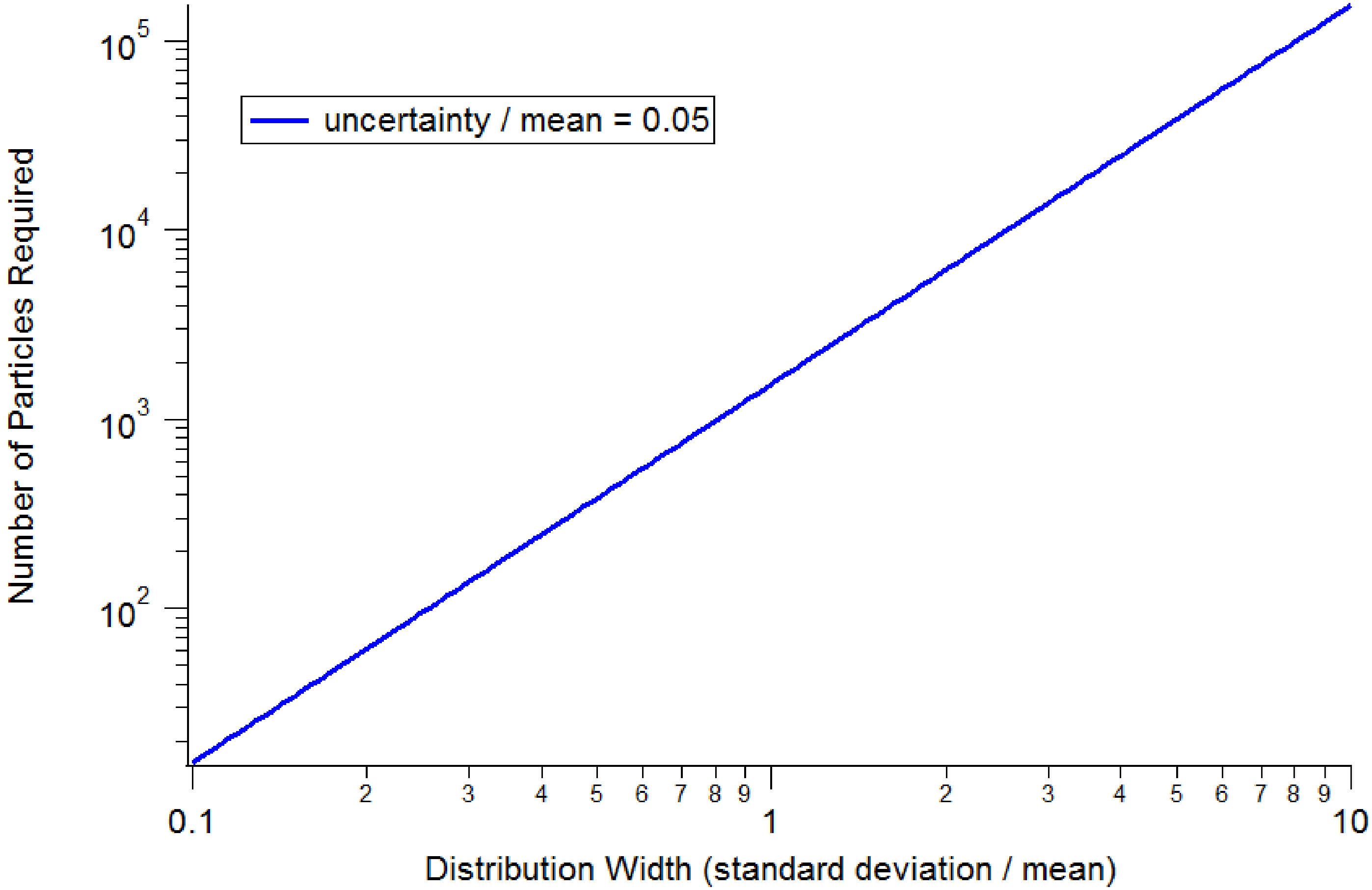
400 particles



Compare With USP



How Many Particles?



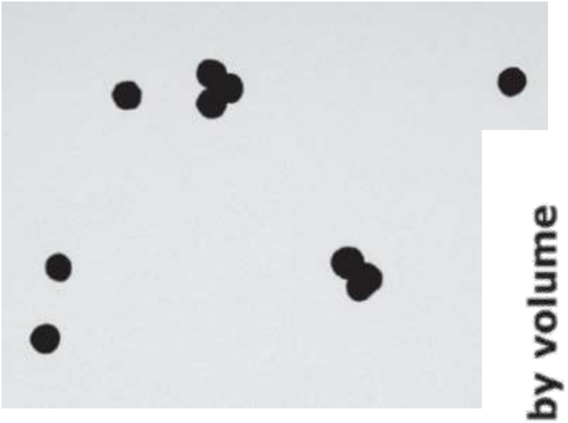
Some materials have a distribution such that SD/Mean ~ 1.
To obtain reliable mean values, measure ~1500 particles.
To obtain more details about the distribution, (10x?) more particles need to be measured.

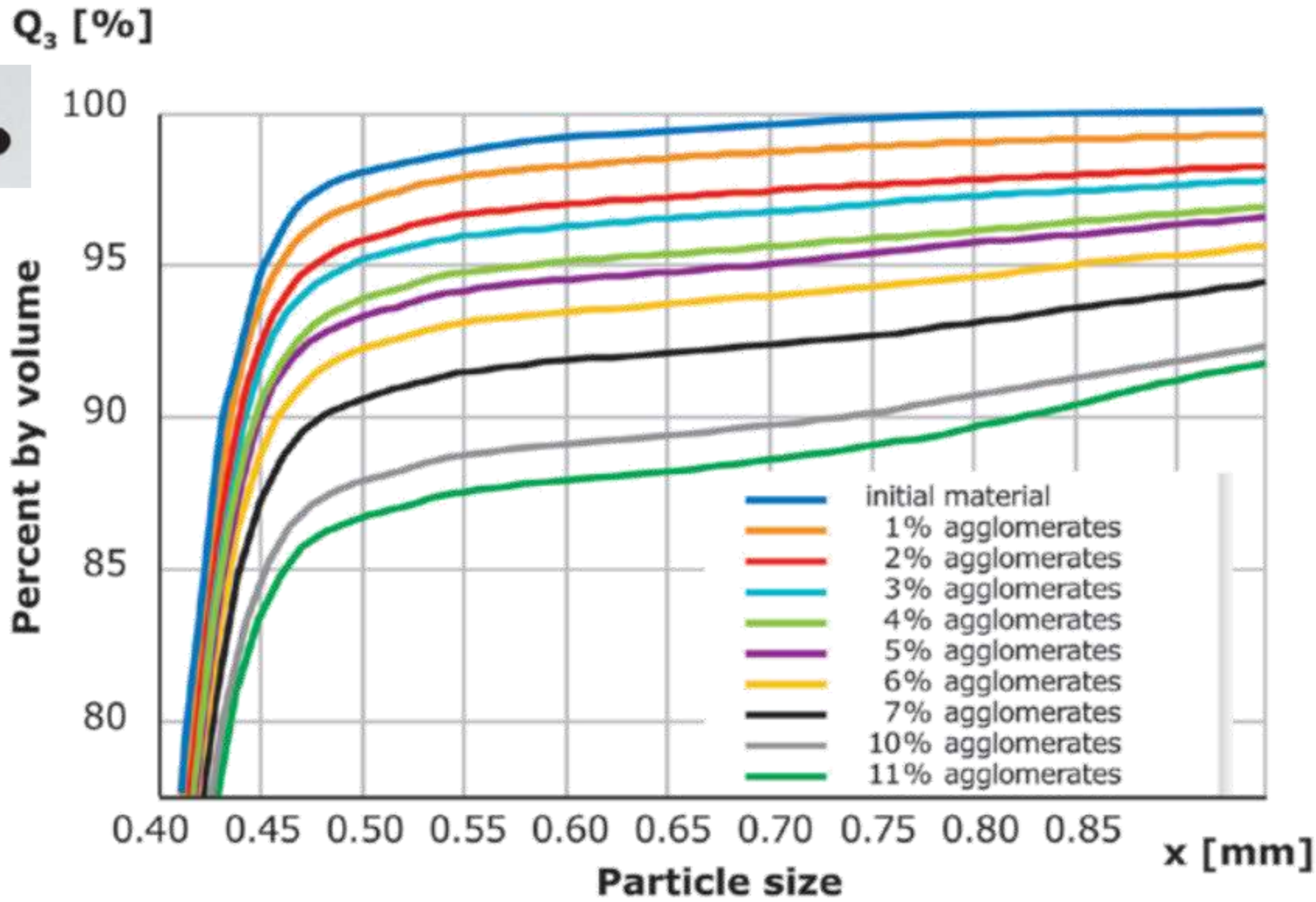
Examples

Cool things not mentioned elsewhere

Agglomerates in powders

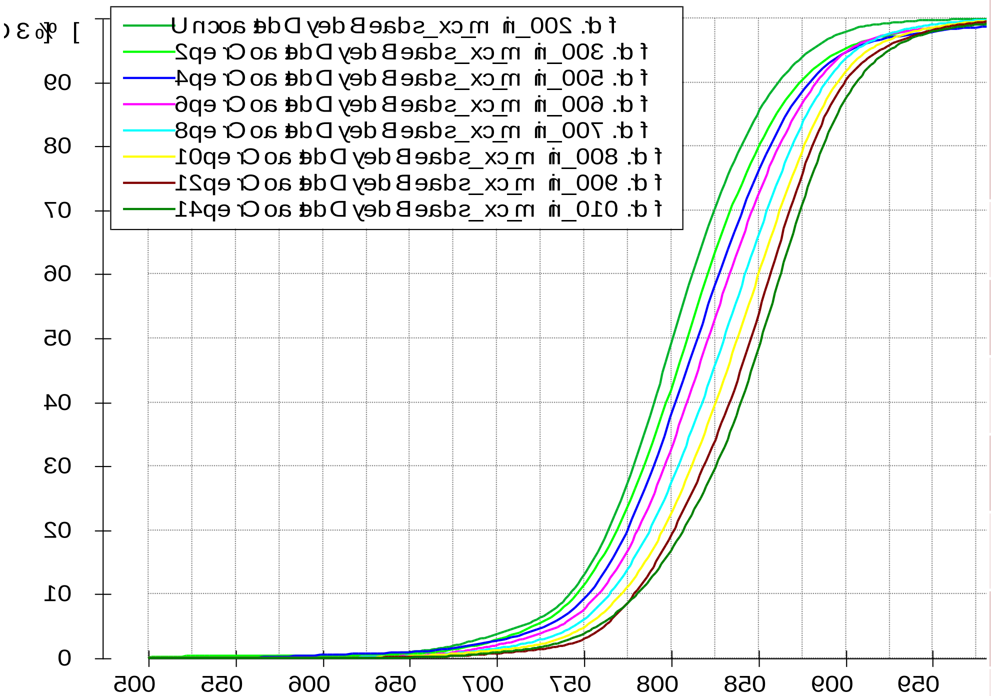
By measuring a lot of particles, can detect a small fraction of aggregates





Coating process

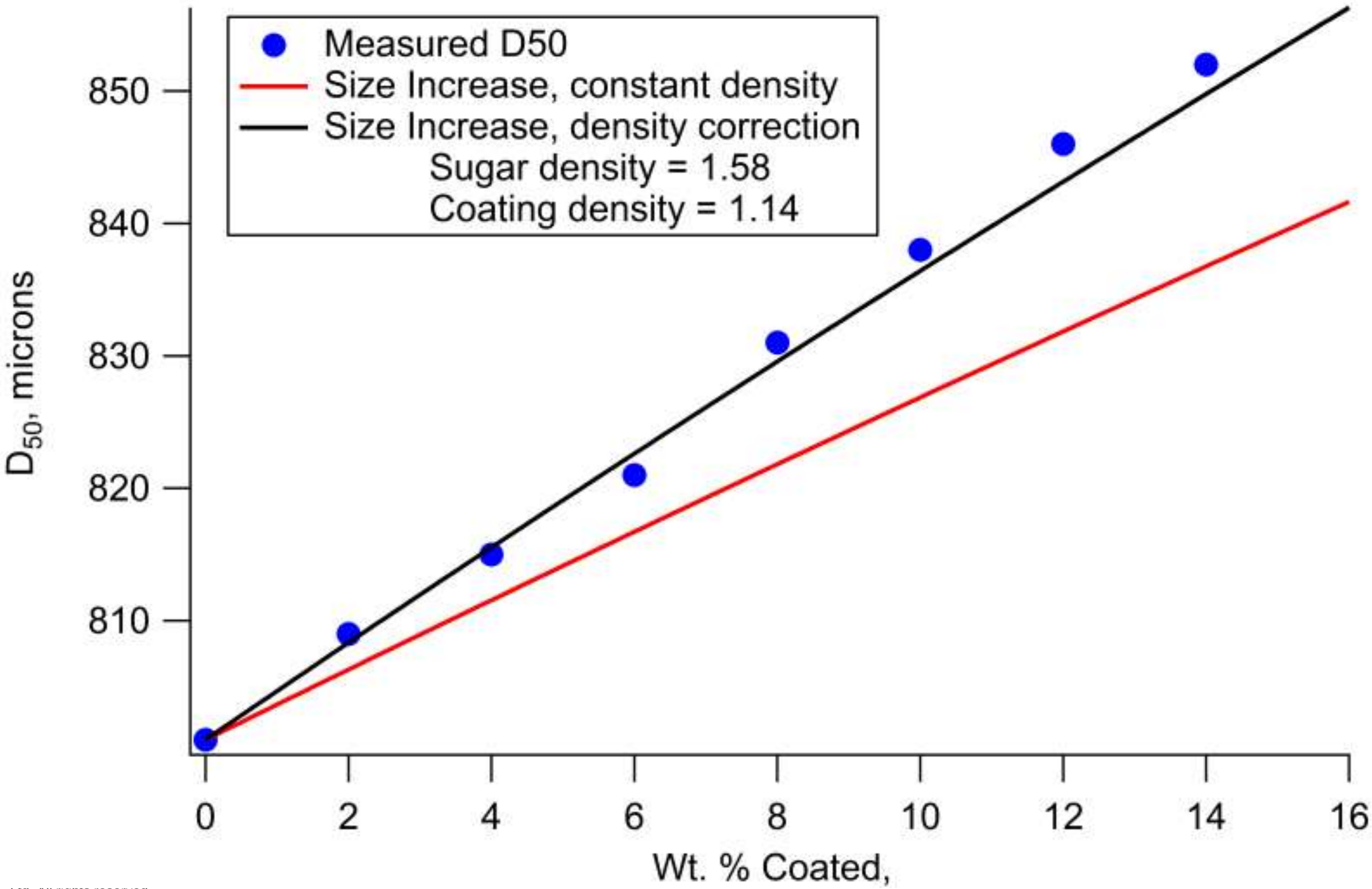
Can resolve 2% increase in coating weight: about 10 microns



Sample	D50, microns	Diff, microns
Uncoated, dyed beads	801	
2%	809	8
4%	815	6
6%	821	6
8%	831	10
10%	838	7
12%	846	8
14%	852	6

Monitoring Coating

Precision requires measuring enough particles.



Wrapping up

Closing comments

Why dynamic image analysis

- **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**

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**

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Static or Dynamic Image Analysis?

- **Dynamic**
  - **Broad size distributions (since it is easier to obtain data from a lot of particles)**
  - **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

- 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

- **Keep reading our newsletter:** https://www.horiba.com/us/en/scientific/products/particle-characterization/request-information/

- **Jeffrey Bodycomb, Ph.D.**
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- **800-446-7422**

Thank you very much for your attention.

HORIBA

Explore the future