CAMSIZER Method Development

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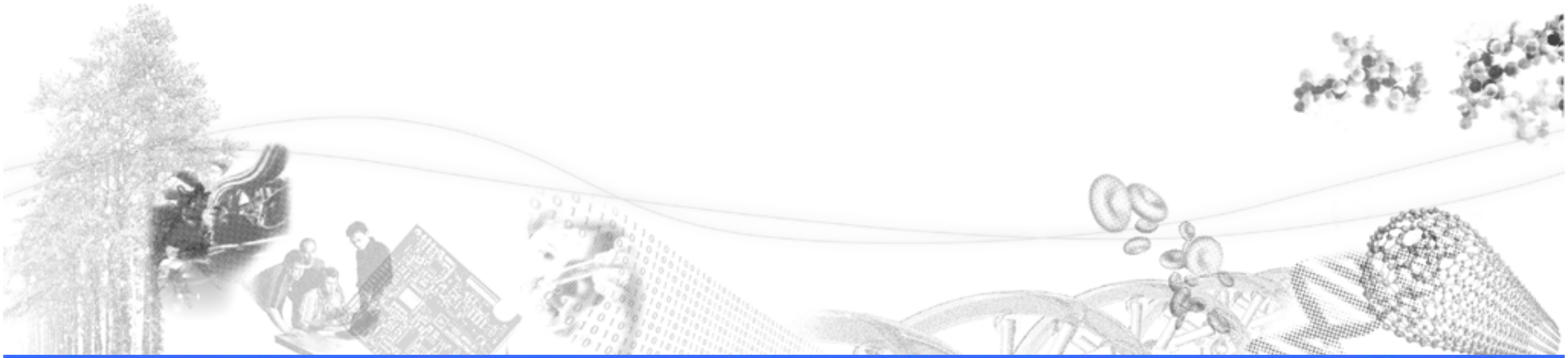
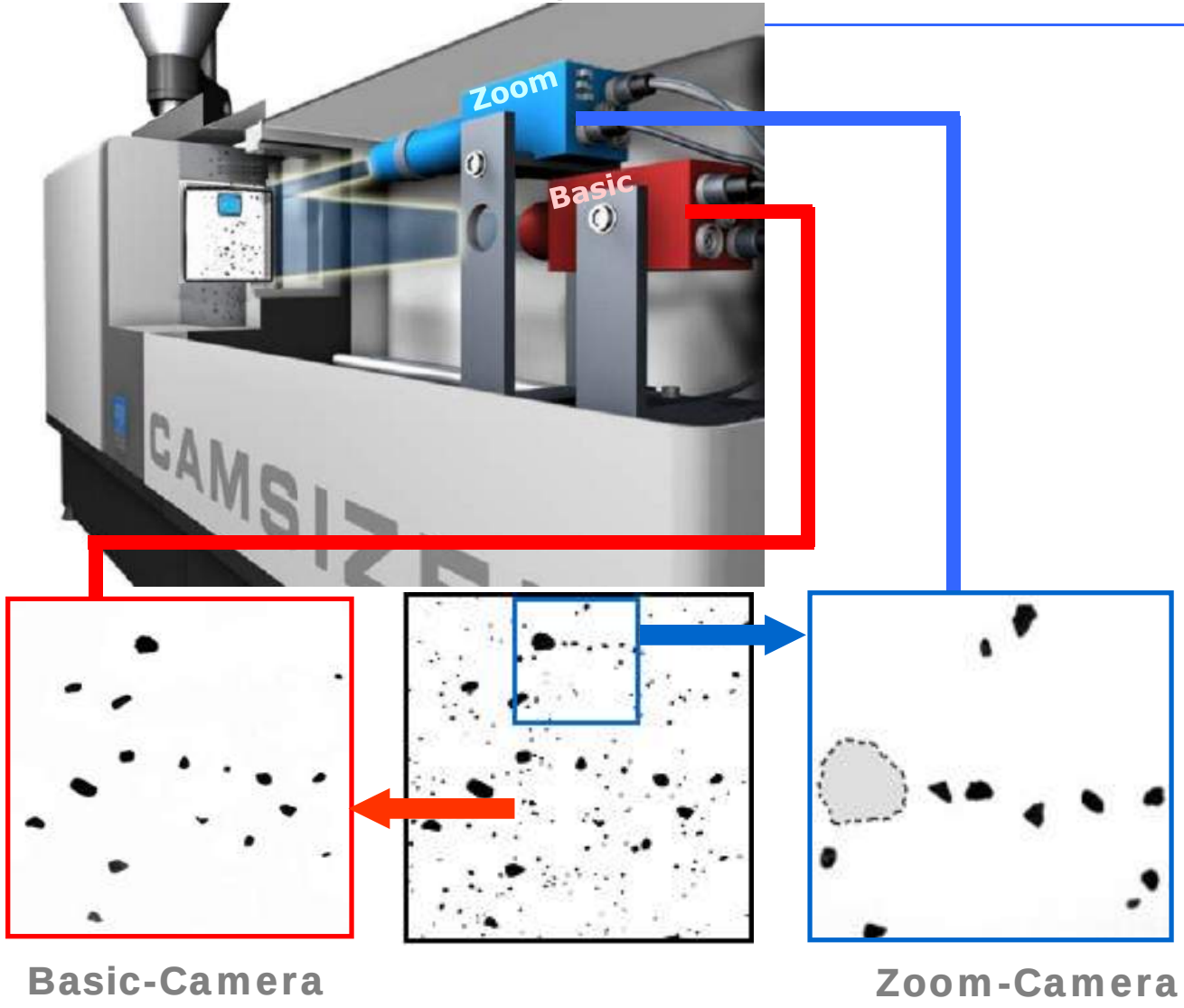
Contents

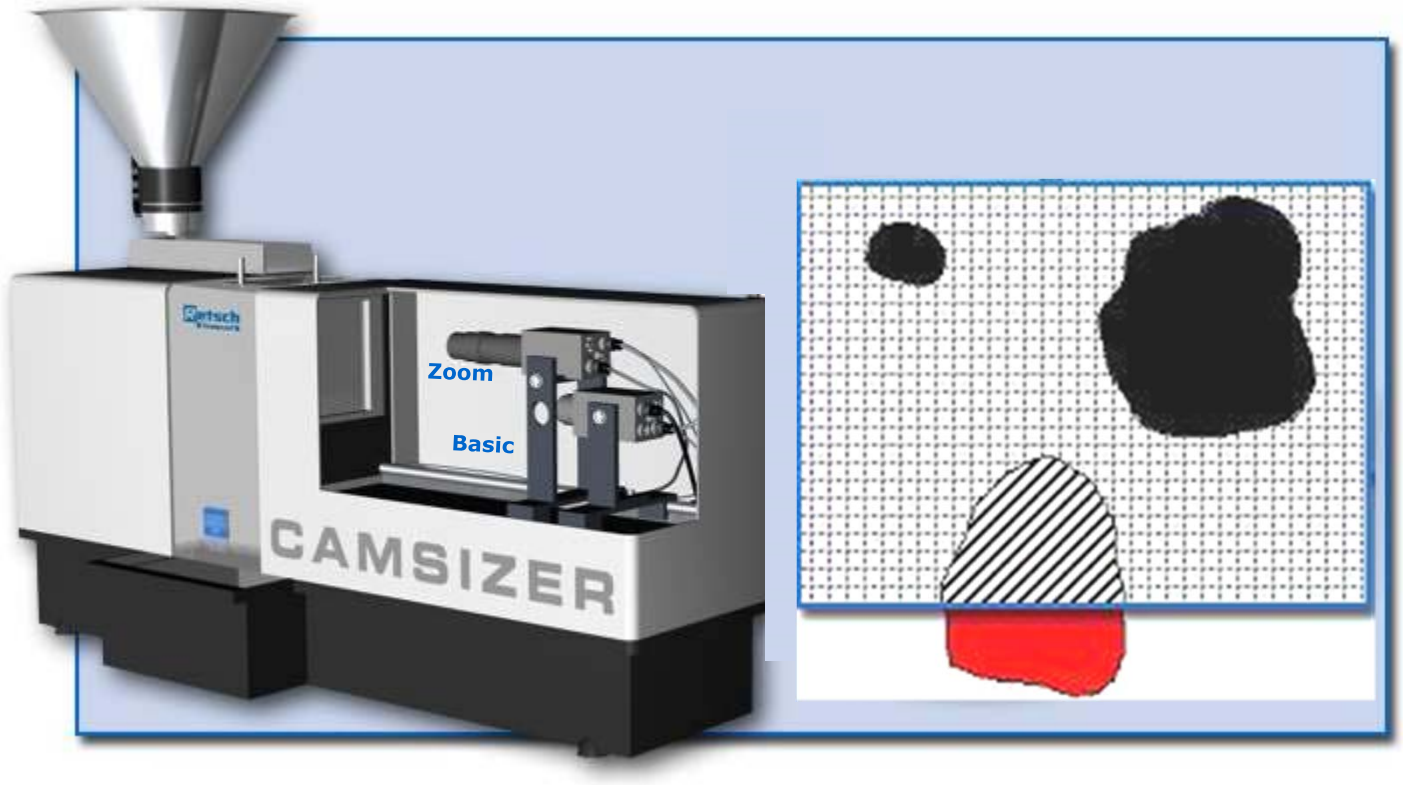
- Overview of CAMSIZER
- Sampling Techniques
- CAMSIZER Hardware
- Size and Shape Parameter Choices
- Software Set-ups

Quick Overview of CAMSIZER



Two-Camera-System





Advantages

- precise frame taking of particle projections (60 frames/second)
- large dynamic measuring range: **30µm to 30mm**

Camera Resolution

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Detection of particles

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

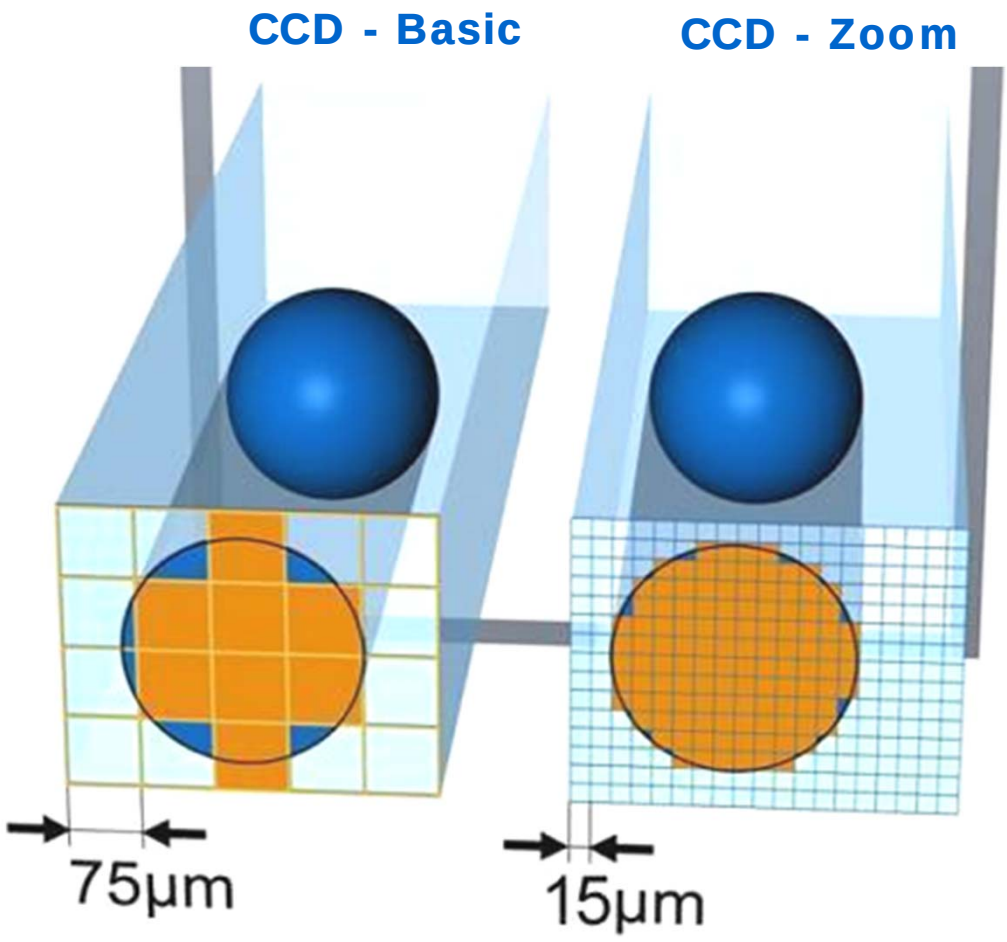
Basic Camera Range:
240 um – 30000 um

Zoom Camera Range:

30 um – 1250 um

Overlapping Range:

625 um – 1250 um



Range of use

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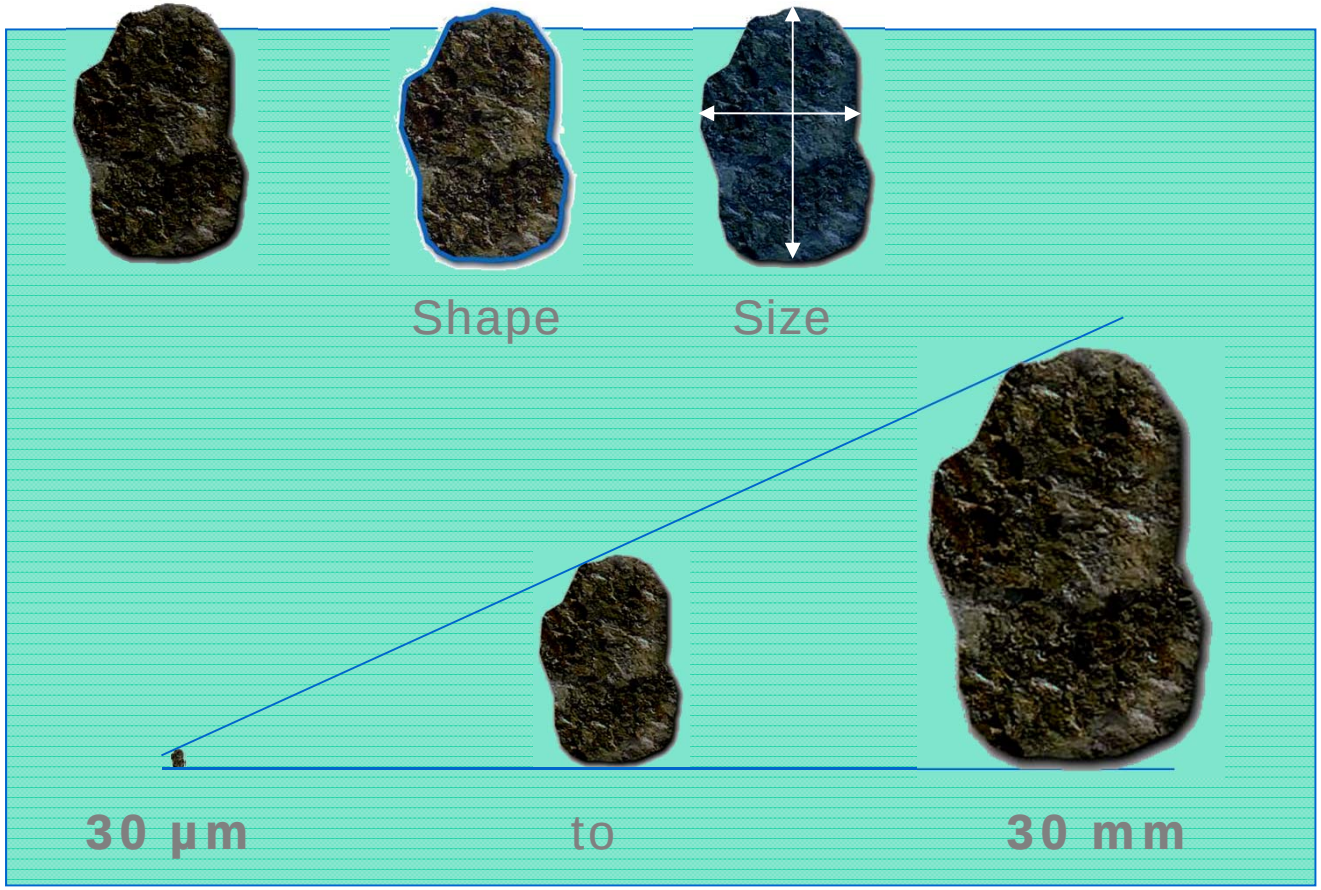

CAMSIZER Samples Must Be…

1. In dry state
2. Free flowing
3. Lager than 30 um
4. Preferably not cohesive and/or statically charged
    a) Flow-agent can be added (Fumed Silica)
    b) Ionizer can be used
    c) Ultrasonic probe can be used

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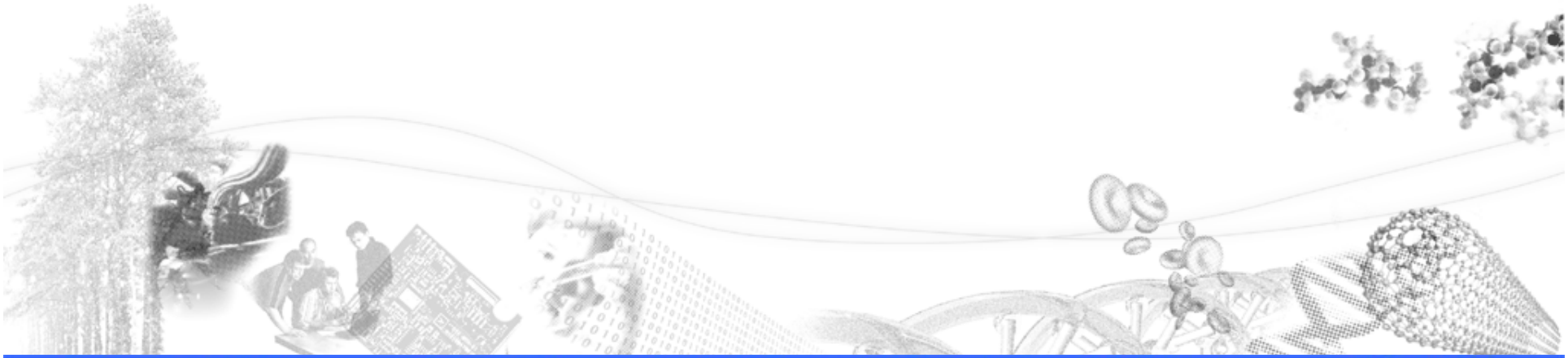
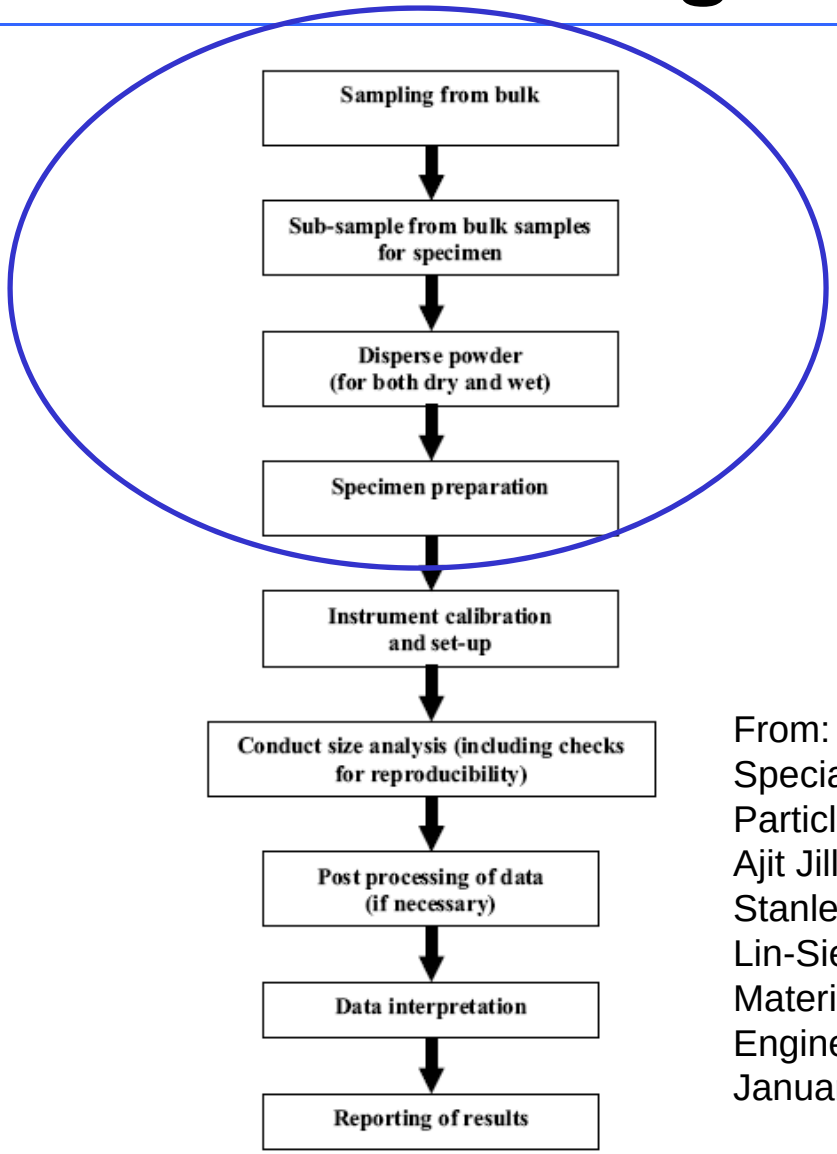
Good Sampling Practices

Learn to Love Your Riffler!



Particle Sizing Workflow



From: NIST Recommended Practice Guide
Special Publication 960-1
Particle Size Characterization
Ajit Jillavenkatesa
Stanley J. Dapkunas
Lin-Sien H. Lum
Materials Science and
Engineering Laboratory
January 2001

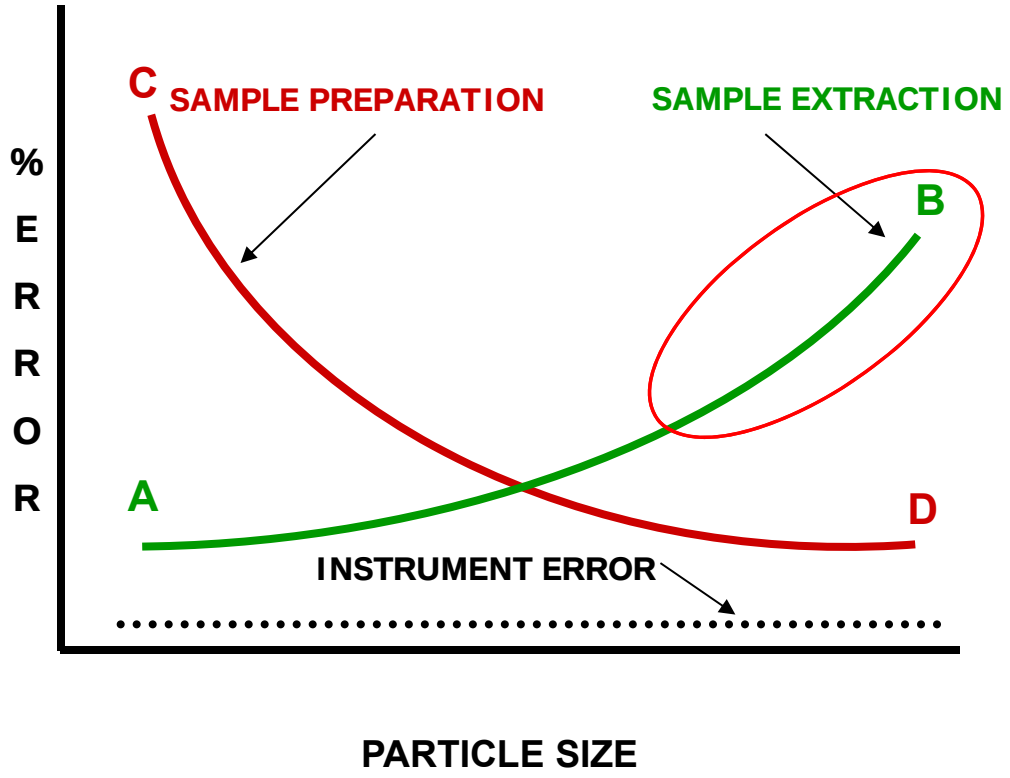
Measurement Error Sources

SMALL PARTICLES

- POTENTIALLY SMALL EXTRACTION ERRORS (A)
- POTENTIALLY LARGE SAMPLE PREP ERRORS (C)

LARGE PARTICLES

- POTENTIALLY LARGE EXTRACTION ERRORS (B)
- POTENTIALLY SMALL SAMPLE PREP ERRORS (D)



INSTRUMENT ERROR IS SMALL AND RELATIVELY CONSTANT

Sample Extraction Overview

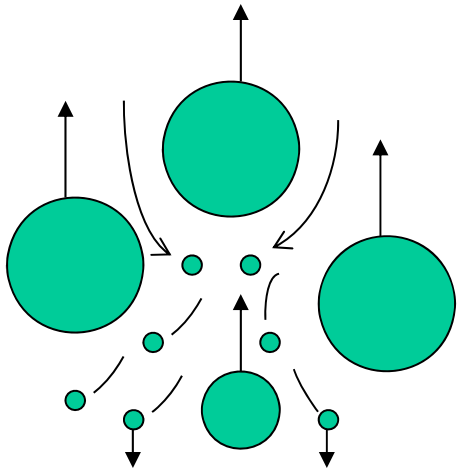
- Sampling from bulk
- Sub-sample for measurement
- Sometimes second sub-sampling
- Perform measurement

Technique: Grab Sampling

- **PLACE SPATULA INTO POWDER**

  EXTRACT SMALL AMOUNT FOR ANALYSIS

  ACCEPTABLE FOR NARROW DISTRIBUTIONS



SEGREGATE LARGE AND SMALL WHEN POLYDISPERSE

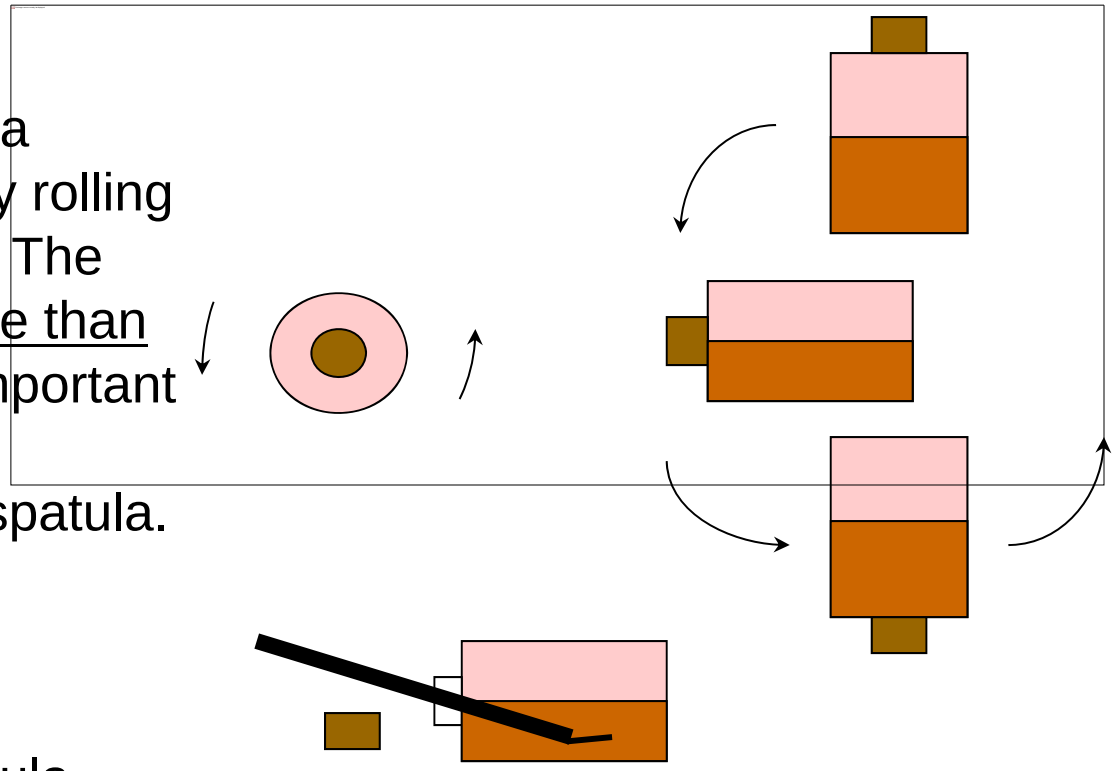
- LARGE PARTICLES PERCOLATE UPWARD
- SMALL PARTICLES GRAVITATE DOWNWARD

EASY METHOD BUT NOT THE BEST METHOD

MOST USED METHOD

Grab Sampling from Bottle

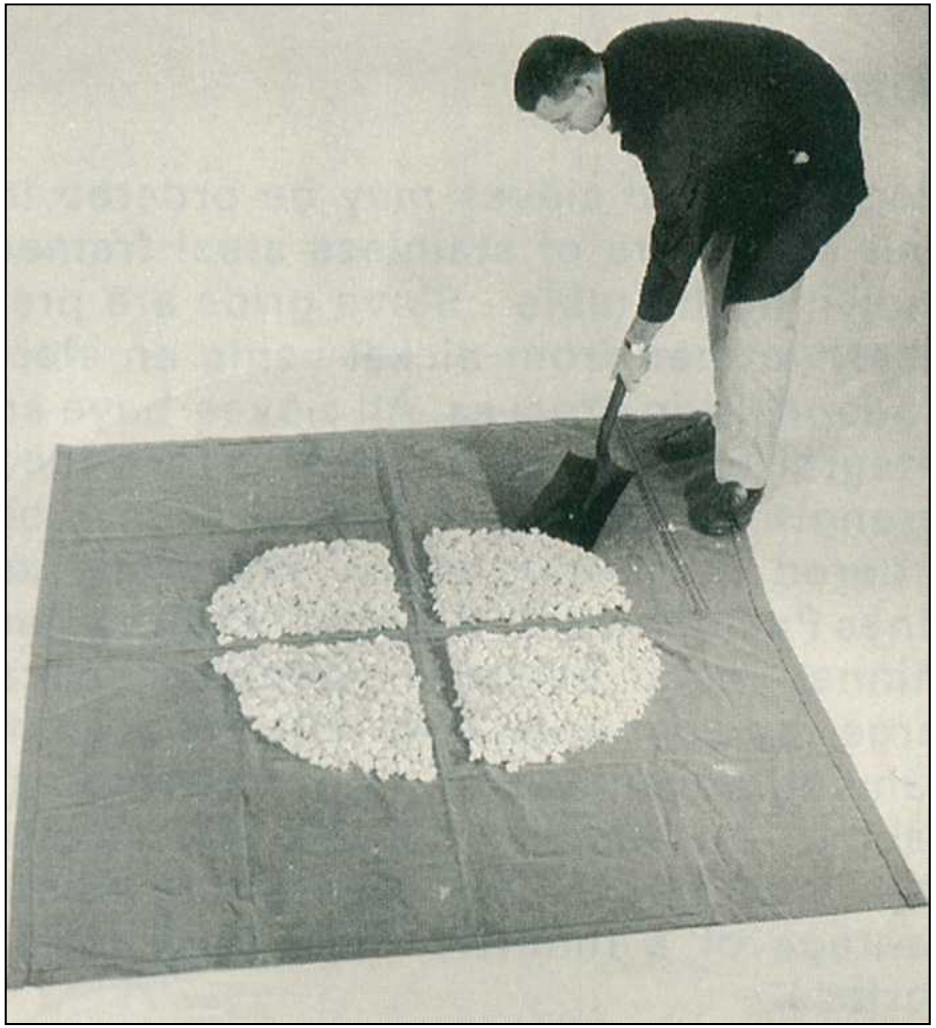
When a powder is stored in a container, it can be mixed by rolling and tumbling the container. The container should not be more than half to two-thirds full. It is important to perform this action before "grabbing" a sample with a spatula.



Then pull sample with a spatula…..

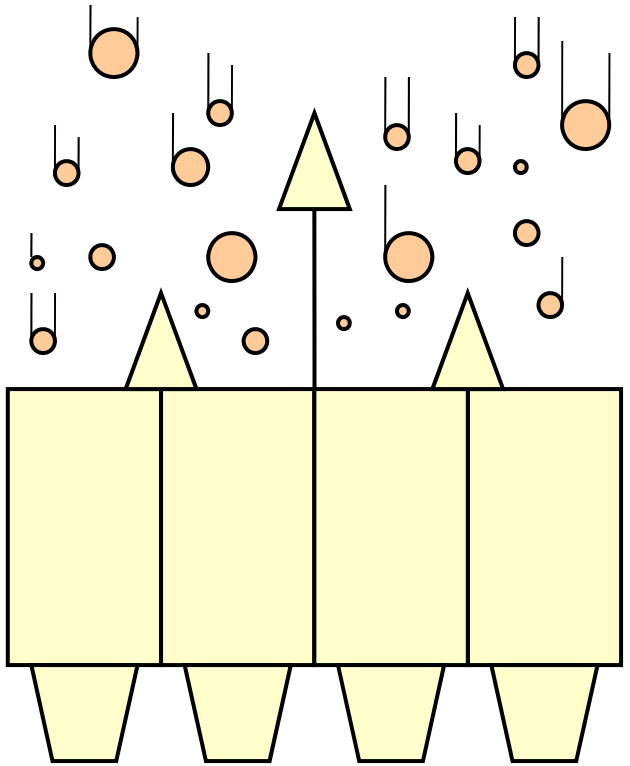
Technique: Coning & Quartering

- Pile of powder is divided into 4 sections.
- Two diagonal sections are discarded, and two are retained and mixed together.
- Mixture is again divided into 4 sections, and two diagonal sections are mixed.
- Process is repeated until remaining sample is correct amount for analysis.
- Can be carried out with very small sample amount or very large samples.



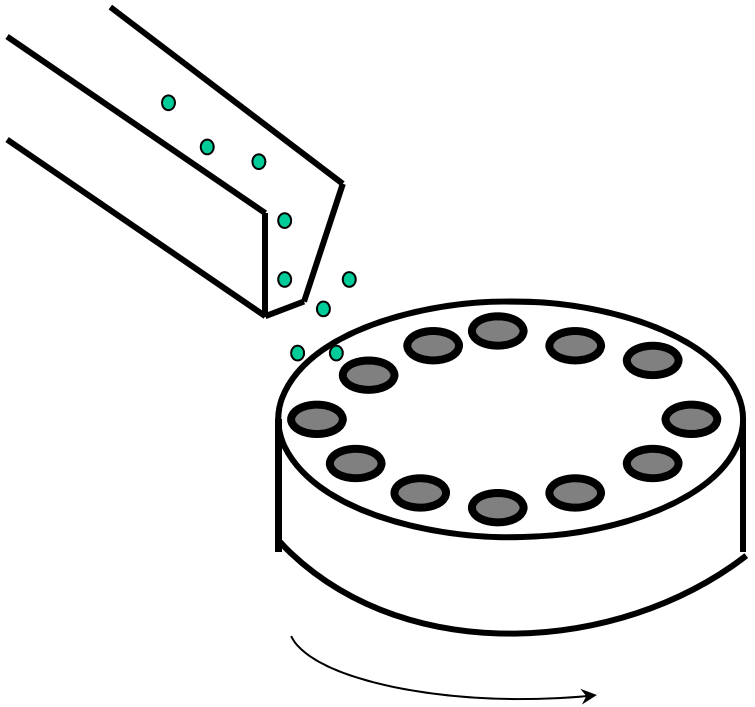
Technique: Chute Riffling

Chute splitting allows sample to vibrate down a chute to vanes which separate the mass into two portions. Each portion moves further where they each are divided into two parts, now giving four parts. This may be continued until usually 8 or 16 portions are obtained.



Technique: Rotary Riffling

The best method of representative splitting of powders is the ROTARY RIFFLER. The complete sample to be split is directed down a chute into open containers. Each container will receive a sample which is representative of the original bulk material because the distribution of material is averaged over time. The complete amount of the original bulk sample must be consumed.



These splitters are commercially available from companies that market various types of sample splitters.

See: www.retsch.com

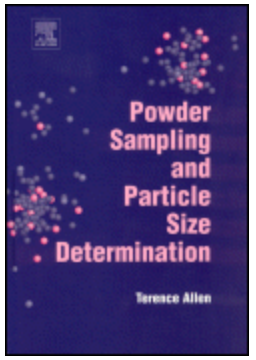
www.quantachrome.com

www.microscal.com

Sampling Technique Error Levels

Standard Deviation (σ) in % Sugar-Sand Mixture

SCOOP SAMPLING	**6.31**
TABLE SAMPLING	**2.11**
CHUTE RIFFLER	**1.10**
SPINNING RIFFLER	**0.27**



Density of sand and sugar respectively 2.65 and 1.64 g/ml

Reference: Allen, T. and Khan, A.A. (1934), Chem Eng, 238, CE 108-112

Method	Relative Standard Deviation (%)
Cone & Quartering	6.81
Scoop Sampling	5.14
Table Sampling	2.09
Chute Riffling	1.01
Spin Riffling	0.125

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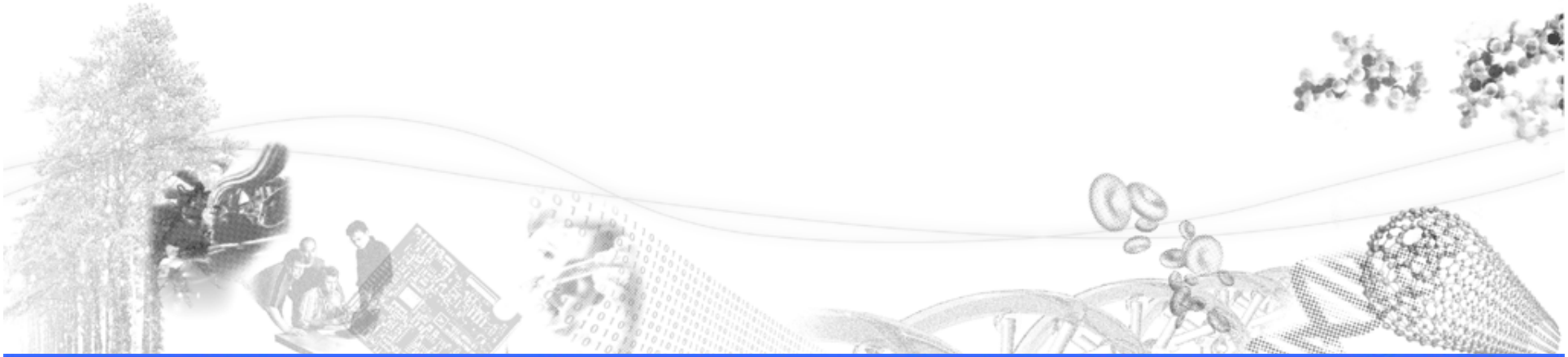
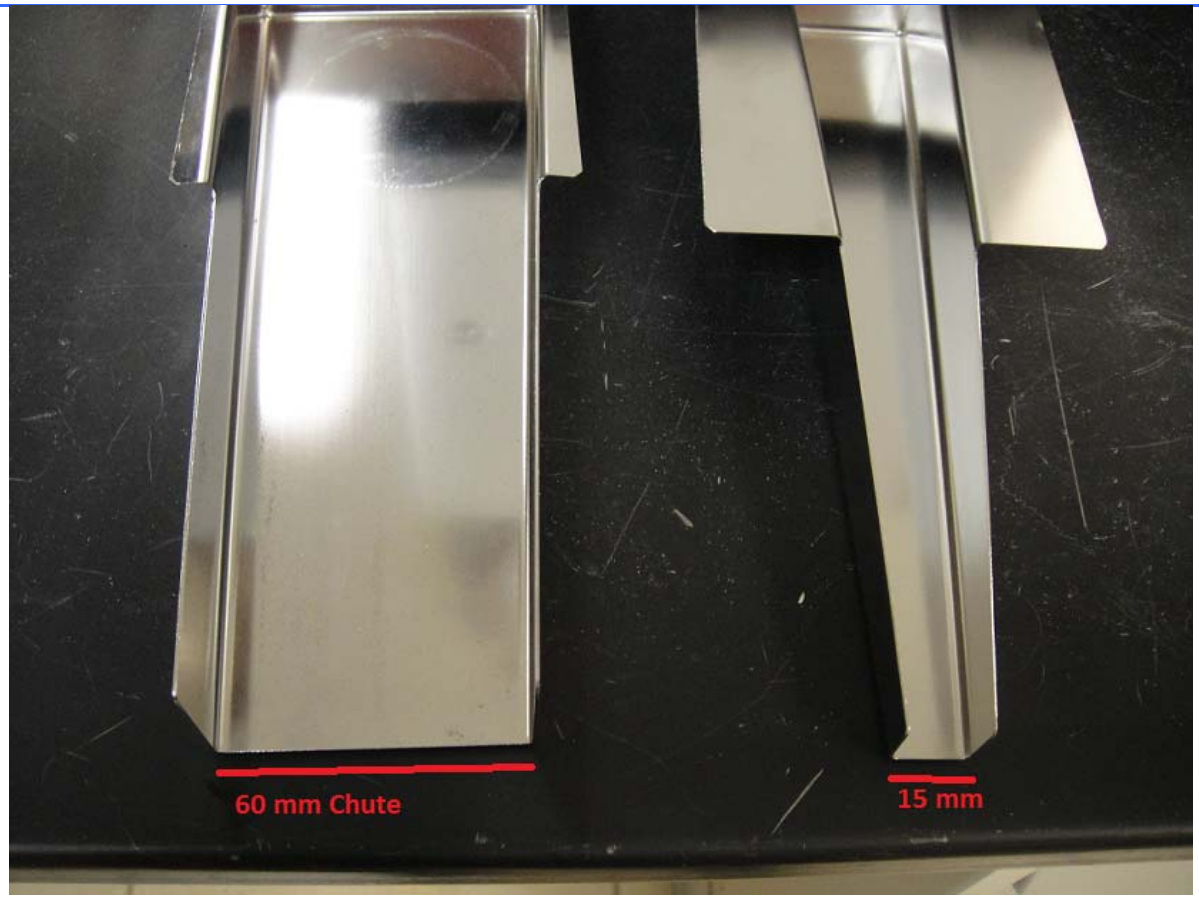
CAMSIZER Hardware

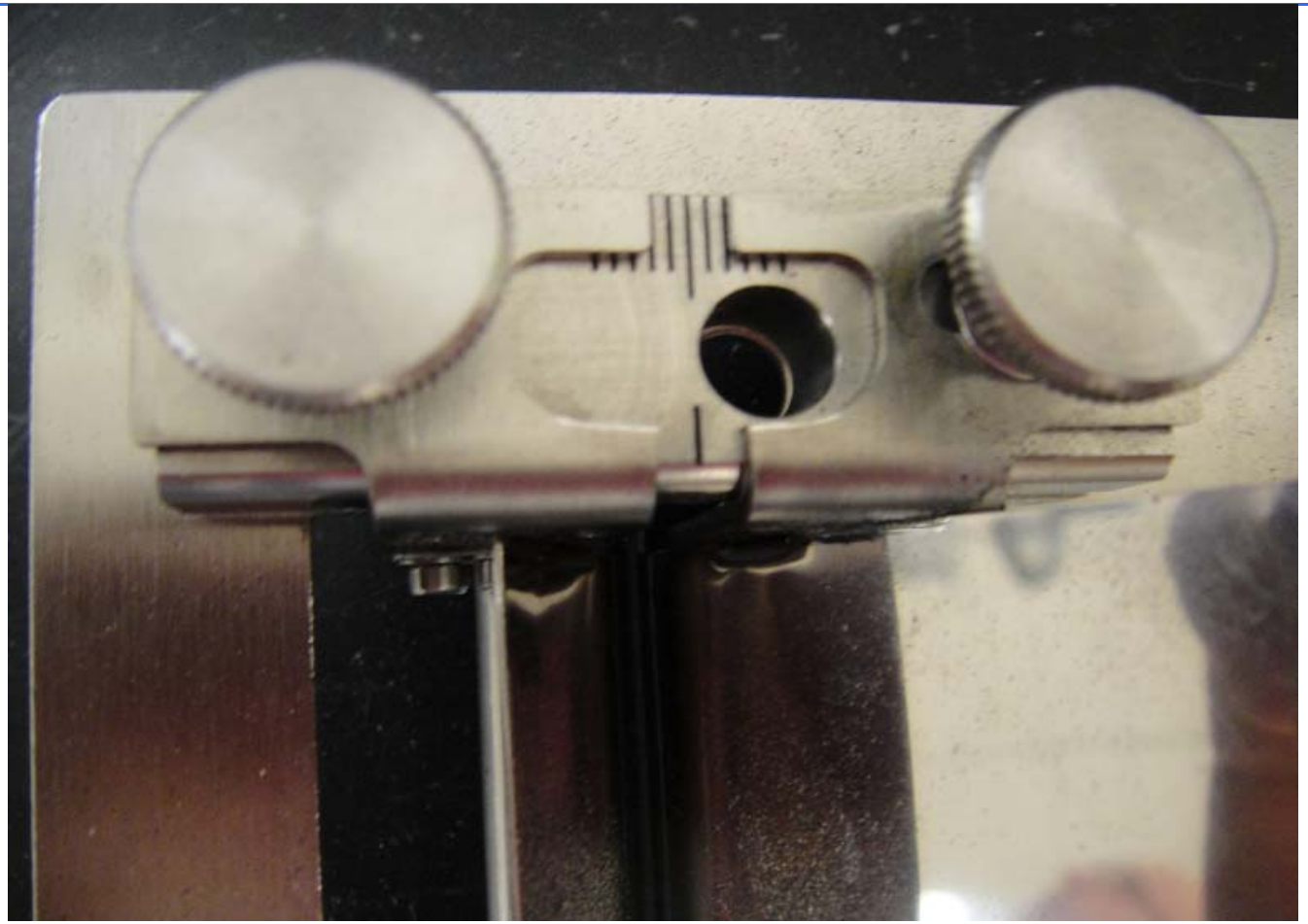


Chute Choice



- 60 mm Chute works well with particles larger than 240 um.
- 15 mm Chute is recommended if you need to be accurate for particles below 240 um (Note: Particles below 625 um will be mainly measured by the Zoom camera)

Guidance Sheet & Vacuum

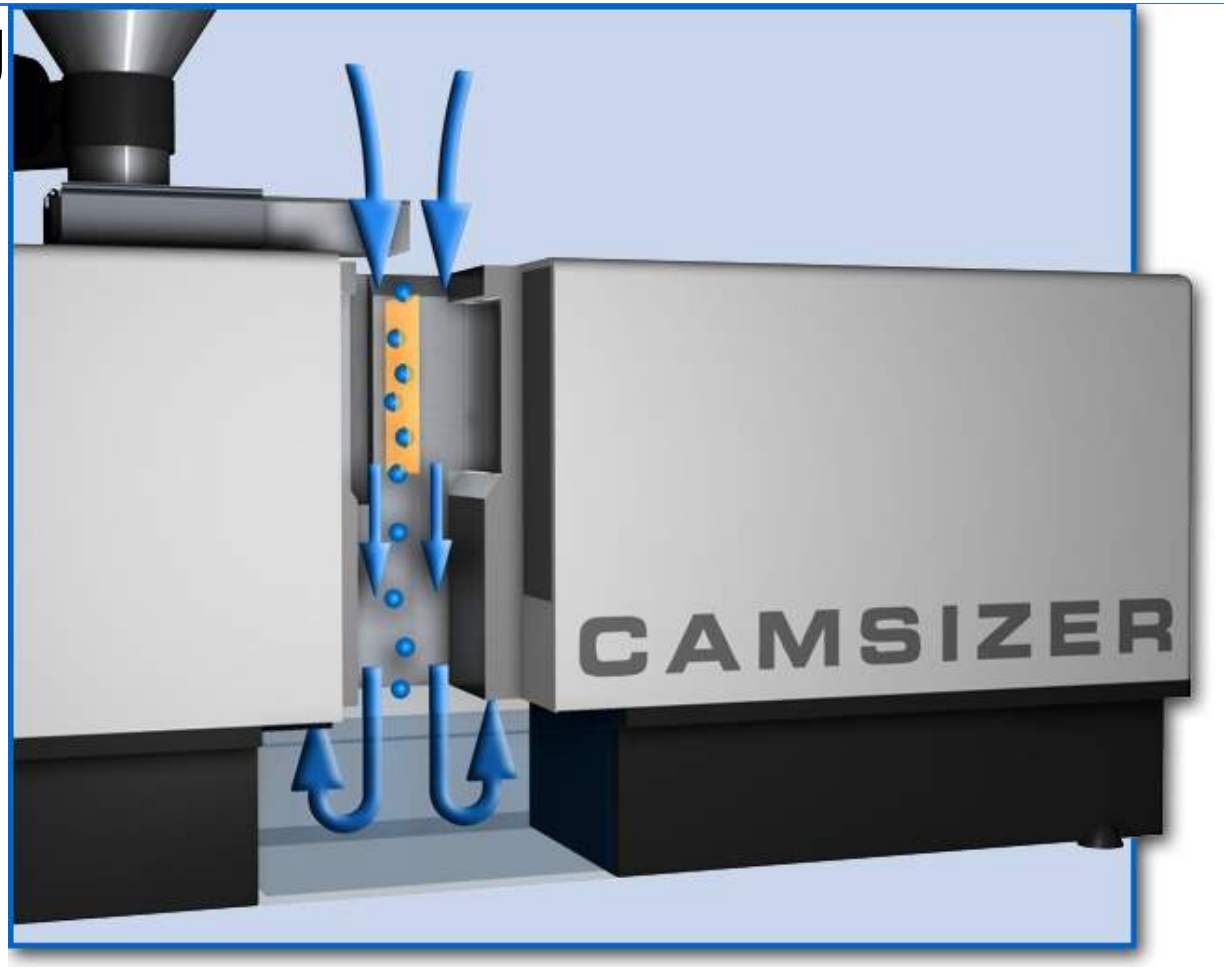


- Each scale is equal to 1 mm (Total 10 scale)
- Unless sample are larger than 10 mm, use the guidance sheet at all time (the gap needs to be larger than the largest particle)

Maintenance-Free by Venturi-Flushing

The guidance sheet will help particles fall on the same plane field and prevent particles from touching windows

Also use Vacuum to keep the windows clean, especially for particles that are cohesive.



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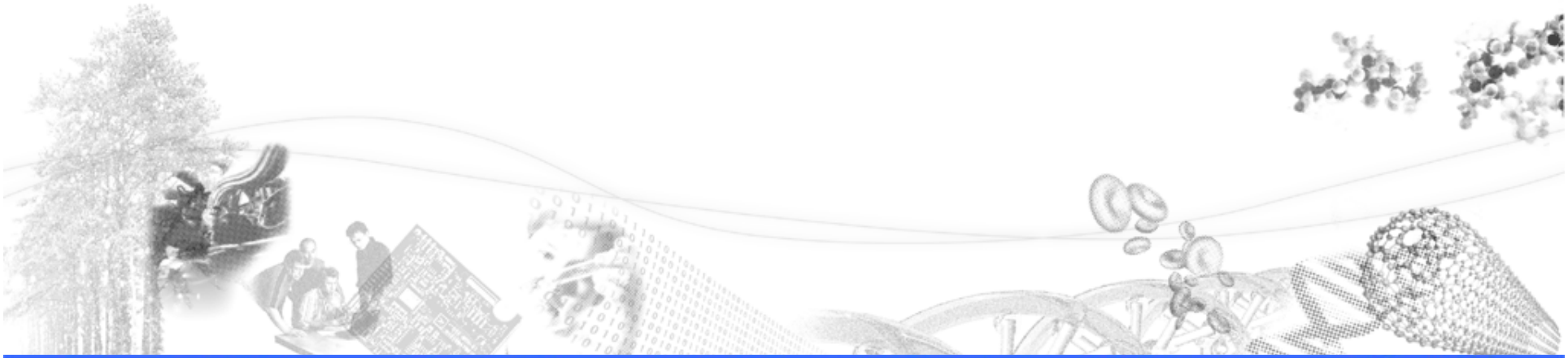
Size and Shape Parameter Choices



Choosing Size Parameters

- **How do I determine which size parameter to use??**

Particle Size

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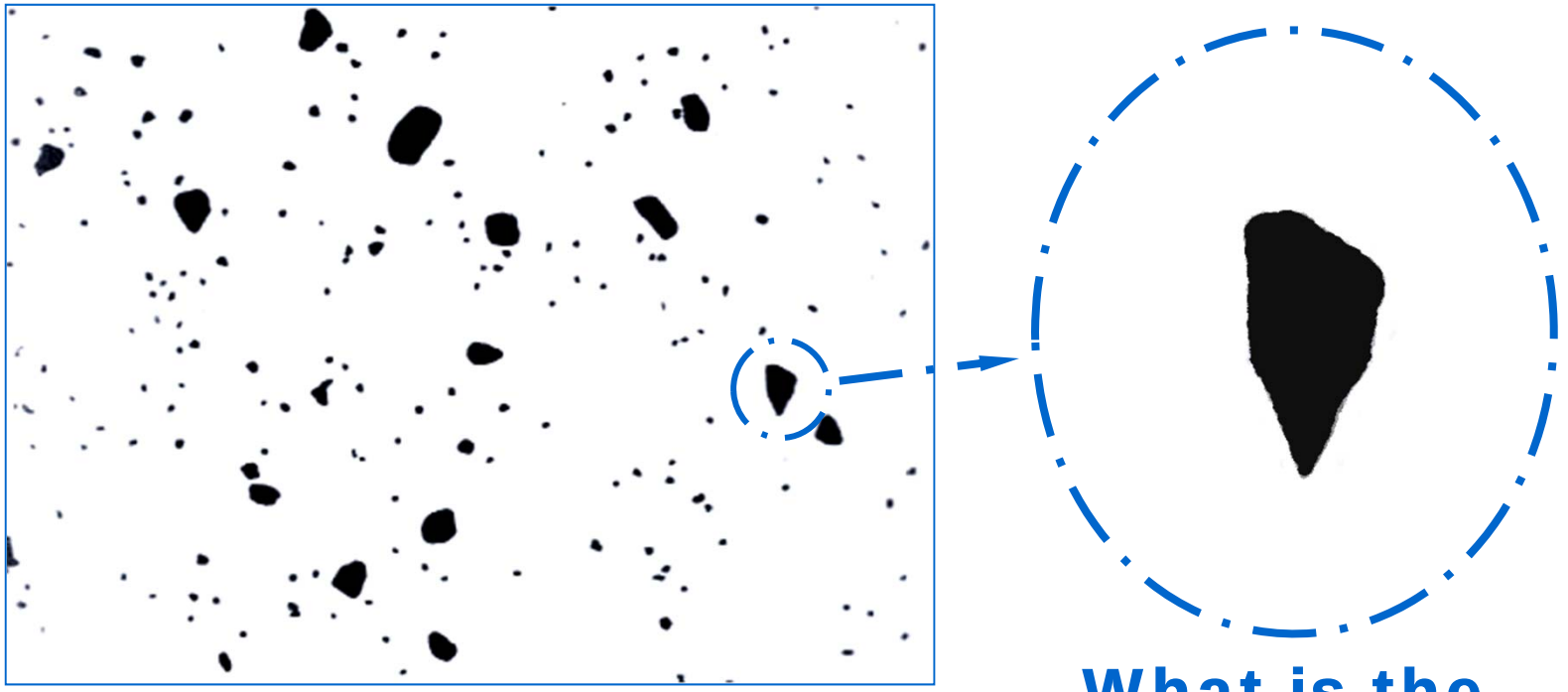
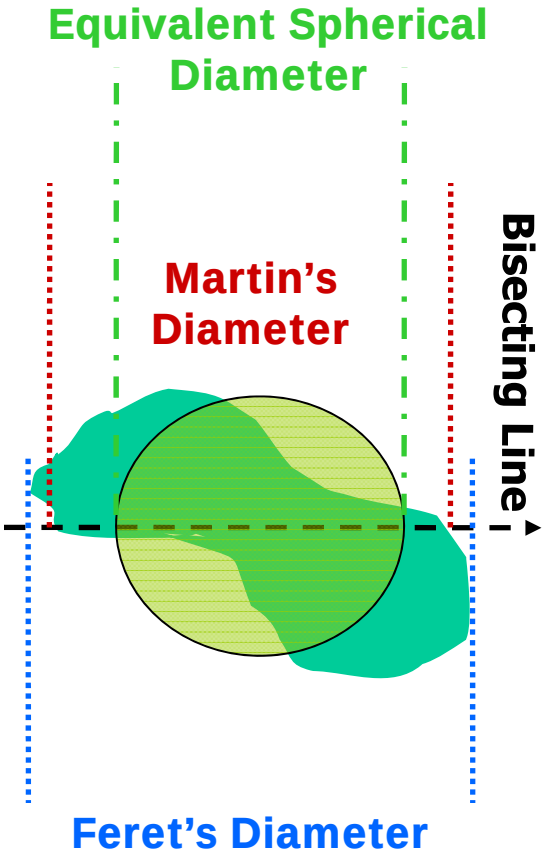


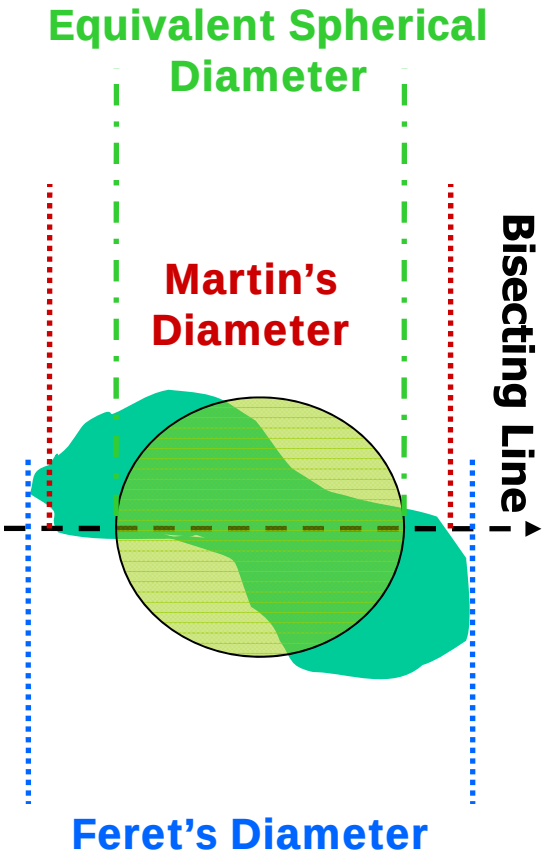
What is the size of this particle?

Size Definitions

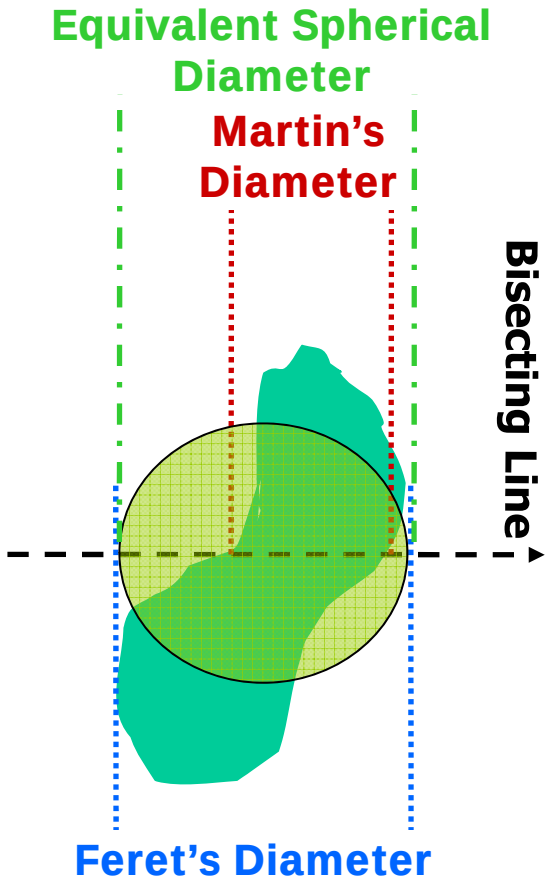
- **Martins's Diameter:** The distance between opposite sides of a particle measured on a line bisecting the projected area. To ensure statistical significance all measurements are made in the same direction regardless of particle orientation.
- **Feret's Diameter:** The distance between parallel tangents on opposite sides of the particle profile. Again to insure statistical significance, all measurements are made in the same direction regardless of particle orientation.
- **Note:** *Both Martin's and Feret's diameters are generally used for particle size analysis by optical and electron microscopy.*
- **Equivalent Circle Diameter:** The diameter of a circle having an area equal to the projected area of the particle in random orientation. This diameter is usually determined subjectively and measured by oracular micrometers called graticules.
- **Equivalent Spherical Diameter:** The diameter of a sphere that has the same volume as the irregular particle being examined.



Particle Orientation



■ Martin's and Feret's Diameter's will vary as particles are viewed in different orientations. The result will be a DISTRIBUTION from smallest to largest.



Particle Size

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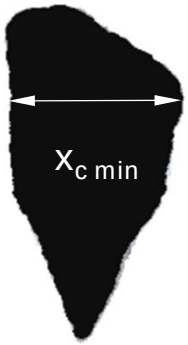
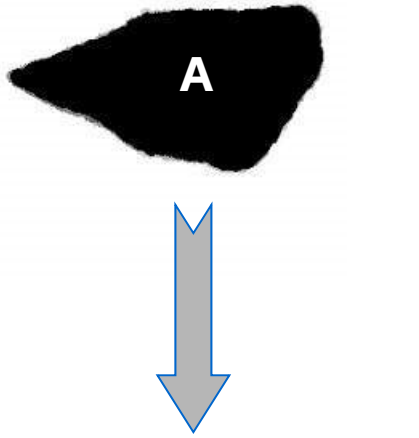
$X_{c\,min}$

"width"



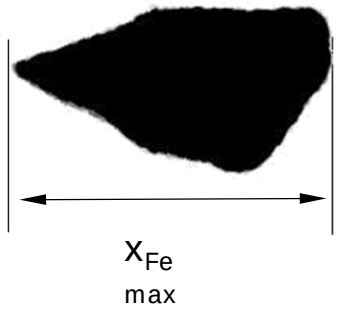
X_{area}

"diameter over projection surface"



$X_{Fe\,max}$

"length"



Comparison of Size Definitions

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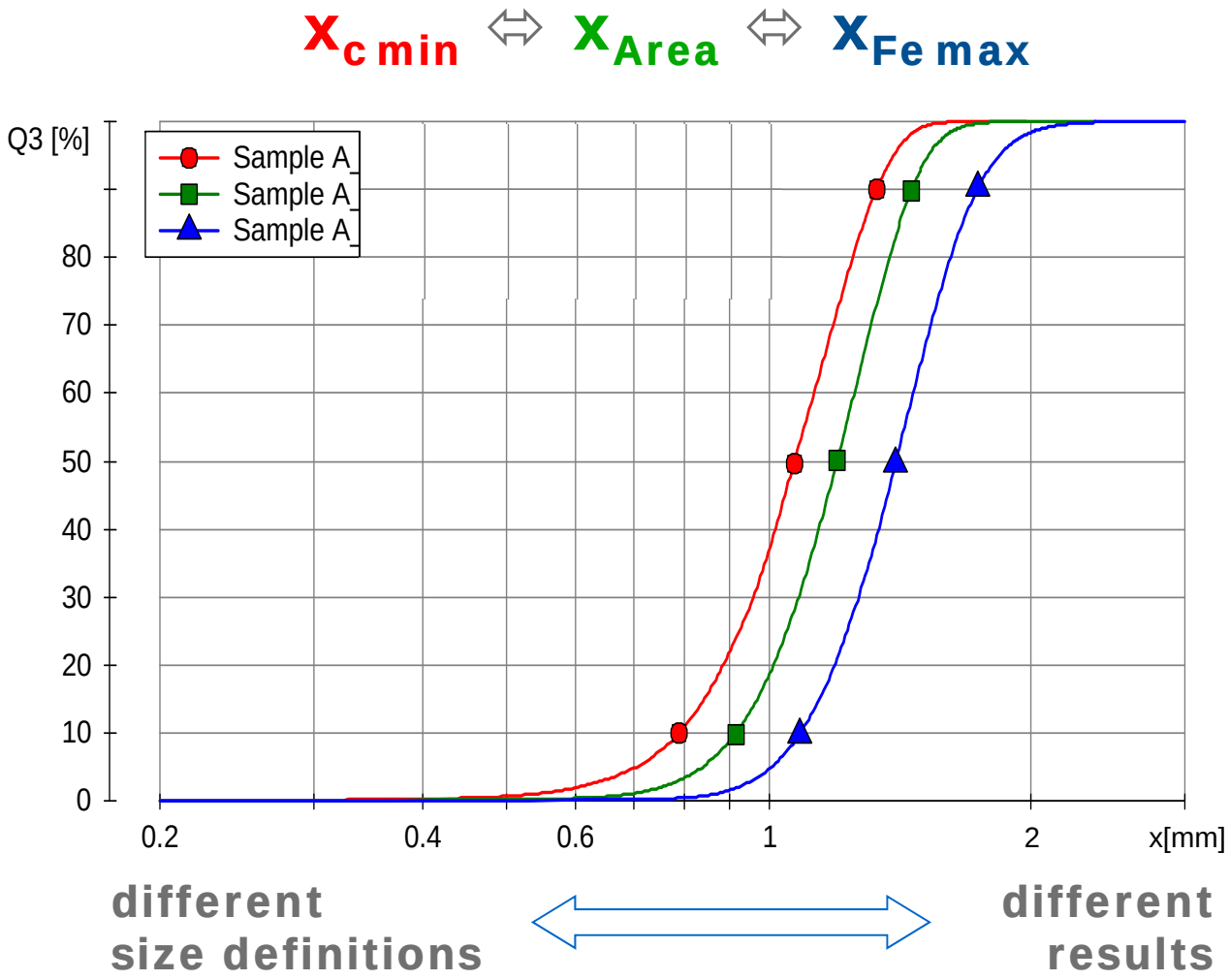


Size Parameter Selection Questions

1. Do I know what size parameter(s) I want to measure? (Maybe you already know)
2. Do I need to correlate the CAMSIZER data to another data? (Sieve data, laser diffraction data, or any other data?)
3. Which technique do I need to correlate?
4. Which parameter will be most critical?

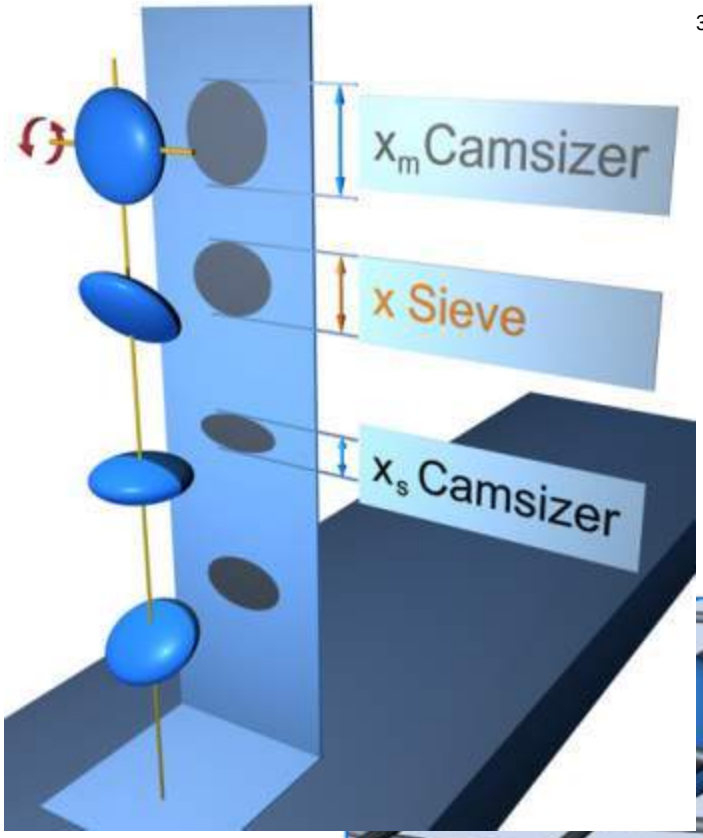
Size Parameter Selection Based on Techniques

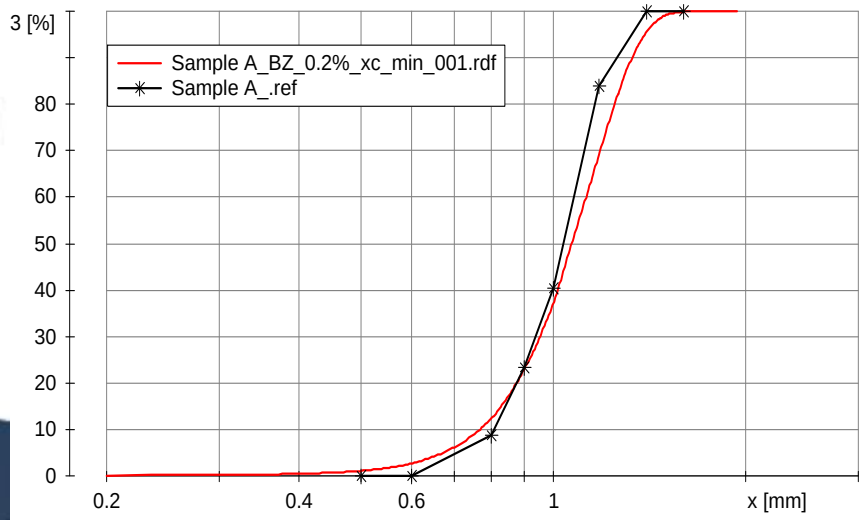
- Xc_min (width): To correlate sieve data (may need a fitting file on the CAMSIZER)
- Xarea (equivalent spherical diameter): To correlate laser diffraction data
- Xfe_max (length): No other techniques can really measure this. If length needs to be measured, one may have to use image analyzer.

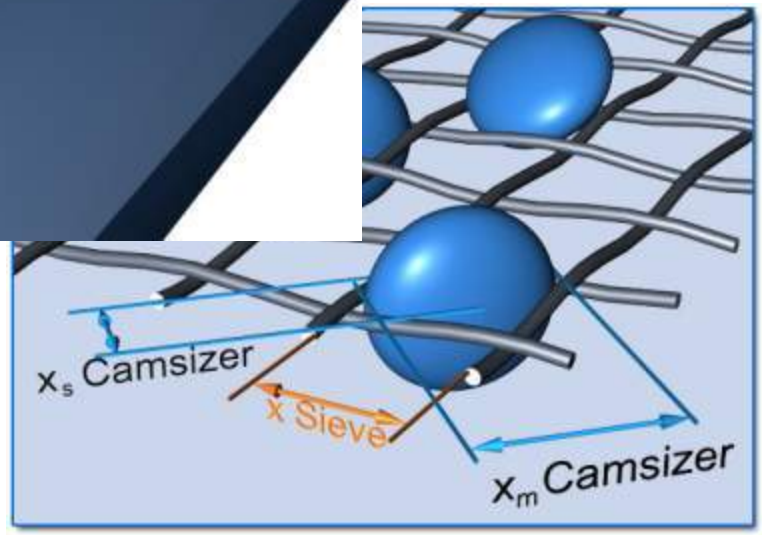
Size Parameter Selection Process

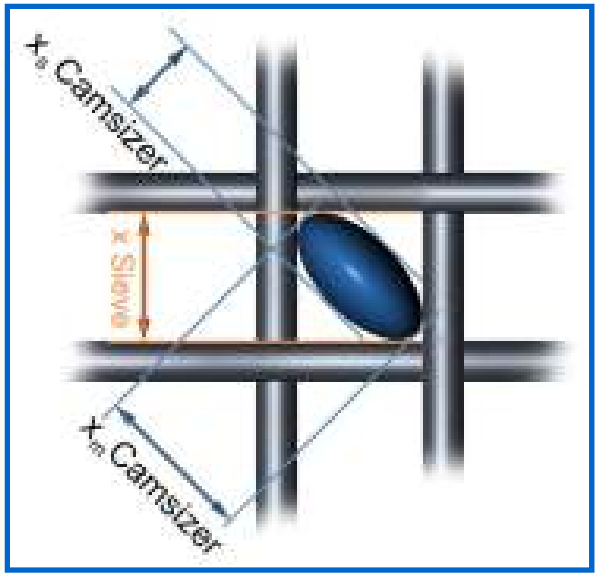
1. Measure multiple size parameters (up to 5 different parameters can be measured with the CAMSIZER)
2. Compare each size parameters and identify the parameter that give the desired data

Digitale Imaging ⇔ Sieving









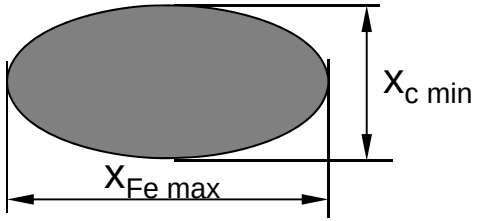
Choosing Shape Parameters

- How do I determine which shape parameter to use??

Particle Shape

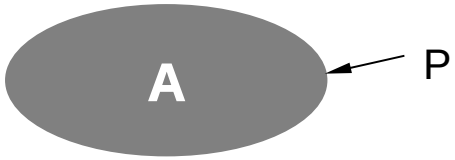
- **width / length = aspect ratio**

$$\frac{X_{c\ min}}{X_{Fe\ max}}$$



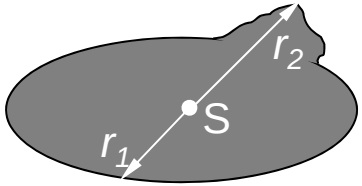
- **Roundness**

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



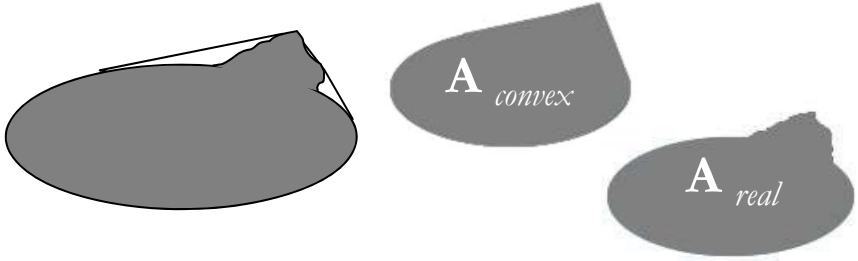
- **Symmetry**

$$\frac{1}{2}\left(1+\min\left(\frac{r_1}{r_2}\right)\right)$$



- **Convexity**

$$\sqrt{\frac{A_{real}}{A_{convex}}}$$



Shape Parameter Selection Questions

1. Do I know what shape parameter(s) I want to measure? (Maybe you already know)
2. Do I want to quantify an outlier?
3. Do I want to differentiate mixtures?

Shape Parameter Selection Process

1. Measure multiple shape parameters (all the parameters that are available in the CAMSIZER software can be measured)
2. Save images to compare particle to particle
3. Compare shape parameters and identify the parameter that give the most discrepancy
4. Set a parameter threshold to clearly identify amount of each group

Image Evaluation

Symm (symmetry) is the parameter with the most discrepancy

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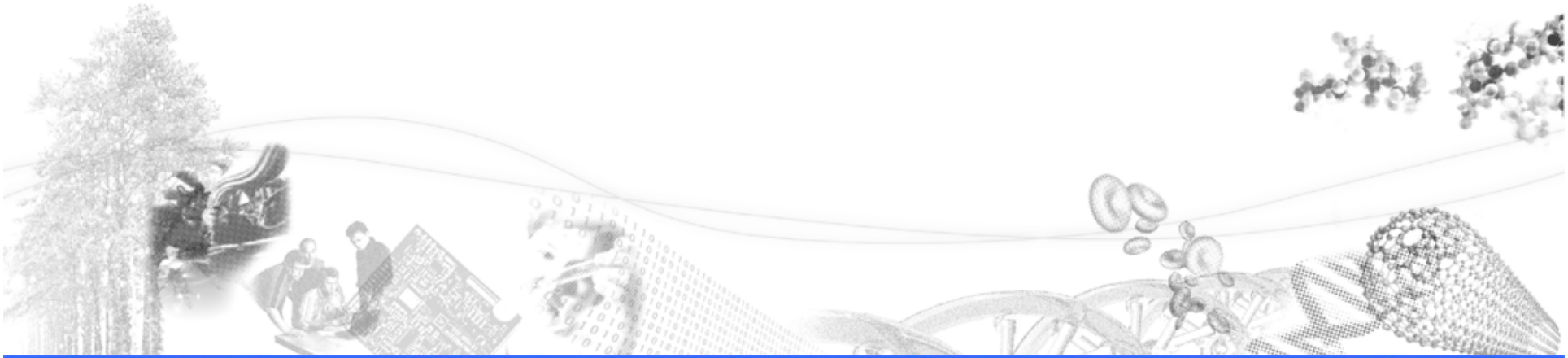
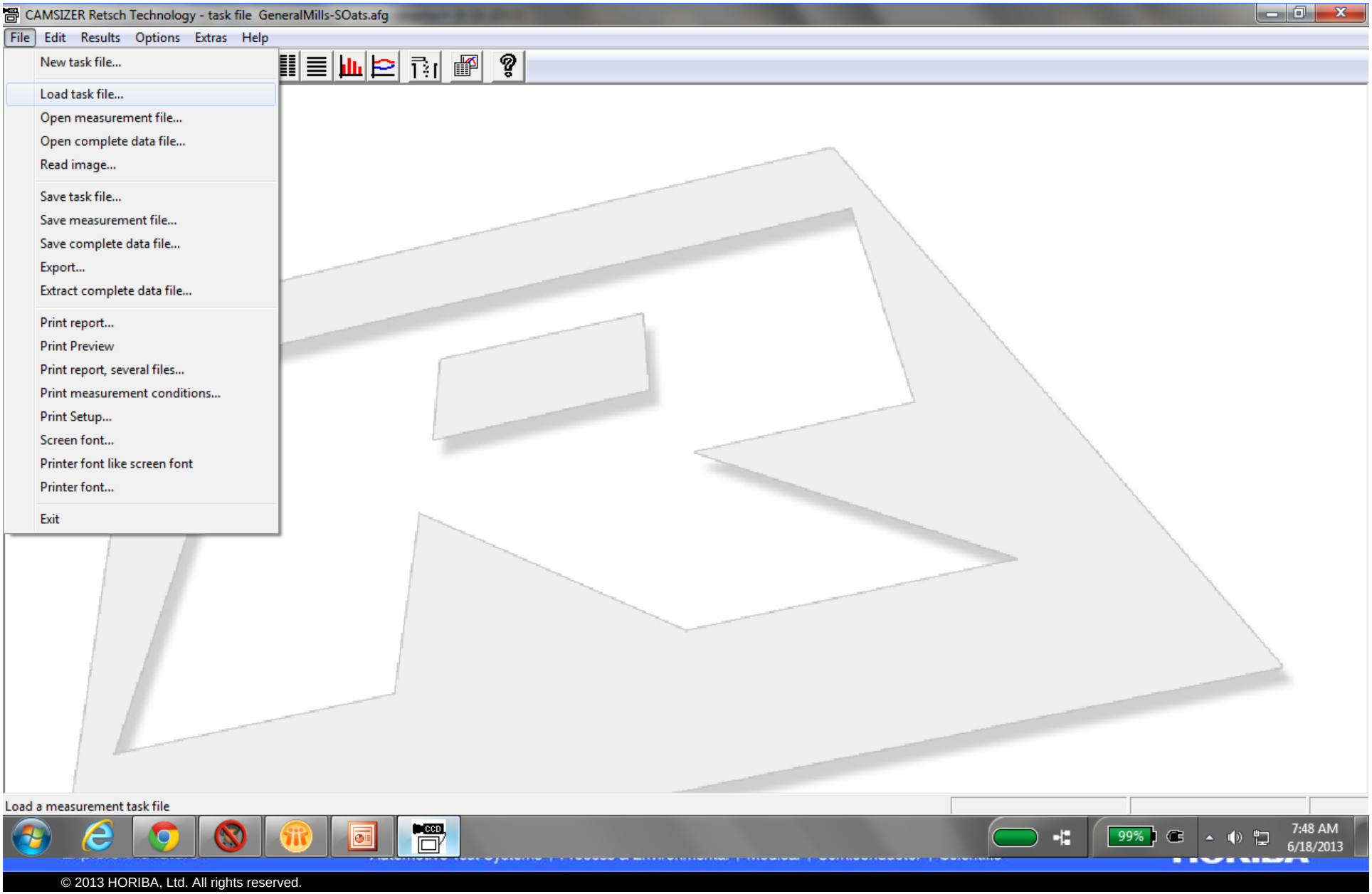
Method Design
(Creating a Task File in the CAMSIZER Software)

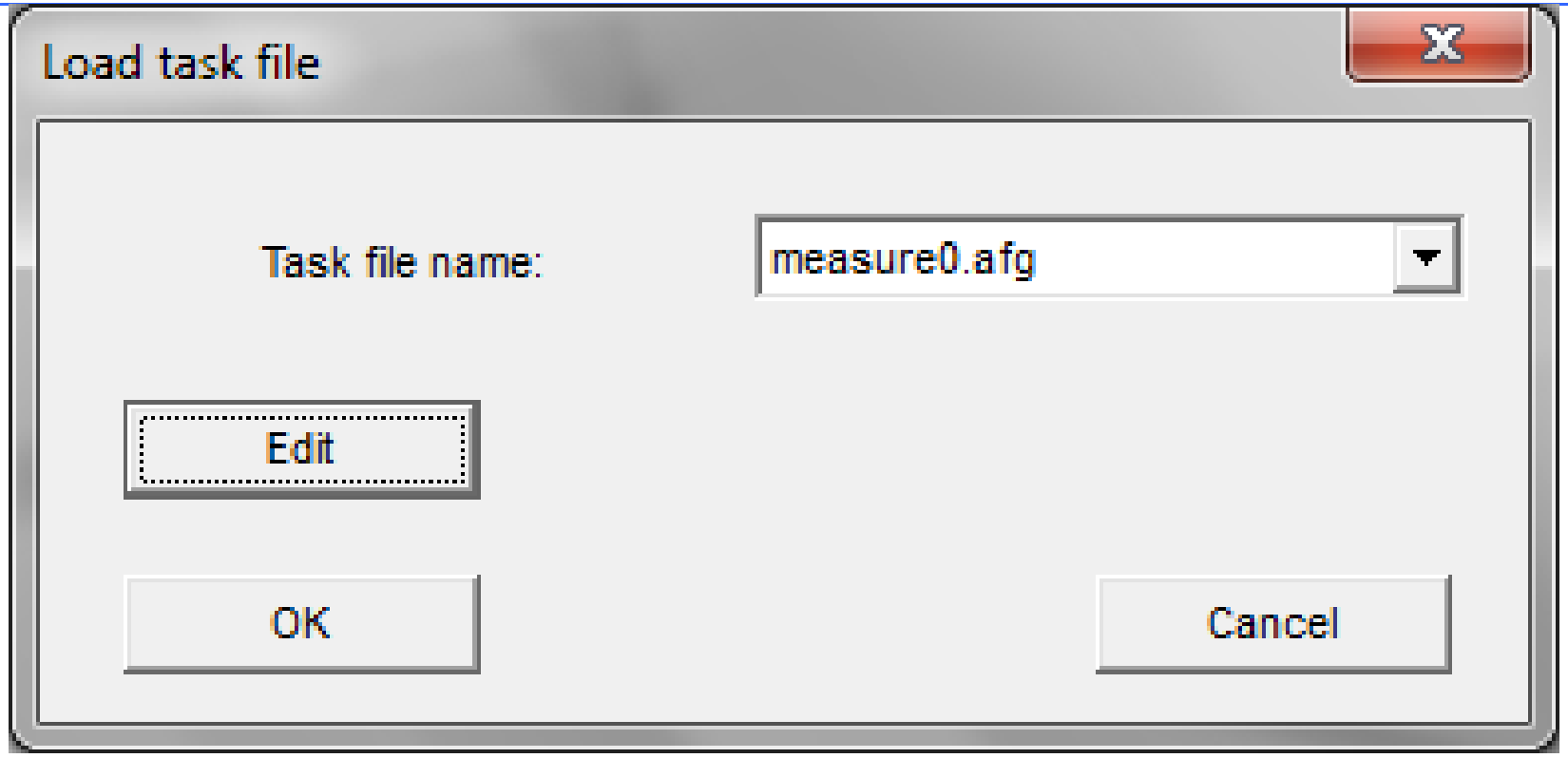


Goal for a Method

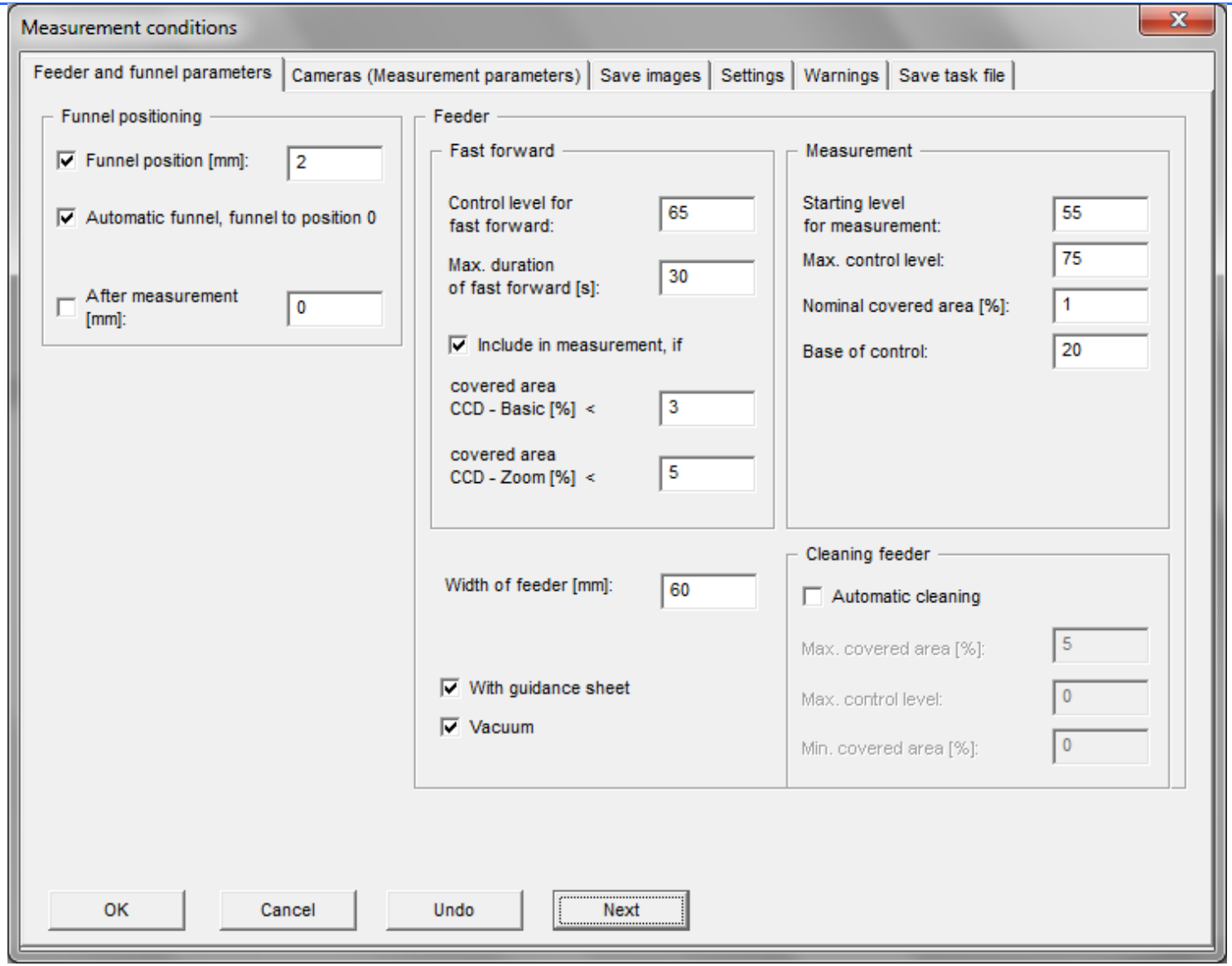
- Accuracy: Not really questioned (Images support its accuracy)
- Repeatability: The CAMSIZER lets users recycle its samples and repeatability is usually good
- Reproducibility: A good sampling technique is the key to achieve reproducibility
- Resolution: Extremely good since the CAMSIZER will use every particle to generate data
- Match historic data (sieves, LD, and etc..): Possible to match any data as long as a correct parameter gets measured

New Task File





Feeder and Funnel Control



Cameras

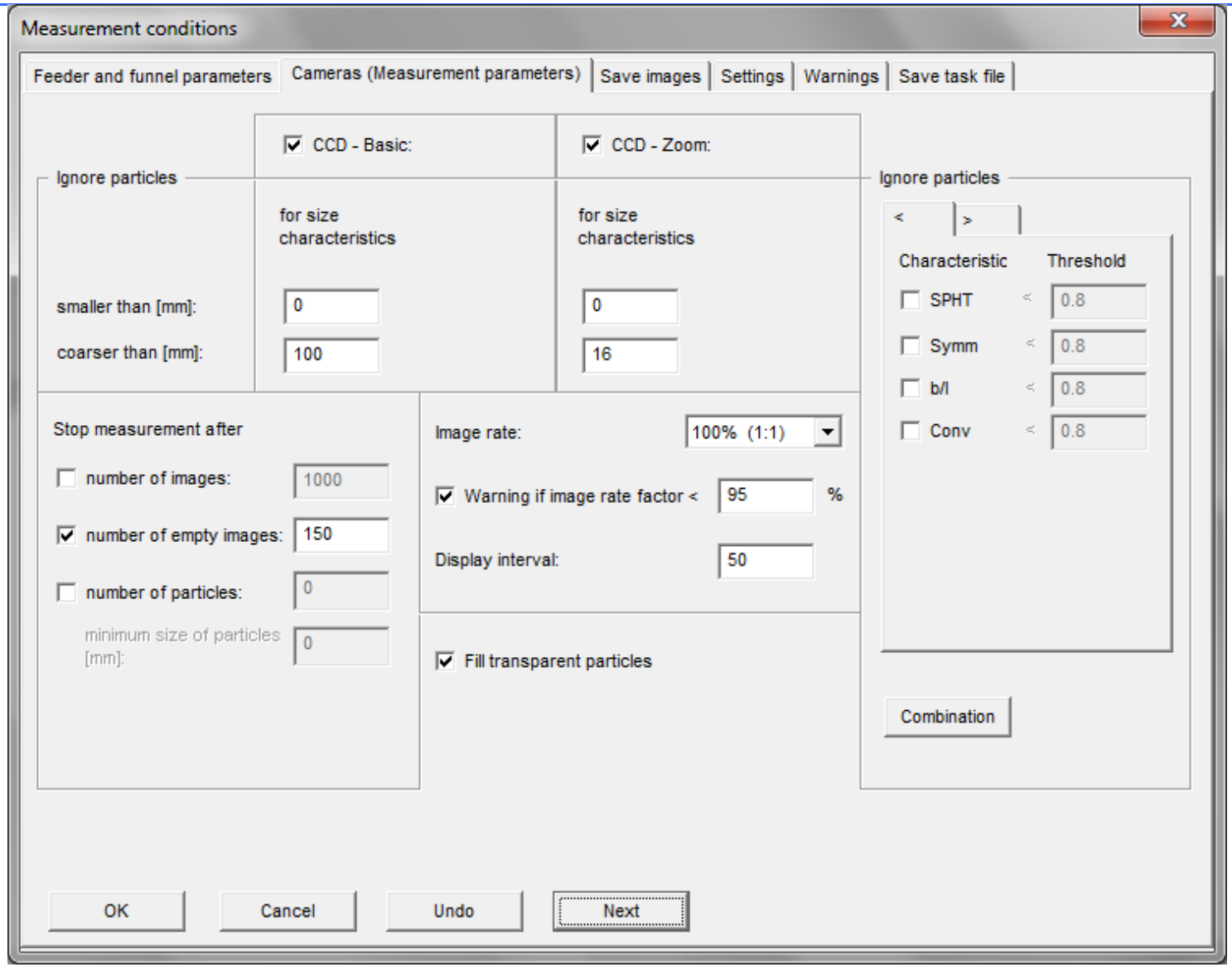
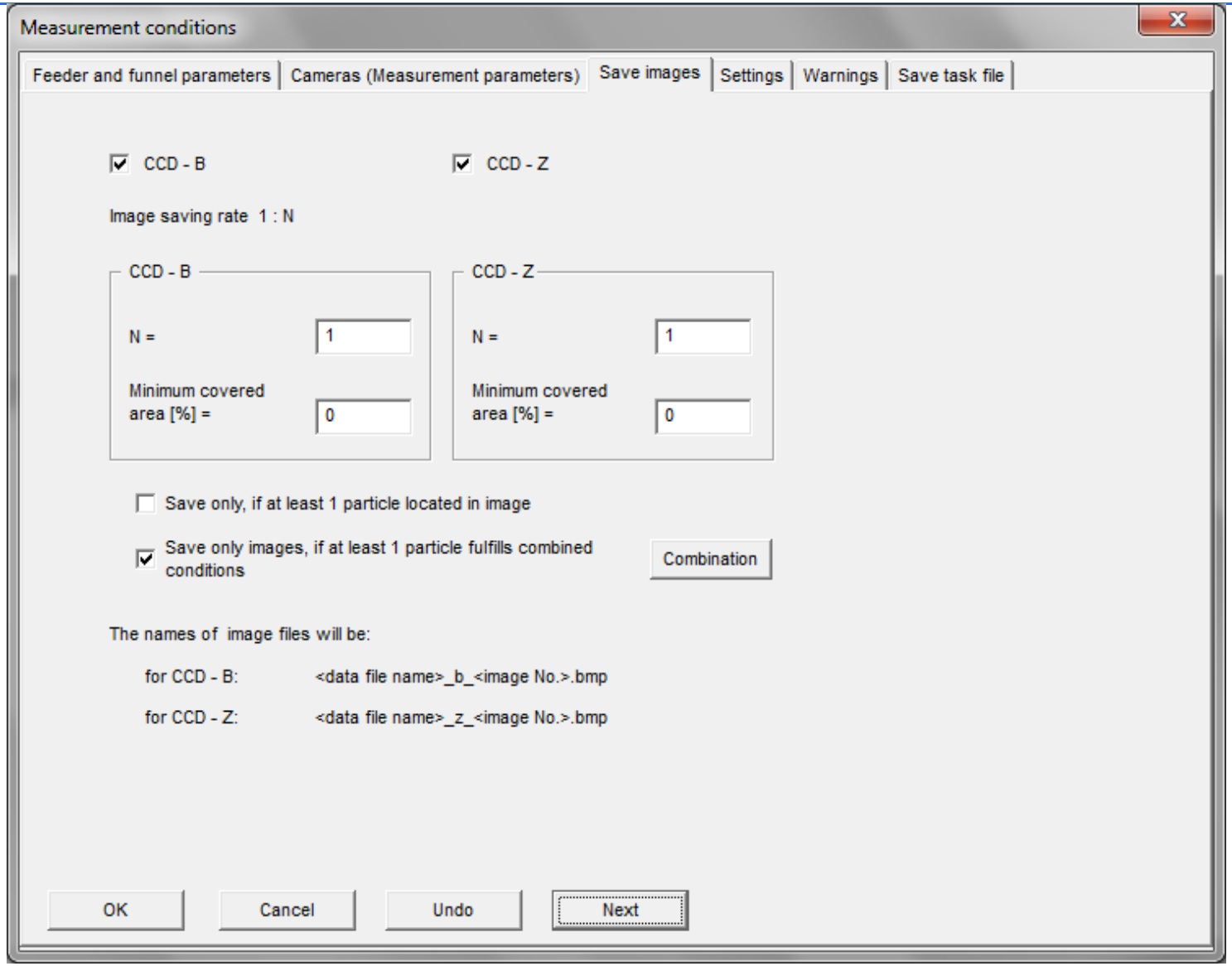
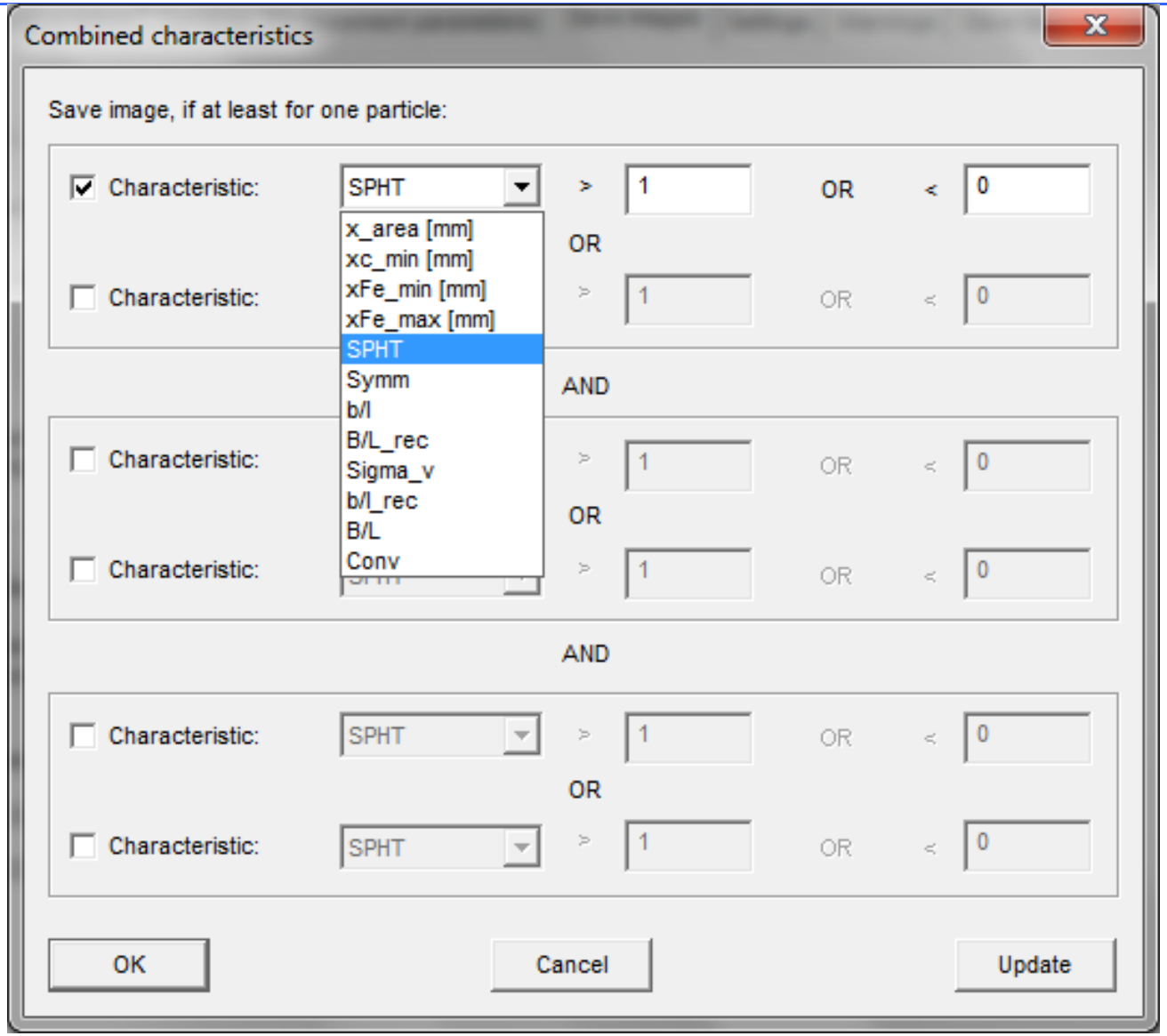


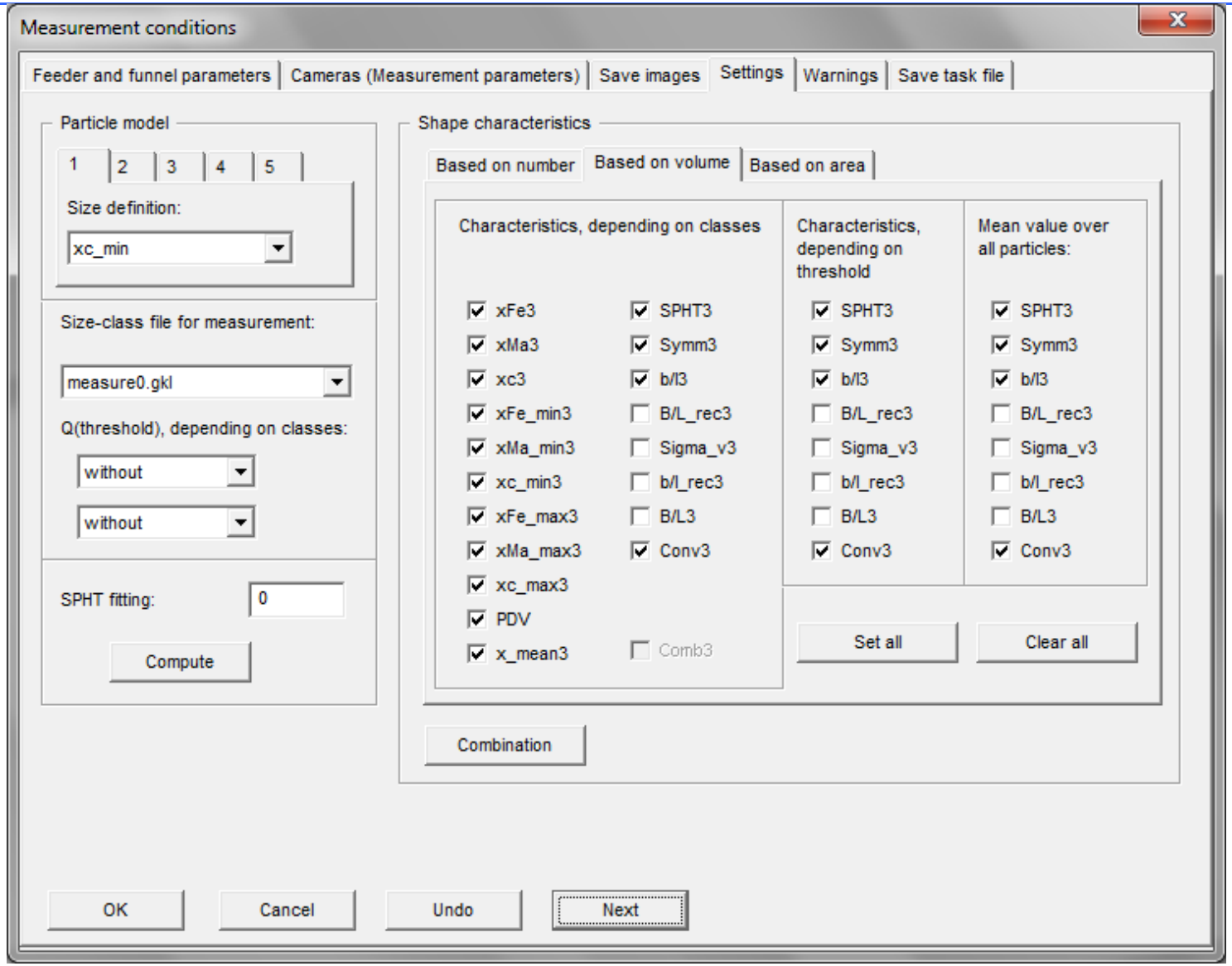
Image Saving



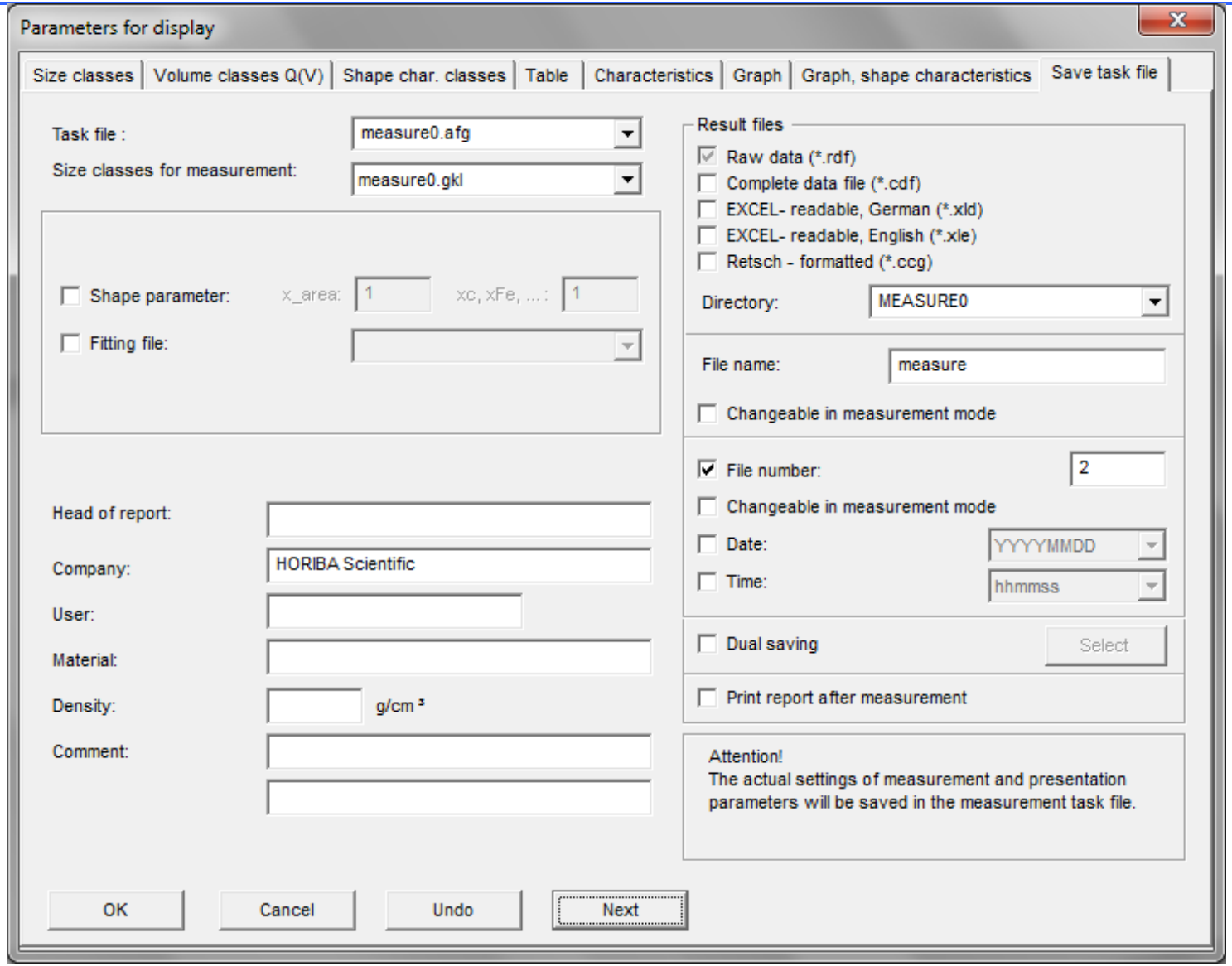
Saving Specific Images



Saving Different Size and Shape Parameters



Saving a Task File



Method Development Summary

1. Know how to practice a good sampling technique: Try to get the representative sample all the time
2. Know your hardware: Choose proper hardware to get more accurate and precise data
3. Know what you want to measure: Choose size and shape parameters will achieve your goal
4. Know the software settings: Choose proper settings to get more robust data

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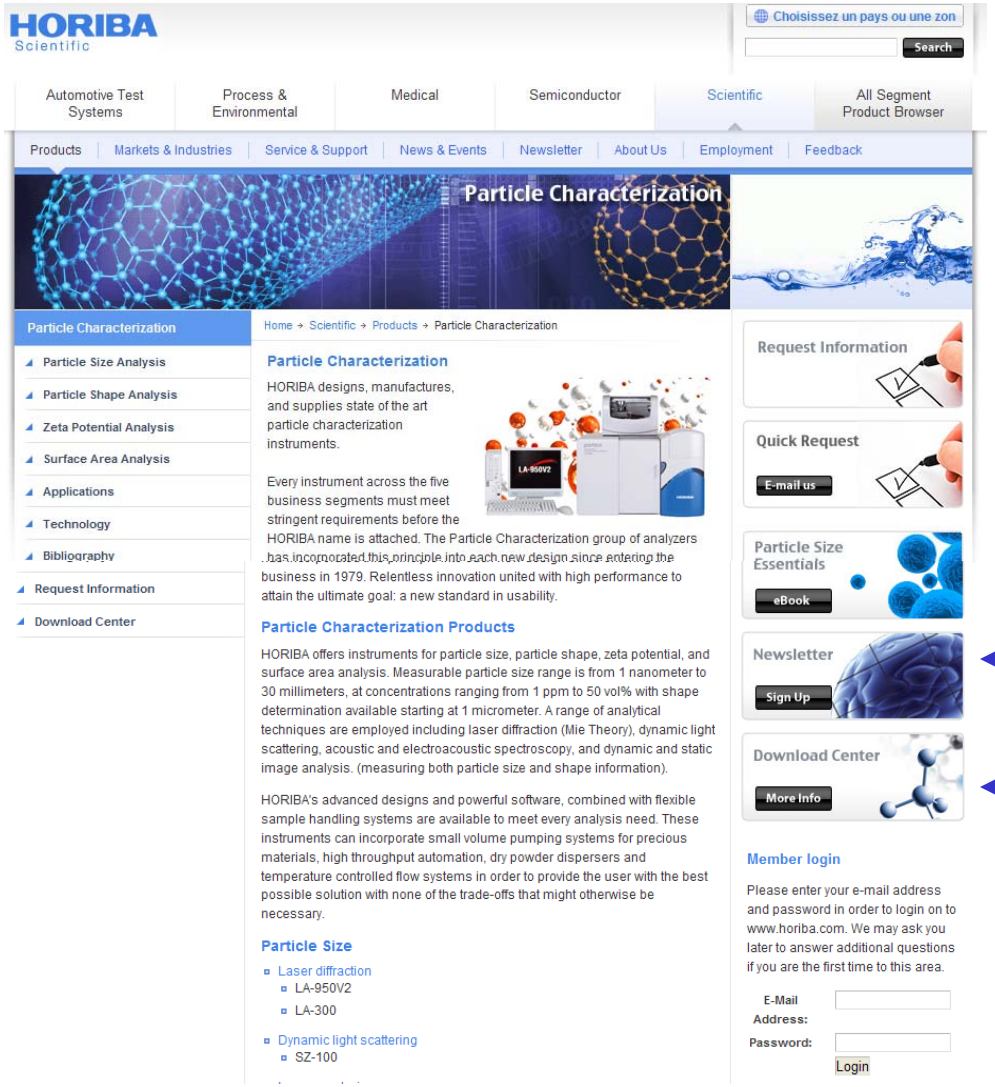

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