# CAMSIZER®

## Particle Size and Shape using Dynamic Image Analysis ISO 13322-2 conform

HORIBA Scientific & Retsch Technology
CAMSIZER Fertilizer Webinar

November 16th 2011

Host: Ian Treviranus, HORIBA Instruments
Speaker: Dipl.-Ing. Gert Beckmann
Retsch Technology GmbH

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# Content

* CAMSIZER measurement principle
* Intelligent matching of sieve results
* SGN, UI and other calculations
* Q & A session

## Device

1. Measuring Principle
2. Measurement Results

## Application

3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods




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# Retsch Technology

**1999**

Development of
CAMSIZER &
CRYSTALSIZER

**2001**

Announcement of
HORIBA
Particle size analyzers
in Europe

**2002**

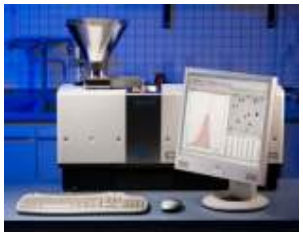
New shape functions
of CAMSIZER,
first Online-Installations

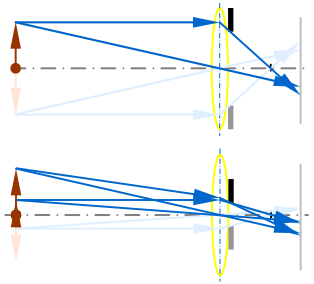
**2003**

Development
of AutoSampler System

**2006**

Improvement of CAMSIZER®
with AutoHeight, LED technology,
Software, improved resolution,
sharpness and contrast





**2011**

Development
of CAMSIZER XT
for agglomerating powders
1µm-3000µm

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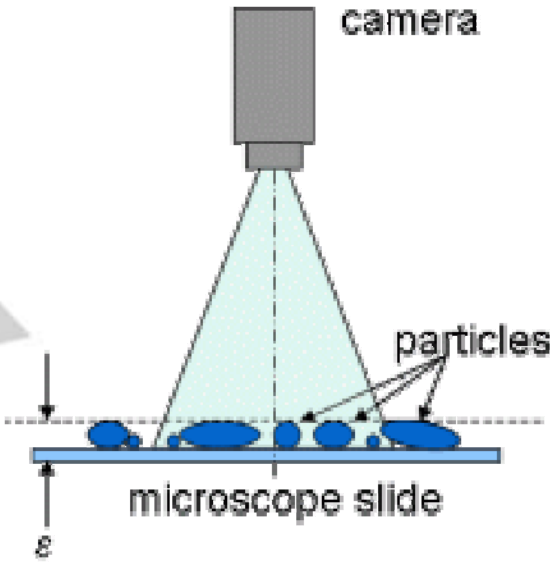
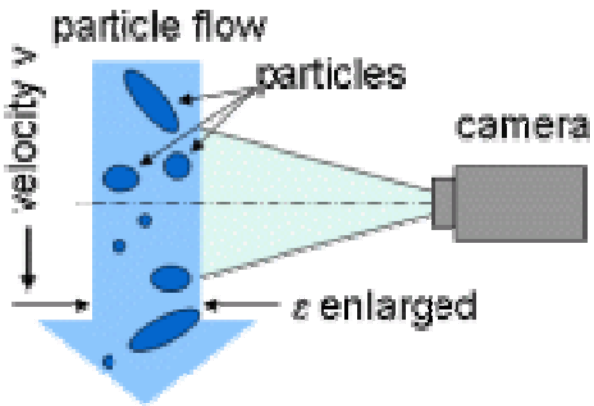
# Comparison between Static ⇔ Dynamic Image Analysis




ISO 13322-1




ISO 13322-2



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# Content

## Device

1. Measuring Principle
   - Two Camera-System
   - Resolution
2. Measurement Results

## Application

3. CAMSIZER Applications in the Fertilizer Industry
4. Competing Measuring Methods




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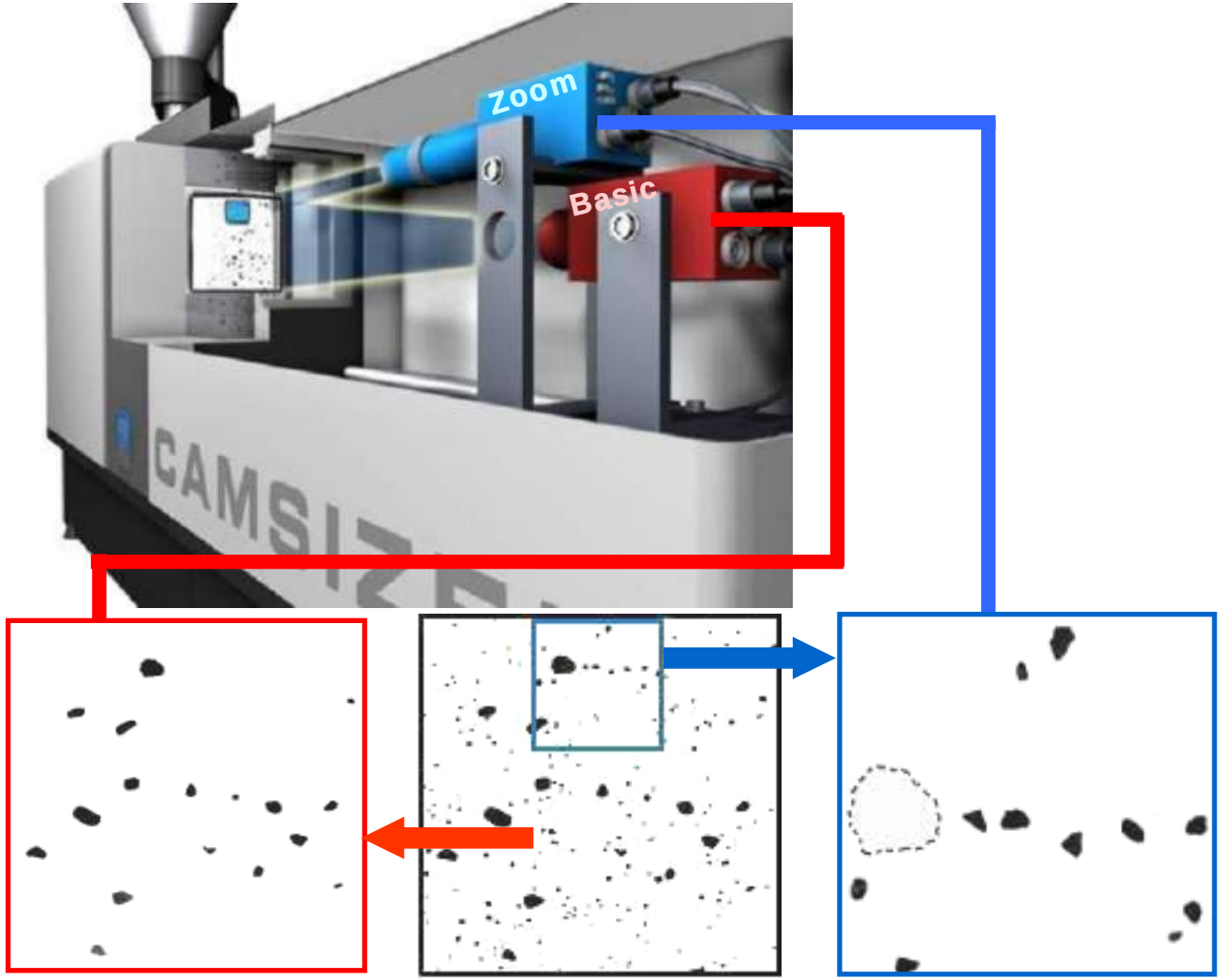
# Two-Camera-System




Basic-Camera

Zoom-Camera



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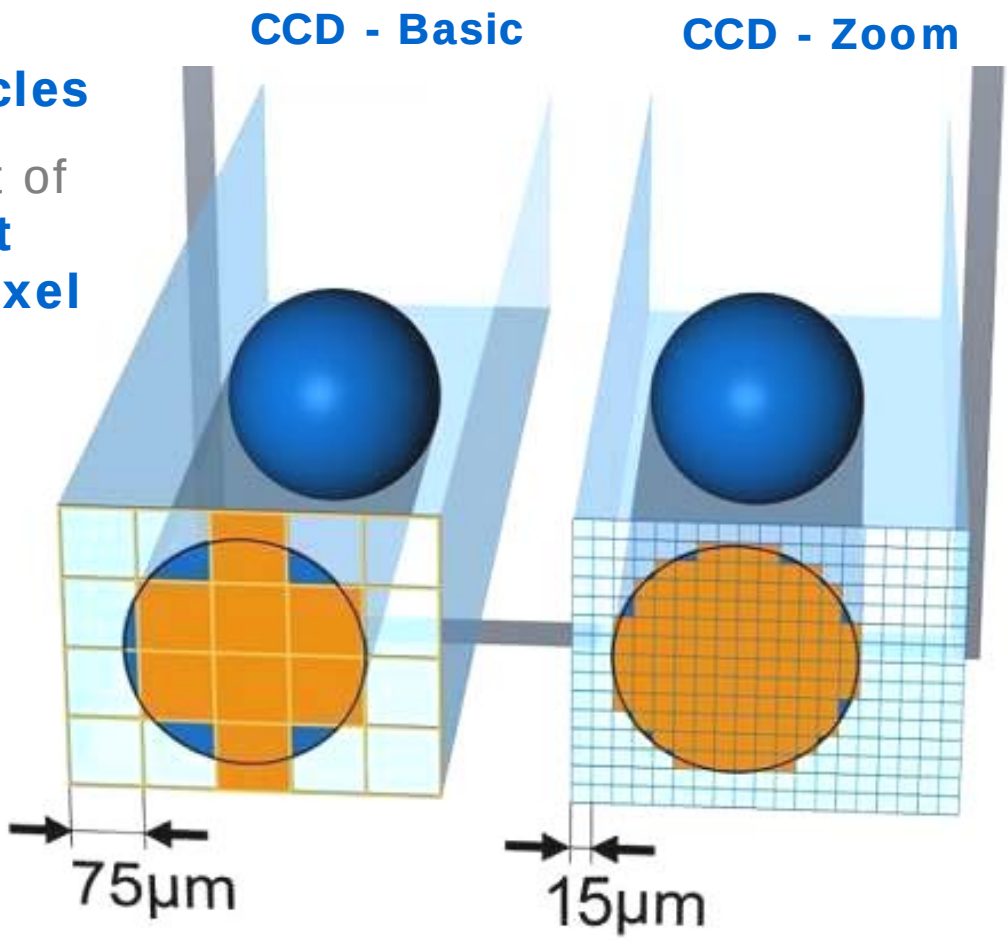
# Resolution

## Detection of particles

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



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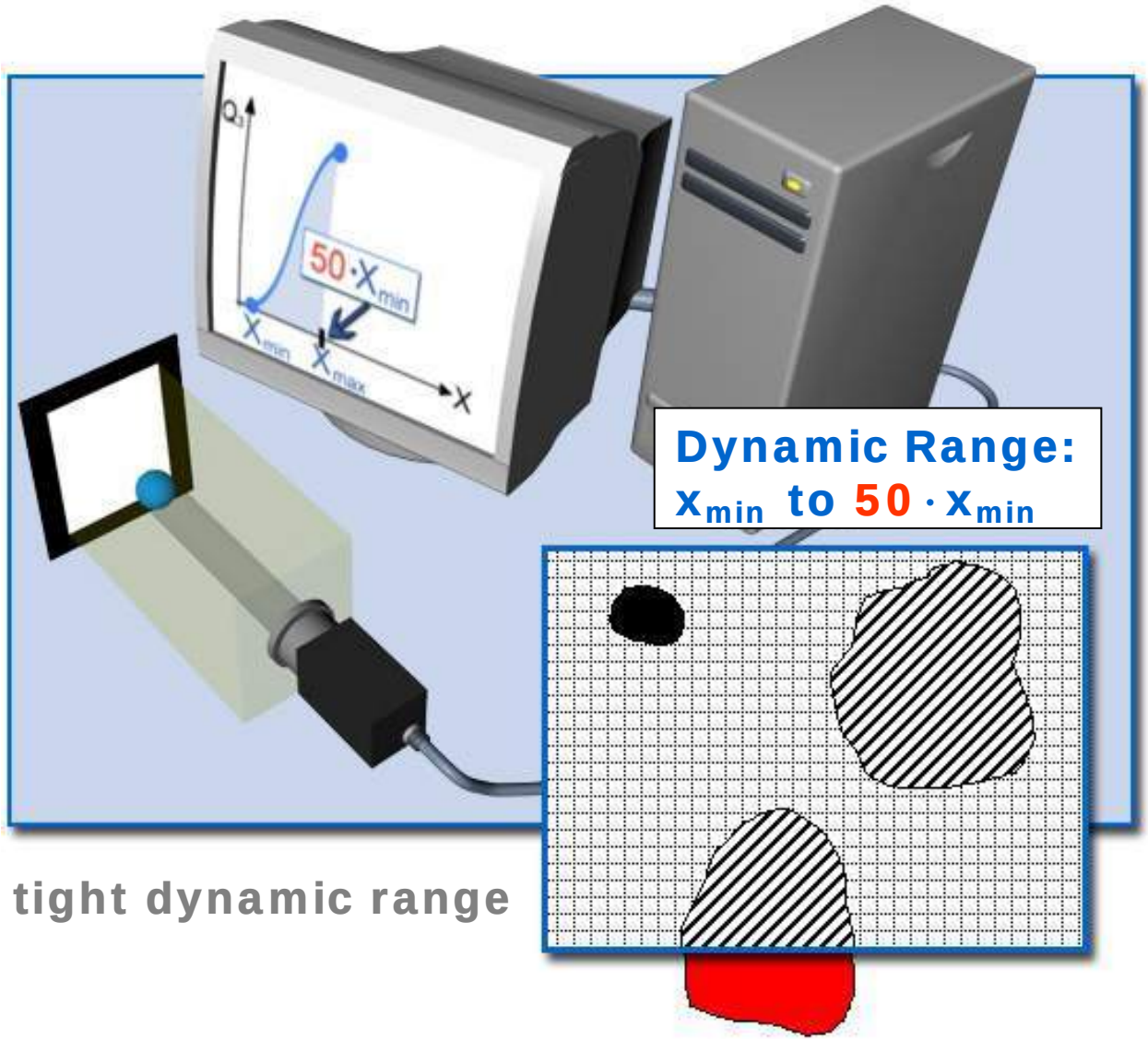
# One Camera Setup




**Drawback**: tight dynamic range



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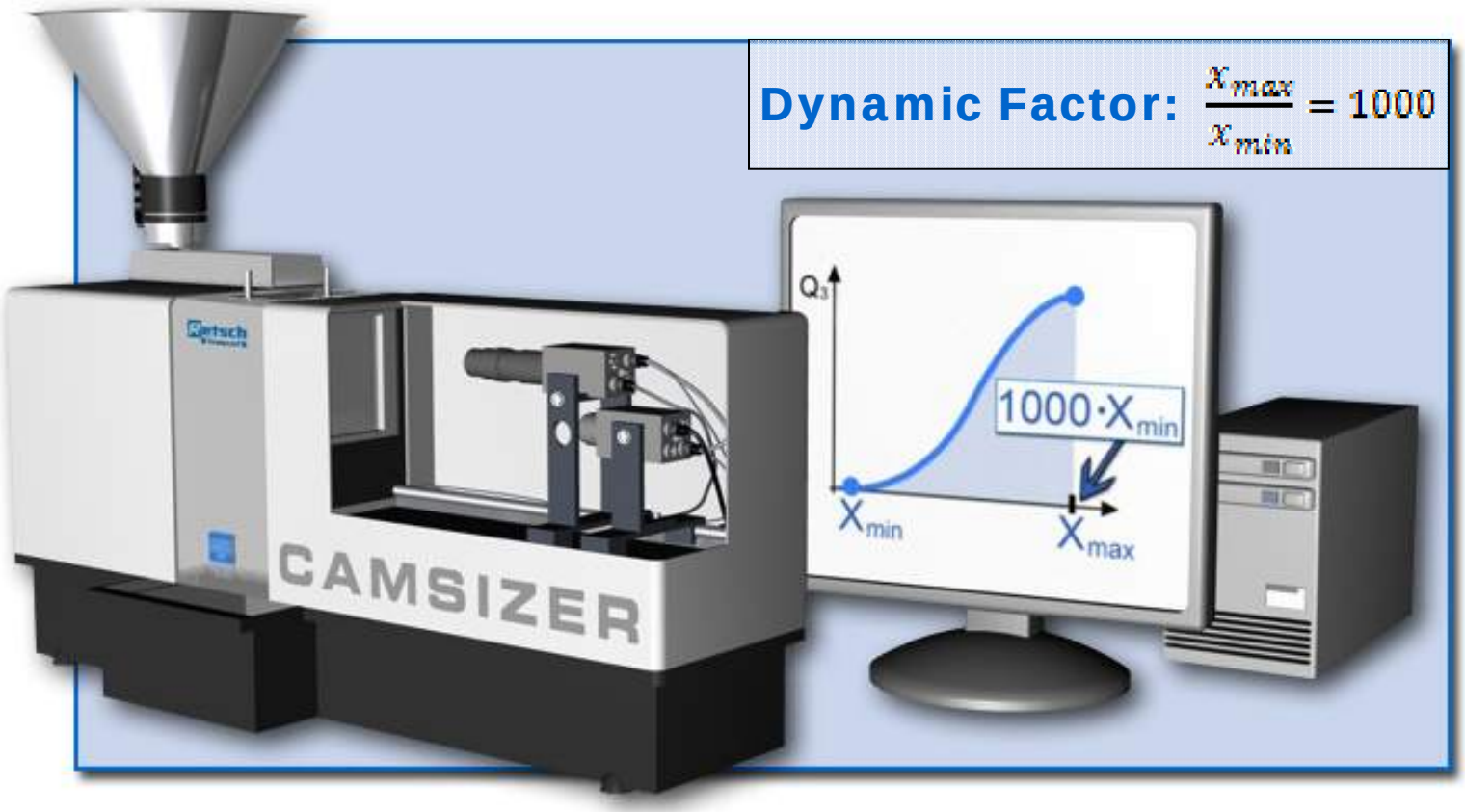
# CAMSIZER Principle (Two Cameras)




## Advantages

- Precise full-frame images
- Wide dynamic range: 30µm to 30mm

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# Content

## Device

1. Measuring Principle
2. Measurement Results
   - Size
   - Shape


## Application

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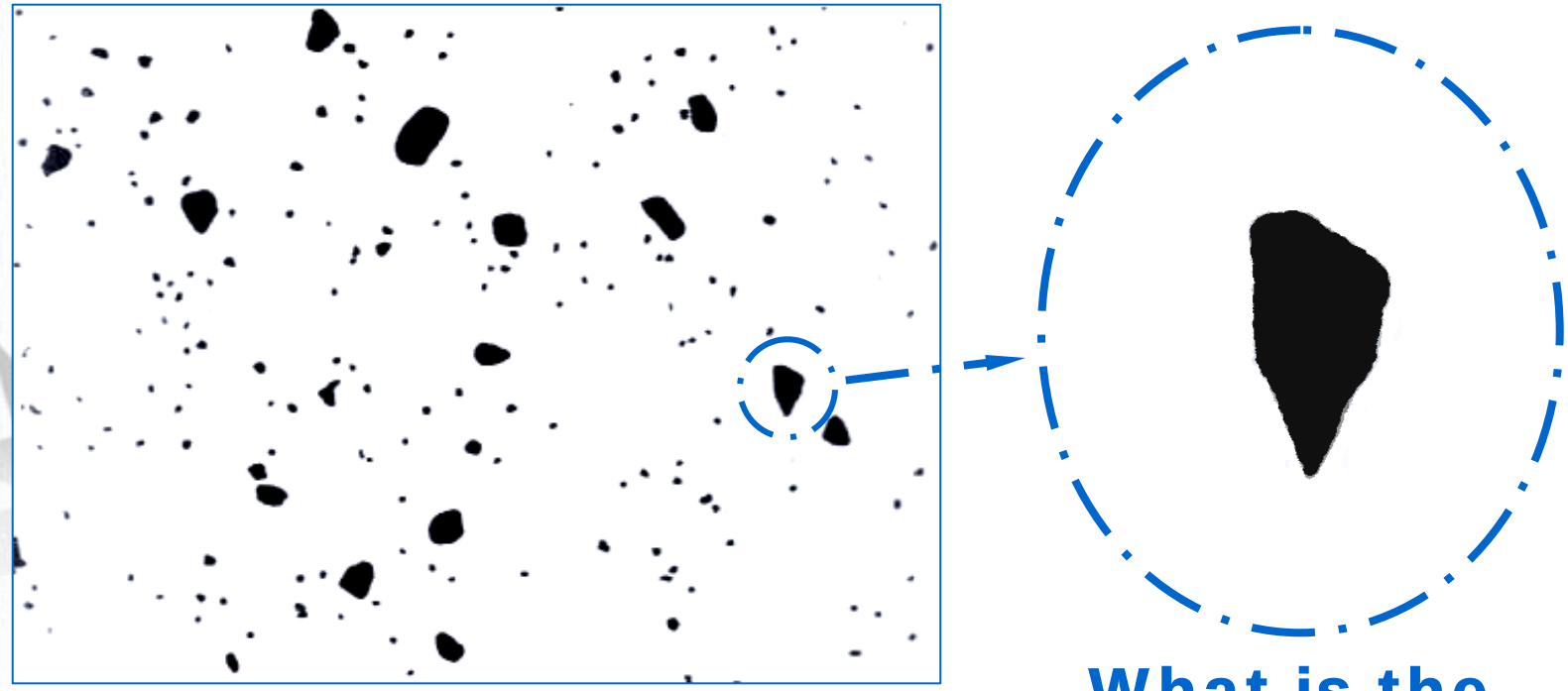
# Particle Size



**What is the size of this particle?**



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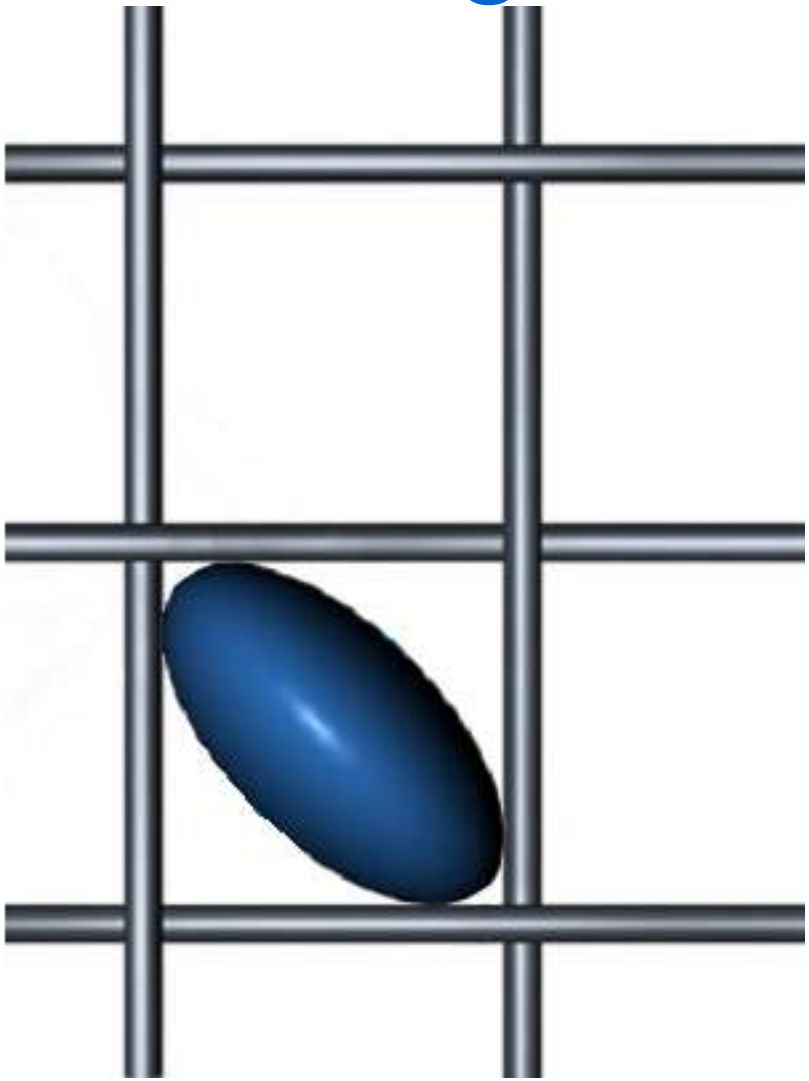
# Measuring Principle:



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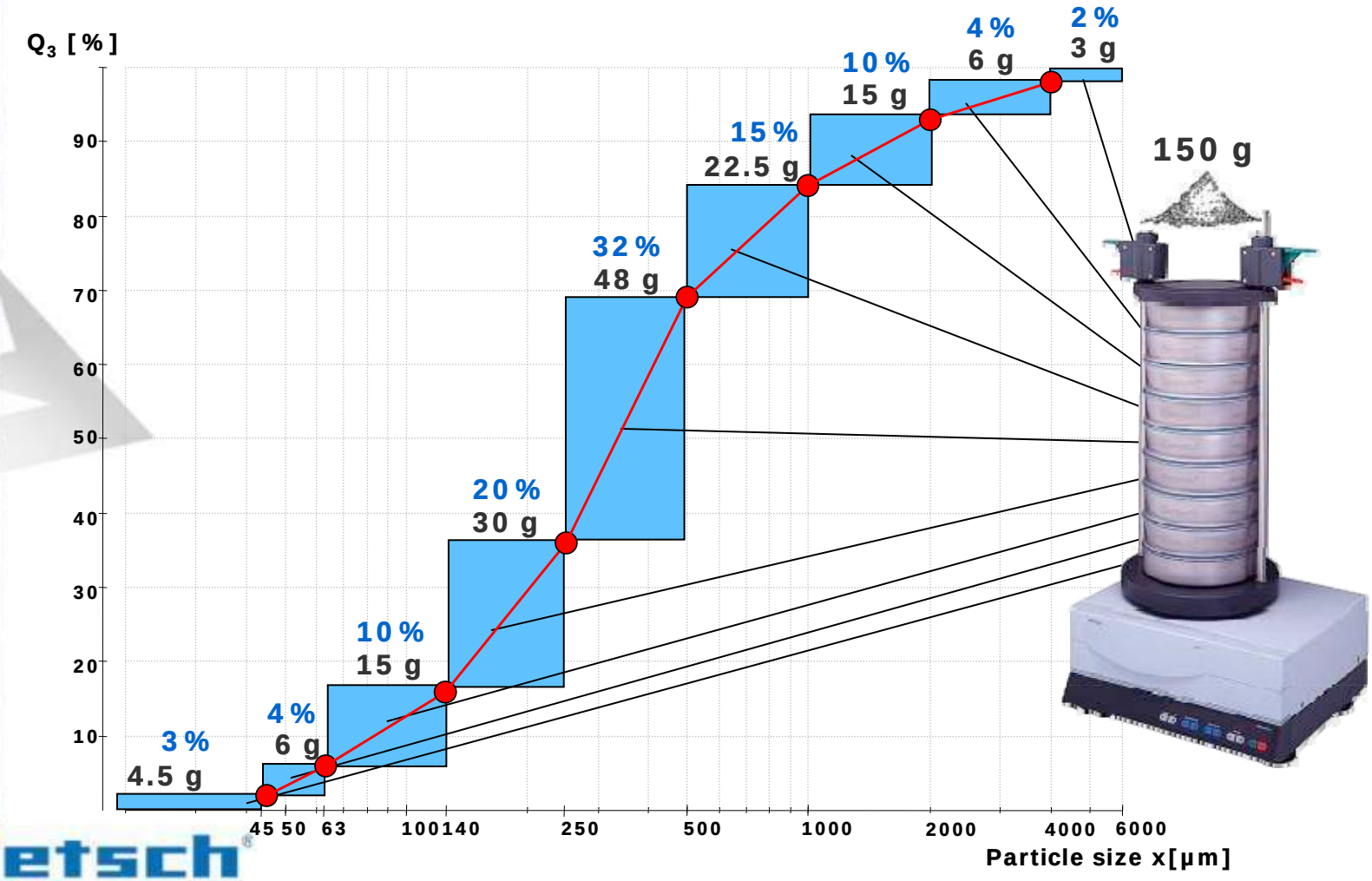
# Visualization
## Cumulative distribution

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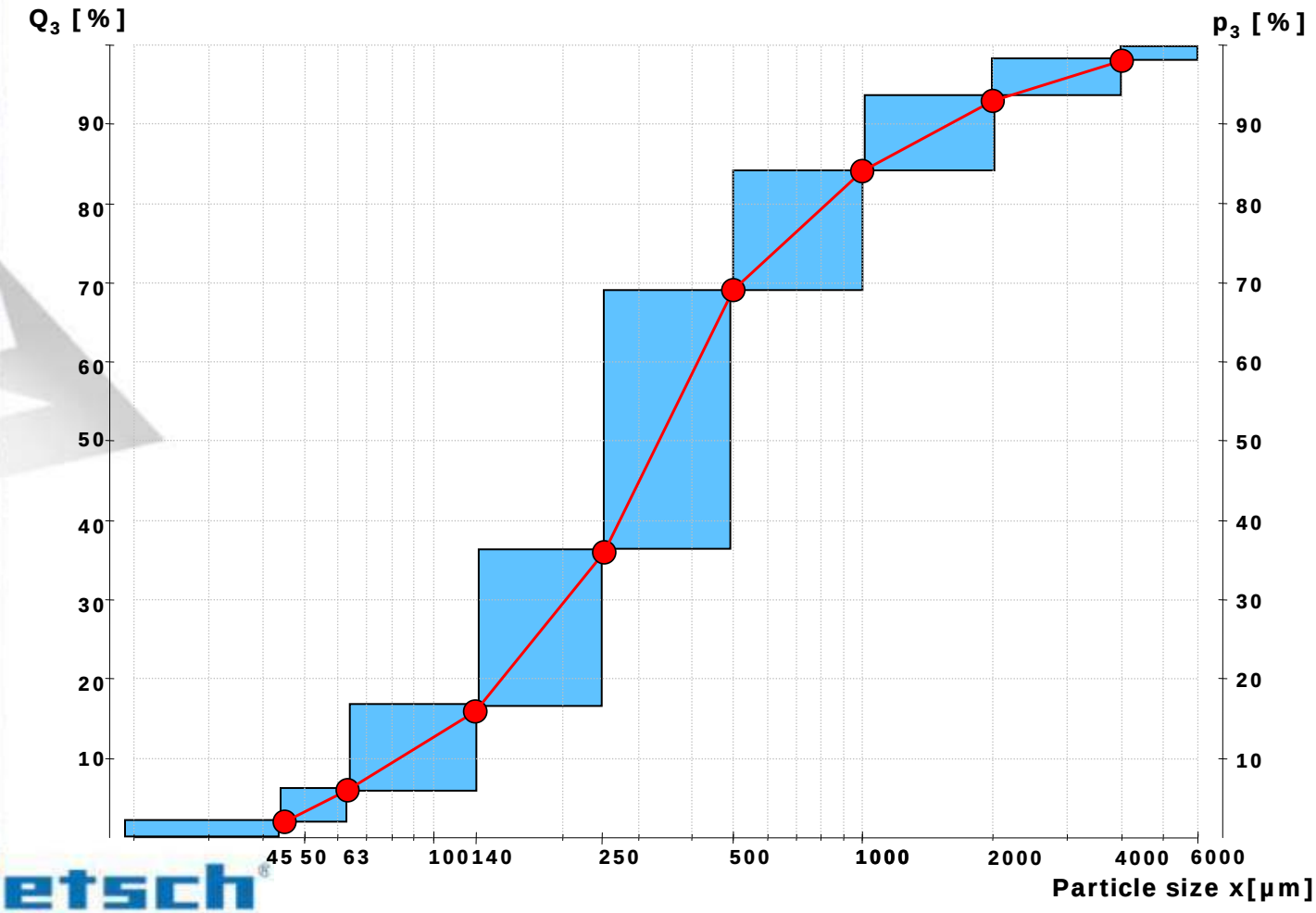
# Visualization
## Cumulative distribution

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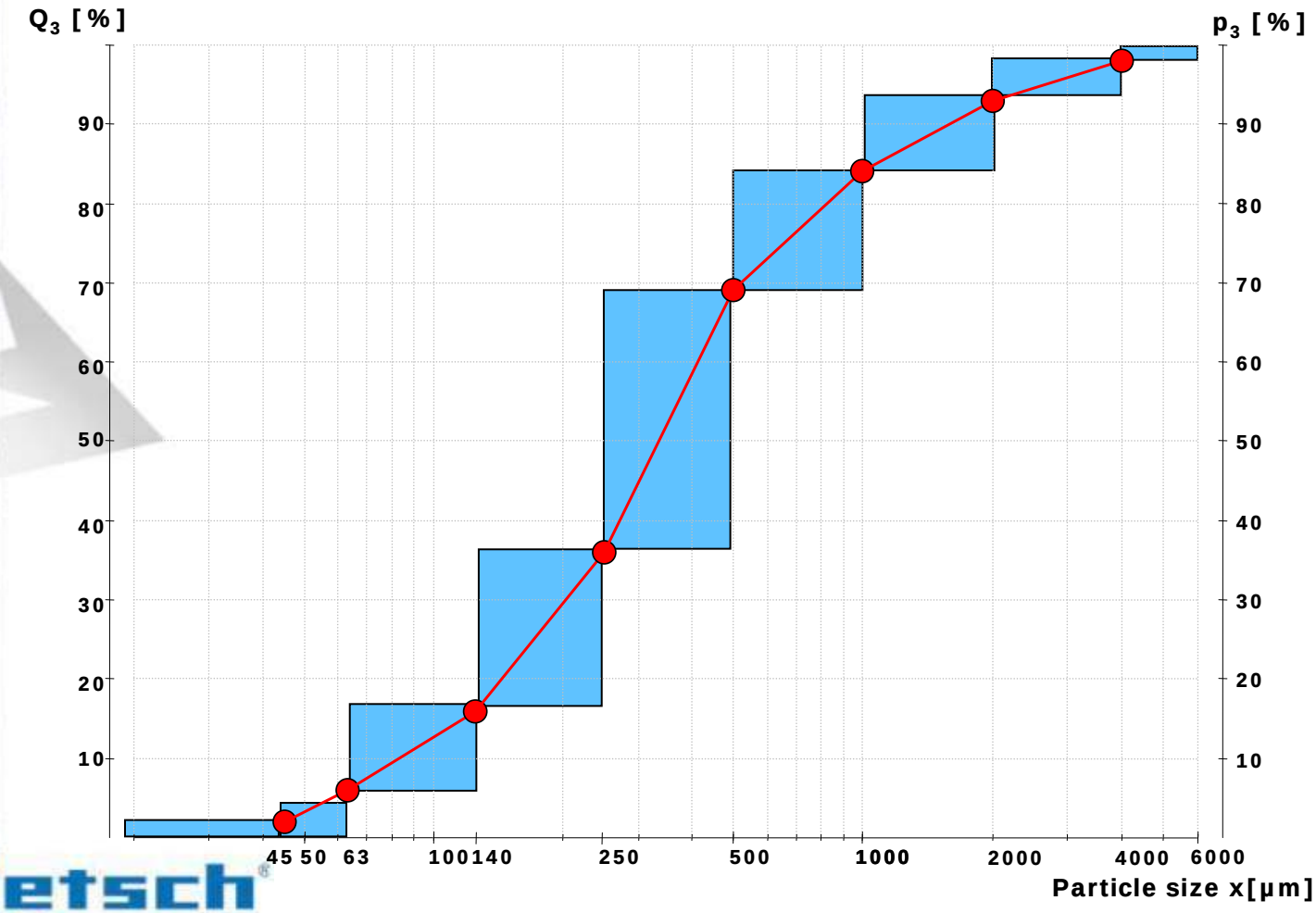
# Visualization
## Cumulative distribution

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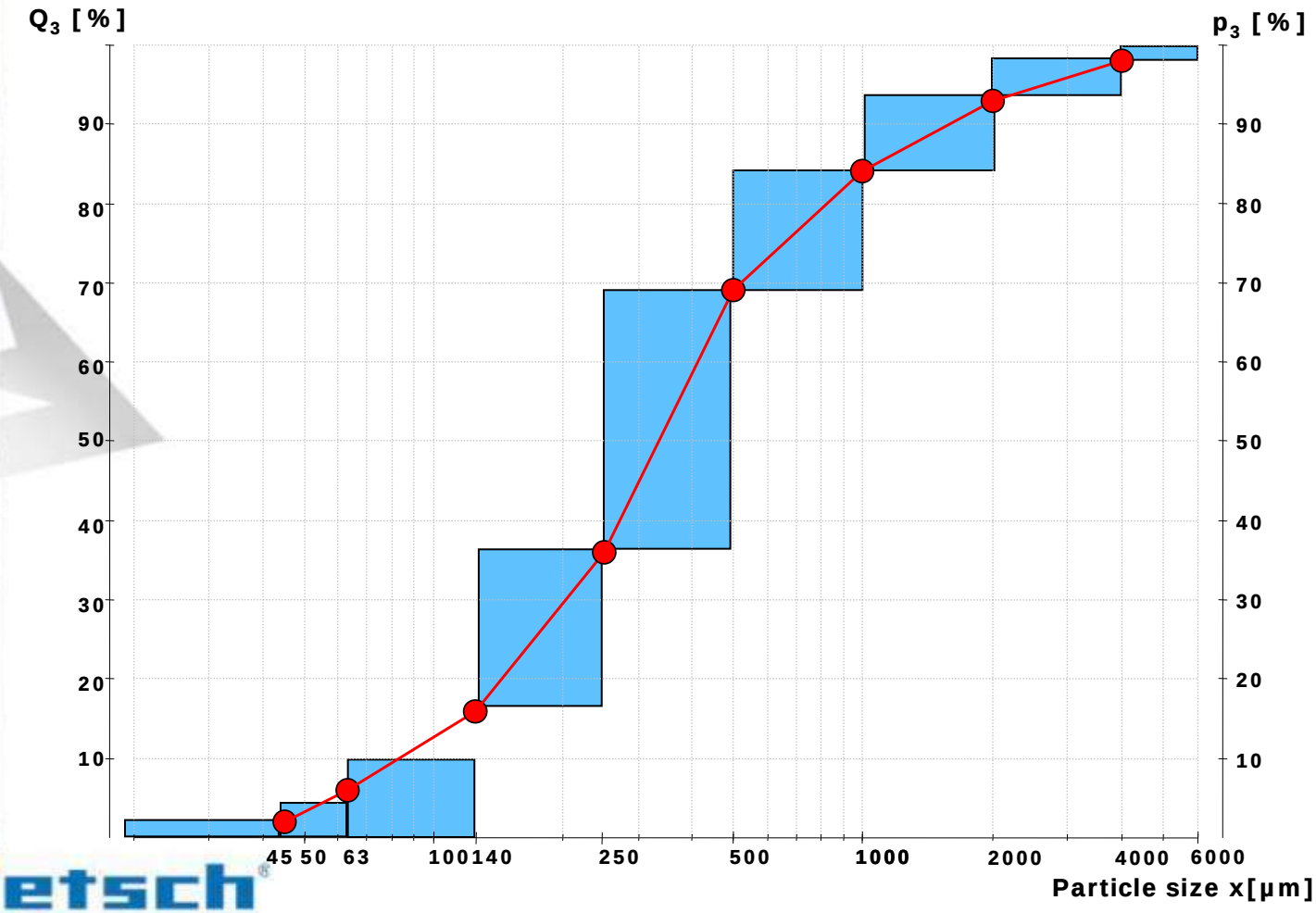
# Visualization
## Cumulative distribution

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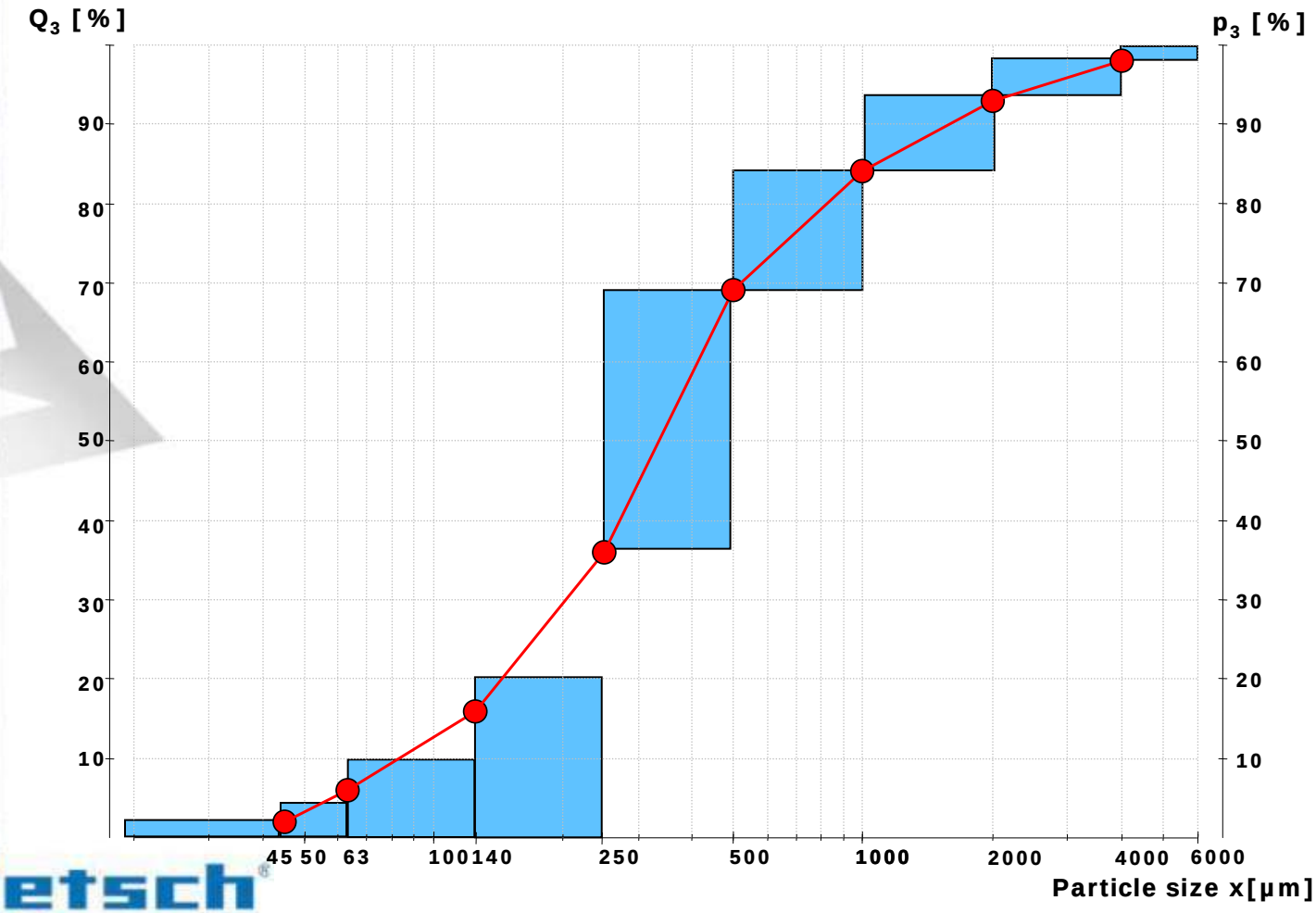
# Visualization
## Cumulative distribution

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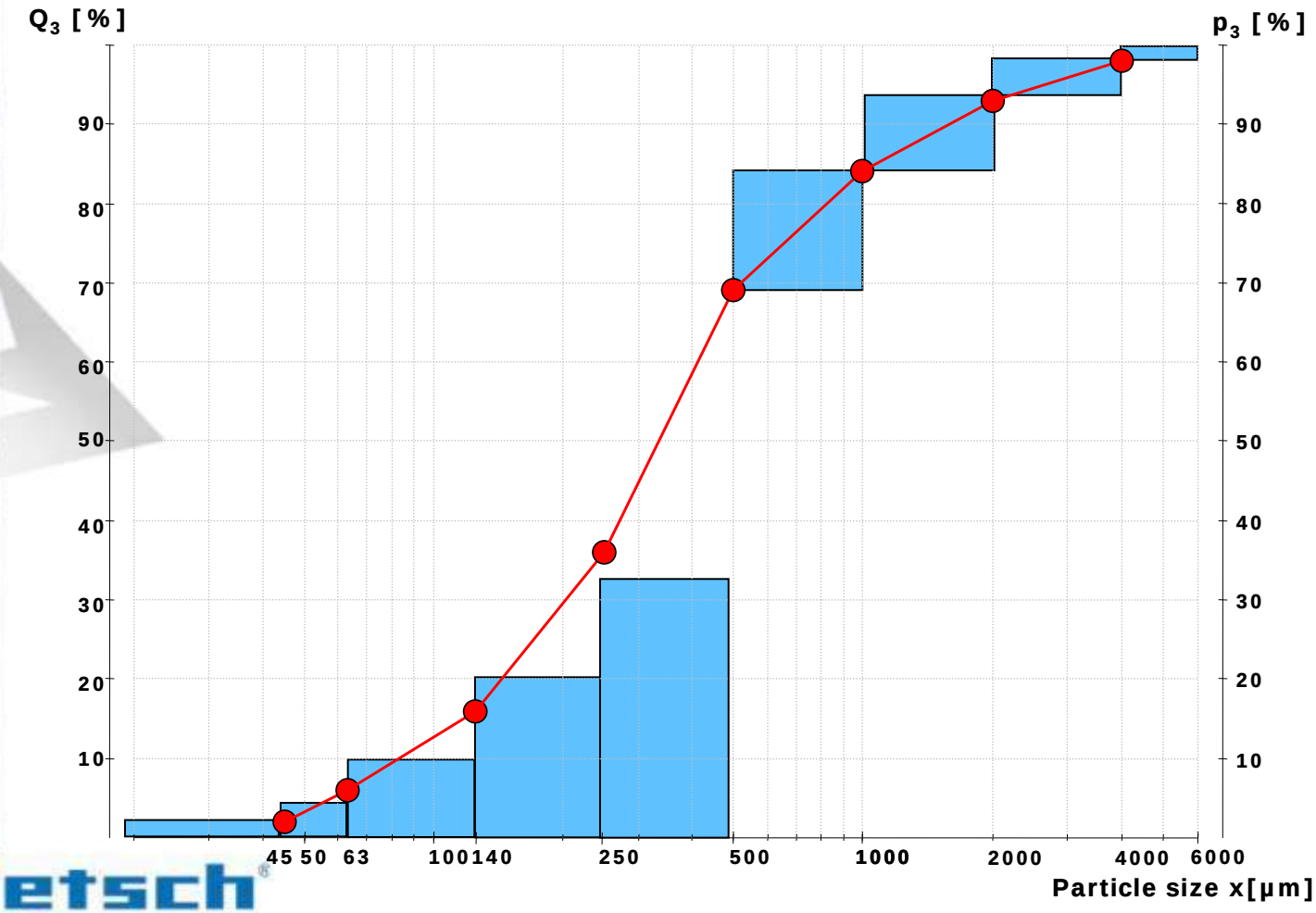
# Visualization
## Cumulative distribution

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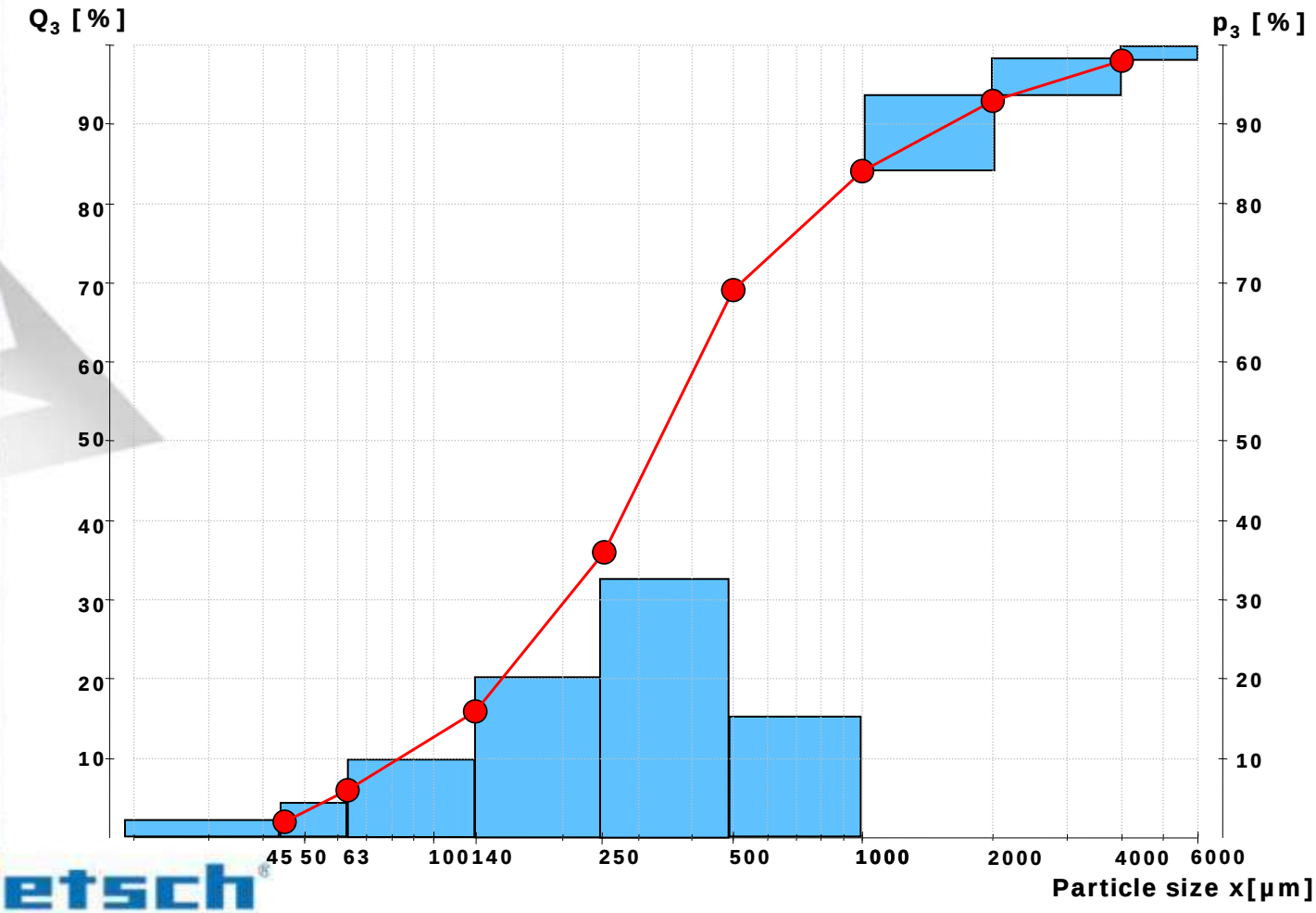
# Visualization
## Cumulative distribution

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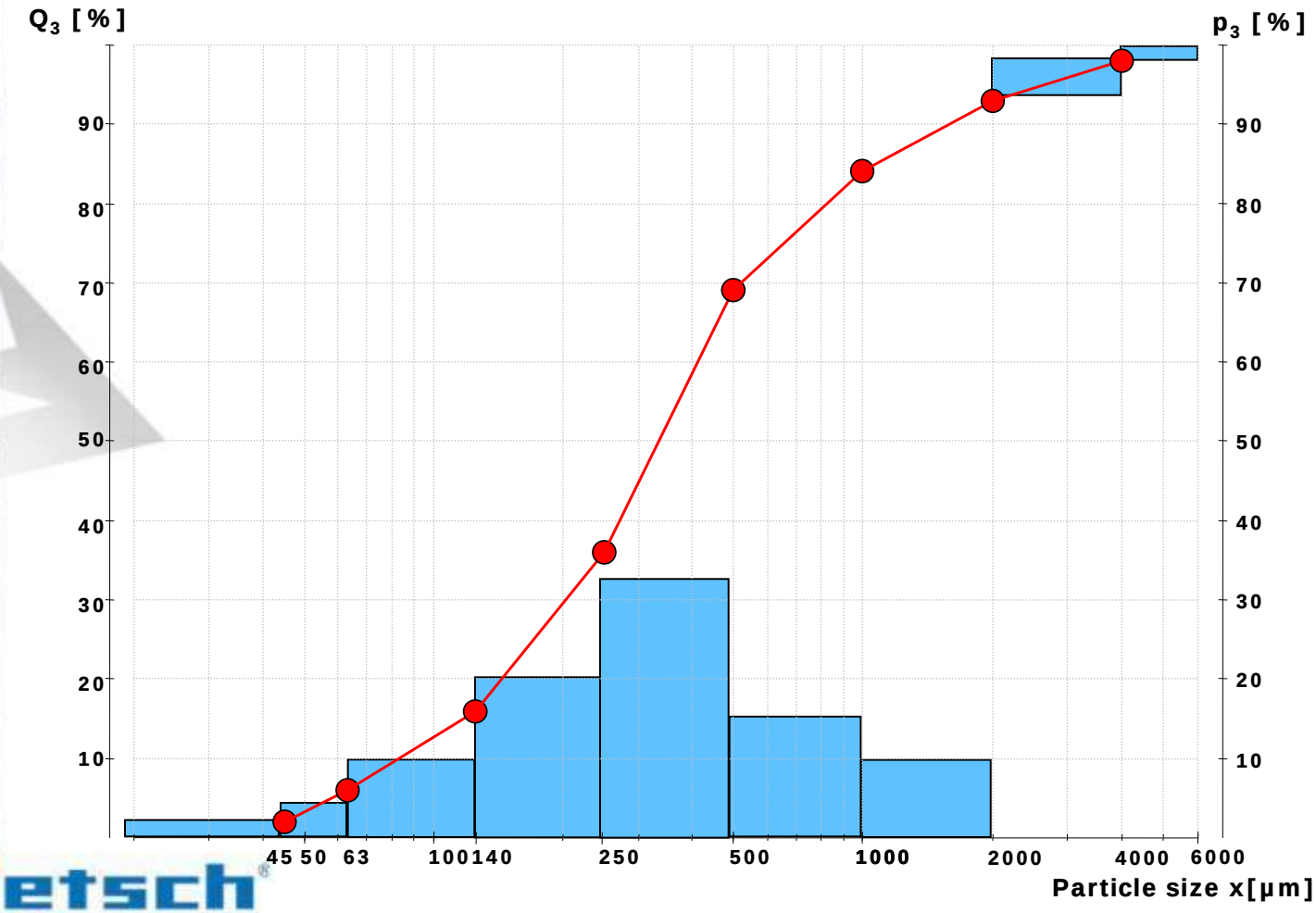
# Visualization
## Cumulative distribution

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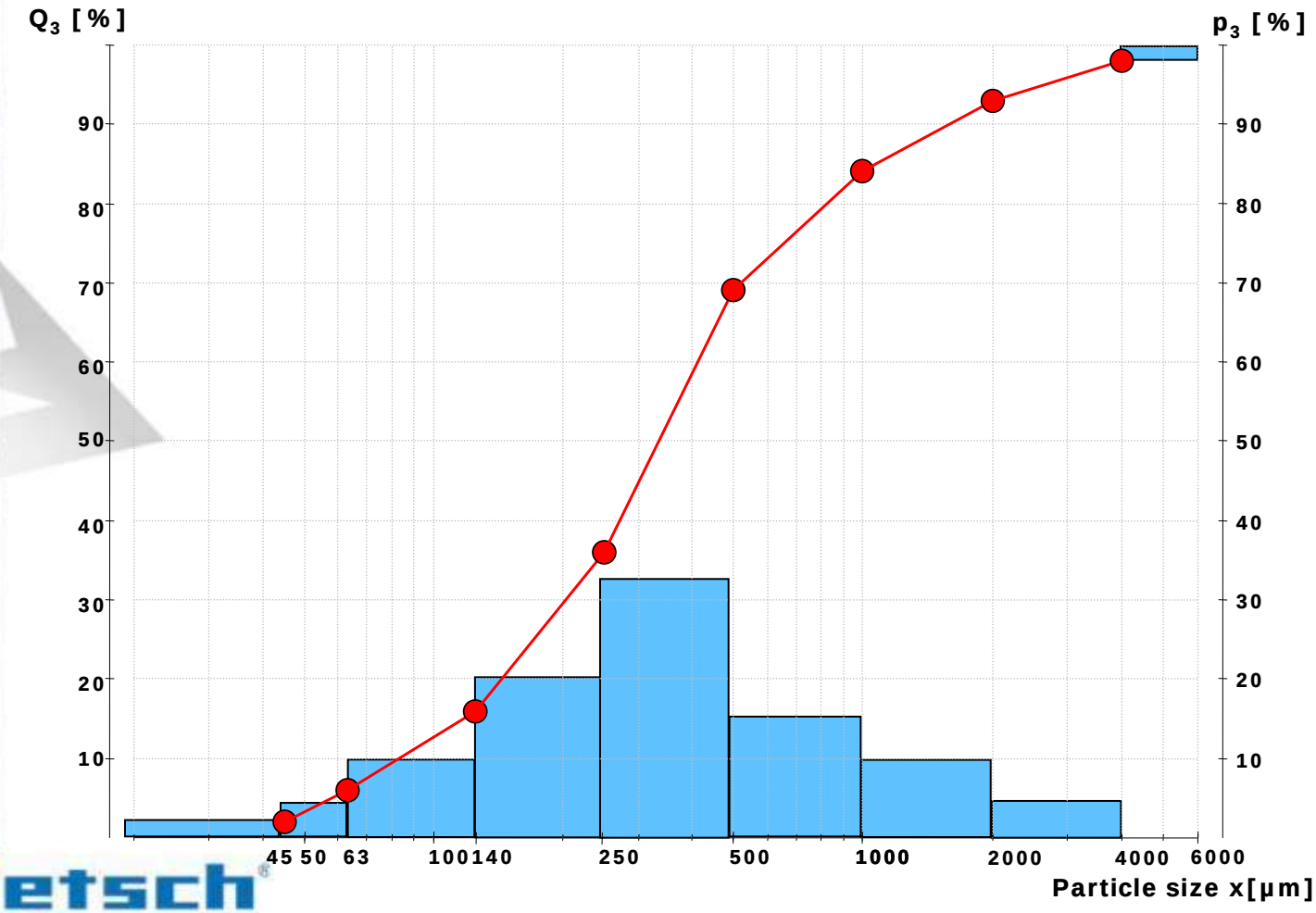
# Visualization
## Cumulative distribution

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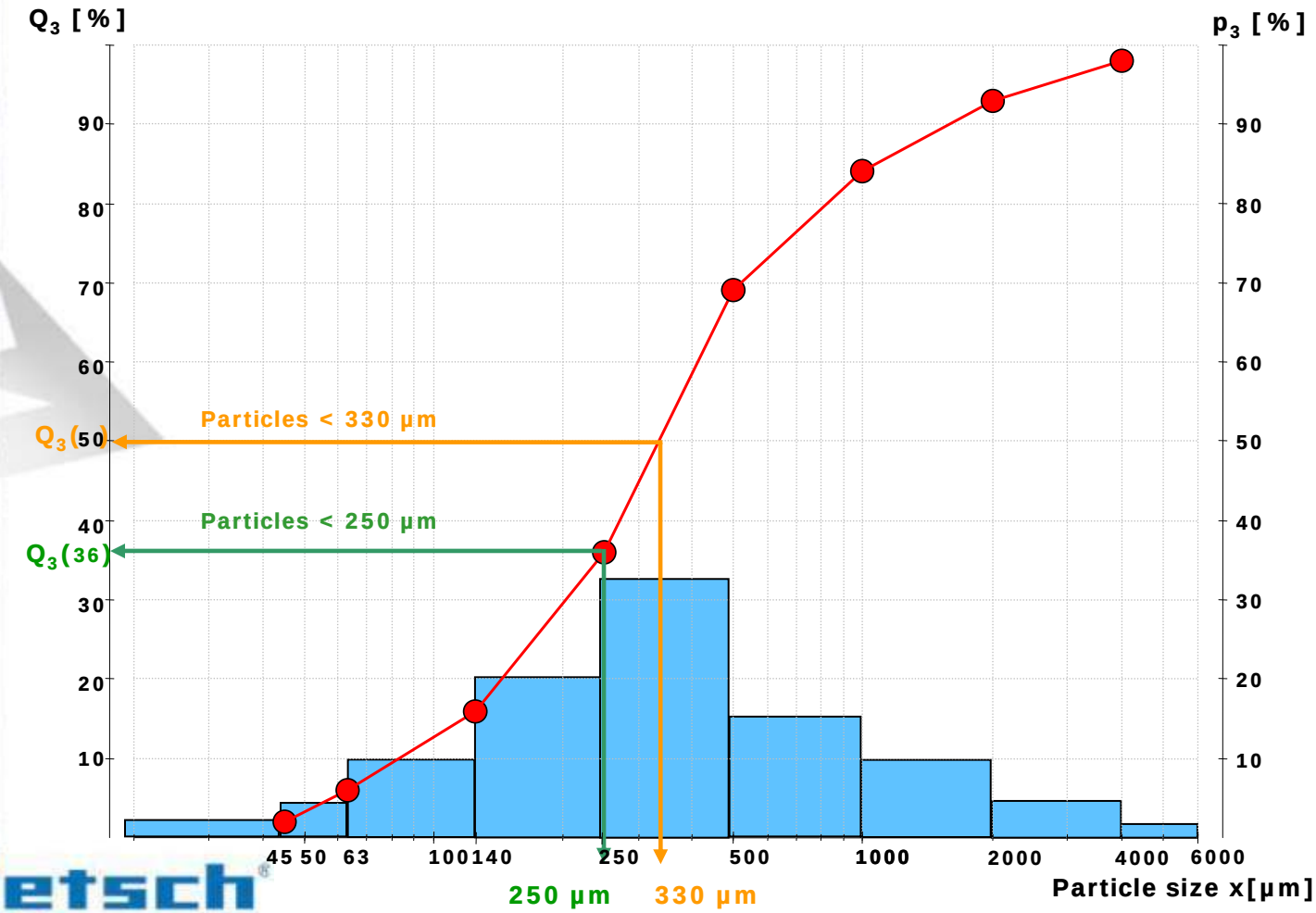
# Visualization

## Cumulative distribution & Fractions

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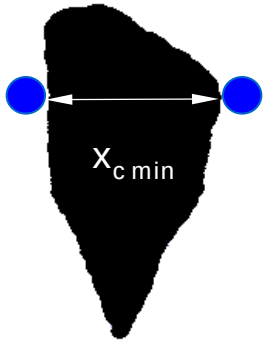
# Particle Size

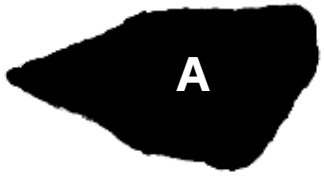
$x_{c\,min}$

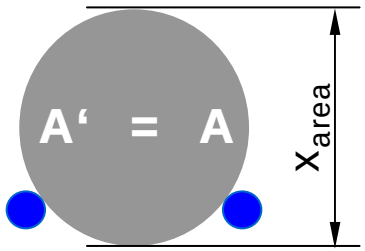
"width"




$x_{area}$

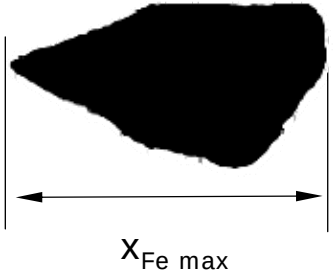
"diameter over projection surface"




$x_{Fe\,max}$

"length"




**CAMSIZER results are compatible with sieve analysis**



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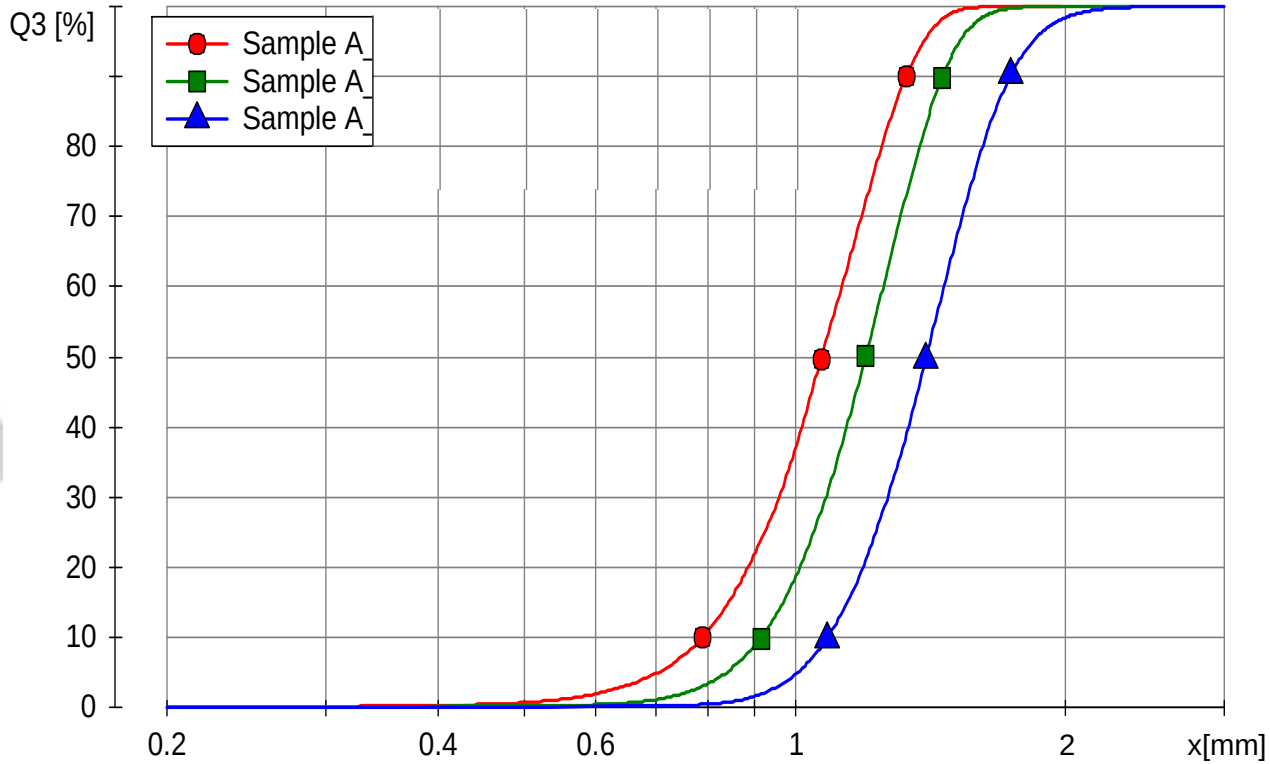
# Comparison of Size Definitions

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




different size definitions ⟷ different results



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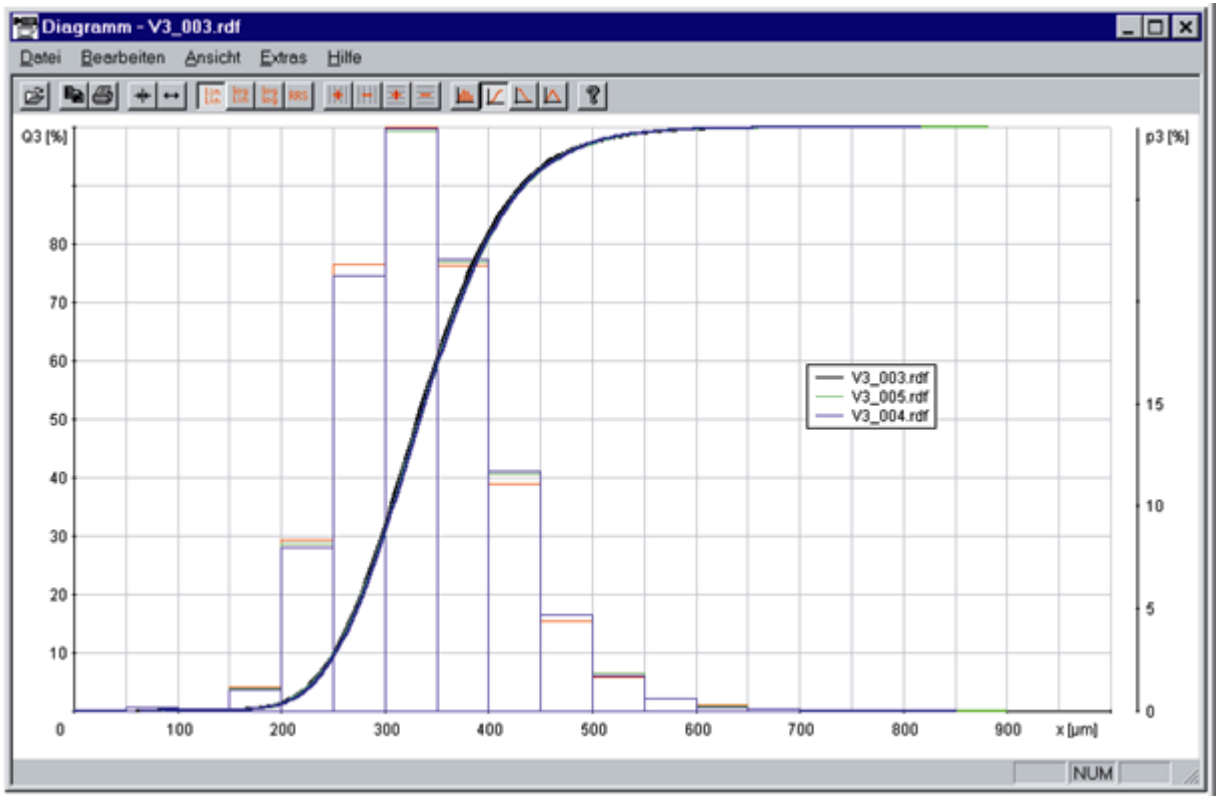
# Reproducibility




**Calibration with traceable standard**

**=> Absolute accuracy**

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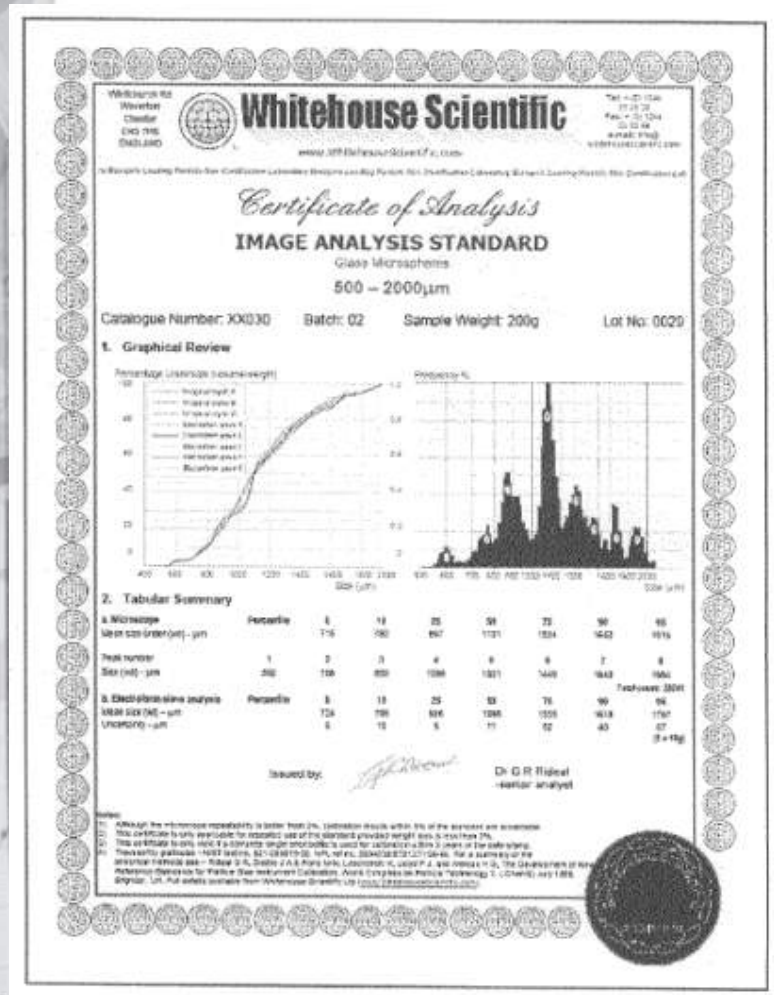
# Whitehouse Standard XX030



Whitehouse Scientific

*Certificate of Analysis*

IMAGE ANALYSIS STANDARD

Glass Microspheres

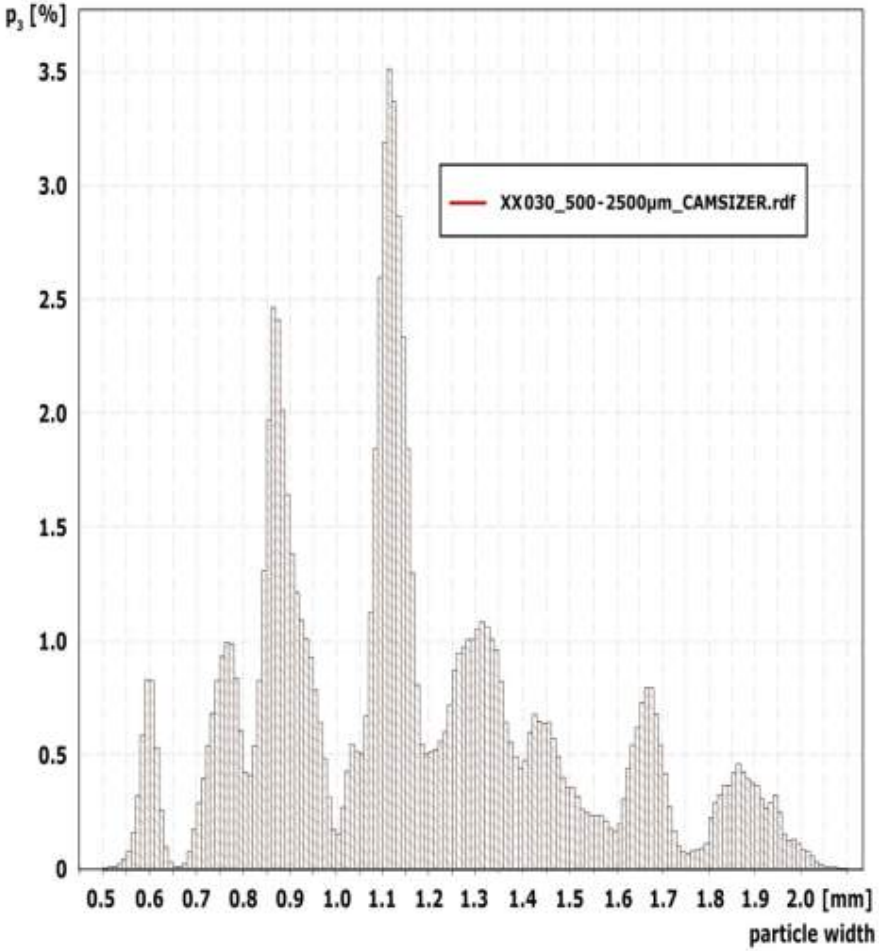
500 – 2000µm

Catalogue Number: XX030 Batch: 02 Sample Weight: 200g Lot No: 0020

1. Graphical Review

2. Tabular Summary

Issued by: Dr G R Rideal



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# Maintenance-Free by Venturi-Flushing




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# Content

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## Application

3. CAMSIZER Applications in the Fertilizer Industry
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# Places of Installation

- laboratories
- next to manufacturing line
- optimized quality control of product and process
- research institutes / universities

## Examples of Applications

- fertilizer
- salt
- crop protection
- pharmaceuticals
- food (human and animals)
-
-

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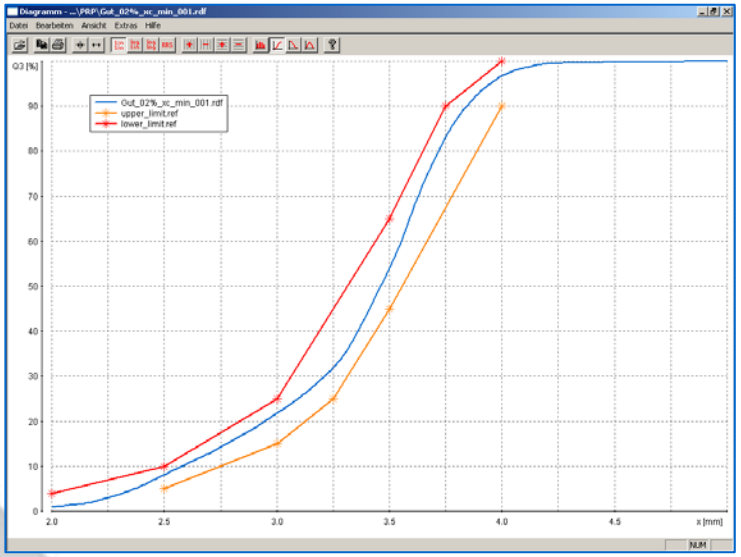
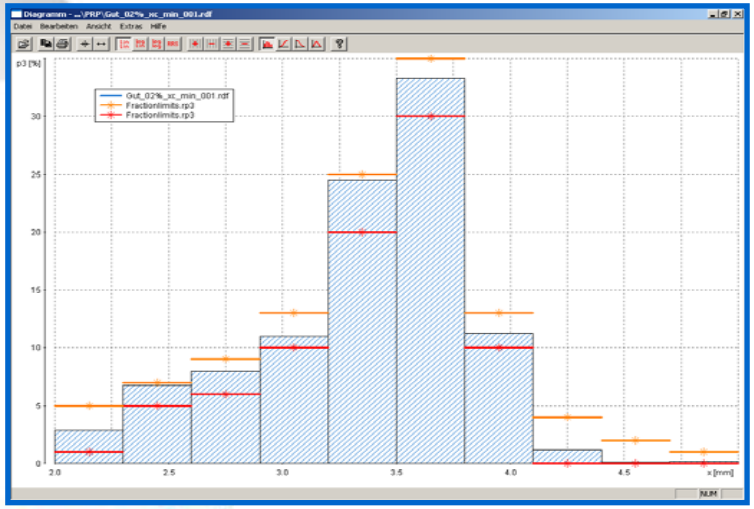
# Quality Control

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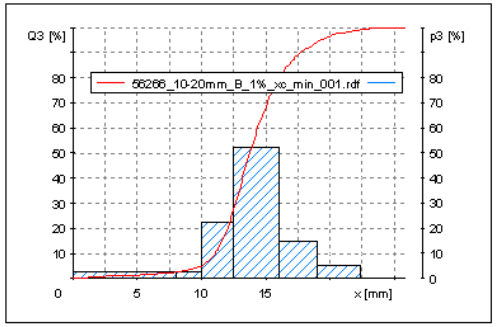
**Reg. 56266; 10-20mm**

| | |
|---|---|
| Company: | Mavit as (Skanlab) |
| User: | T. Pruschitzki |
| Result file: | C:\...MSIZER-SkanLab-Denmark\CAMDATM\axit-Coarse\56266 10-20mm B 1% xc min 001.rdf |
| Task file: | C:\...n-5GB\CAMSIZER-SkanLab-Denmark\CAMSYS\RT841 Skanlab Letklinker10-20 B 1%.afg |
| Time: | 3.11.2005 , 9:09 , duration 38 min 2 s at 1.0 % covered area, image rate 1:2 and 60 mm feeder |
| Particle model: | min(xc) |
| No. of particles: | CCD-B = 452560 , CCD-Z not active |
| Fitting: | no |
| Material: | Letklinker |
| Comment: | Druckluft, 60mm Rinne; Bildrate 1:2 |

| Size class | [mm] | p3 [%] | Q3 [%] | SPHT3 | b/3 | PDO |
|---|---|---|---|---|---|---|
| 0.000 - | 0.063 | 0.000 | 0.000 | 0.726 | 0.507 | 0 |
| 0.063 - | 8.000 | 2.529 | 2.529 | 0.831 | 0.681 | 449853 |
| 8.000 - | 10.000 | 2.542 | 5.071 | 0.907 | 0.739 | 231 |
| 10.000 - | 12.500 | 22.160 | 27.231 | 0.922 | 0.761 | 951 |
| 12.500 - | 16.000 | 51.976 | 79.207 | 0.933 | 0.800 | 1288 |
| 16.000 - | 19.000 | 14.945 | 94.152 | 0.939 | 0.811 | 194 |
| 19.000 - | 22.400 | 5.069 | 99.221 | 0.944 | 0.837 | 39 |
| > 22.400 | | 0.779 | 100.000 | 0.945 | 0.864 | 4 |




Characteristics

| | |
|---|---|
| Q3 [%] | x [mm] |
| 10.0 | 11.083 |
| 50.0 | 13.740 |
| 90.0 | 17.692 |
| SPAN3 = | 0.481 |
| U3 = | 1.297 |
| Sv = | 0.521 / mm |

Mean value SPHT3 = 0.927
Mean value b/3 = 0.785

User

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# UI and SGN values (Literature)

**Potash Processing in Saskatchewan – A Review of Process Technologies.**

By
**Carlos F. Perucca**
Sr. Metallurgist
AMEC Engineering & Construction Services Limited

The uniformity index (UI) is the particle size at which 95% of the material is retained, divided by the particle size at which 10% of the material is retained, multiplied by 100.

The UI is a measure of the uniformity of the size distribution of the product, when all the particles are exactly the same size, the size distribution curve is flat and the UI is 100%. Usual values of Ui for premium products are about 40%.

Figure 1 describes the calculation of the SGN number and UI index.

**Table 2: Fertilizer Grade Potash Specifications**

| Tyler Mesh | Opening [mm] | Typical (% cum.) | | |
|---|---|---|---|---|
| | | Granular | Coarse | Standard |
| 6 | 3.360 | 25.0 | 17.5 | |
| 8 | 2.380 | 73.0 | 53.0 | |
| 10 | 1.680 | 97.0 | 88.0 | 7.0 |
| 14 | 1.190 | 99.5 | 98.8 | 29.0 |
| 20 | 0.841 | | 99.5 | 50.0 |
| 28 | 0.600 | | 99.7 | 78.0 |
| 35 | 0.420 | | | 87.0 |
| 65 | 0.210 | | | 98.0 |
| 100 | 0.149 | | | |
| *SGN* | | *285* | *245* | *85* |
| *UI* | | *49* | *38* | *20* |

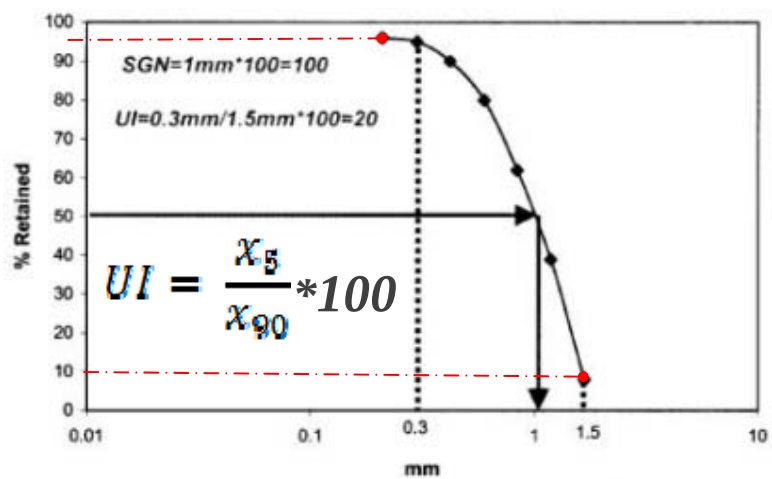


**Figure 1**: Calculation of the SGN number and UI index.



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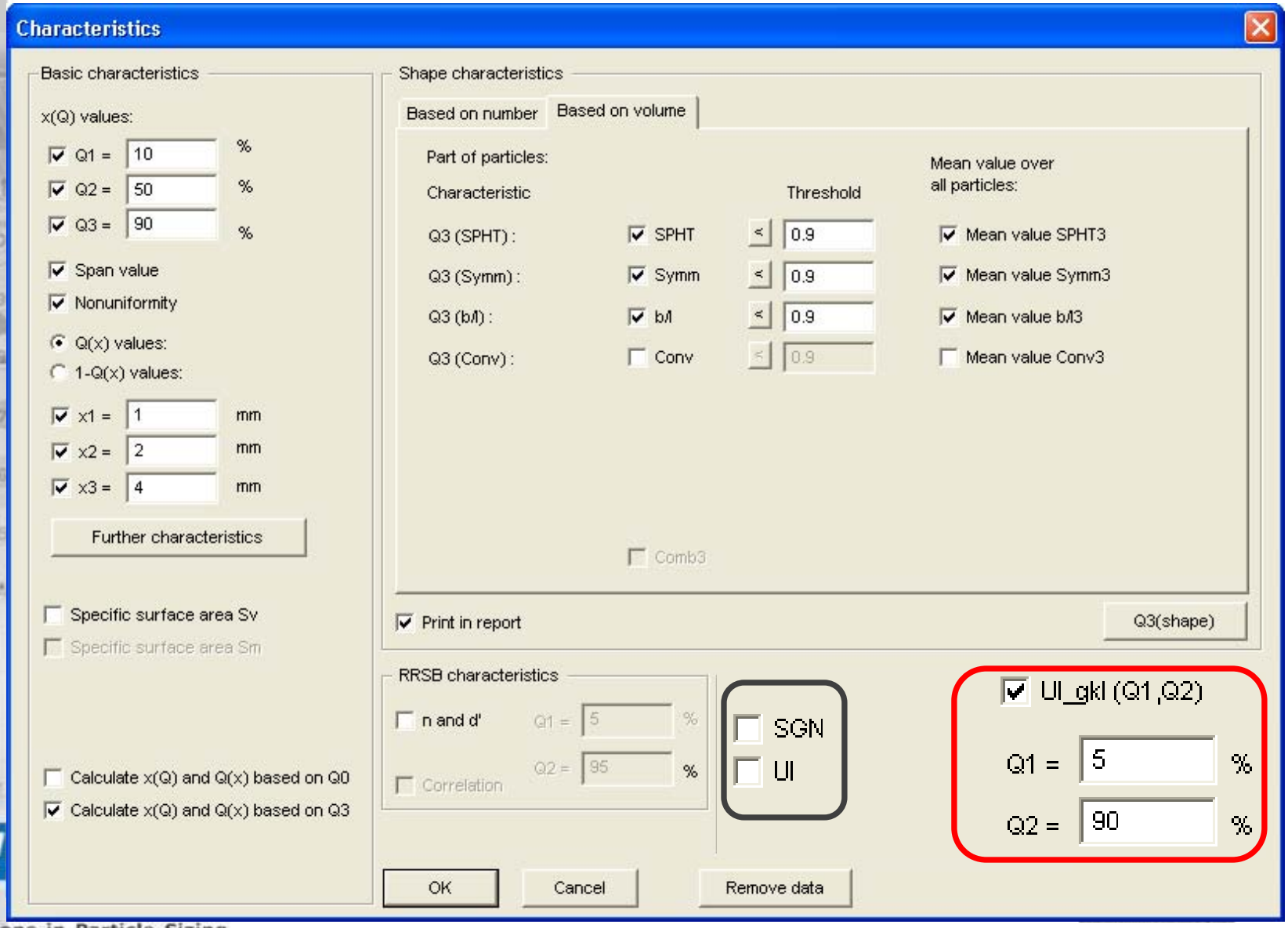
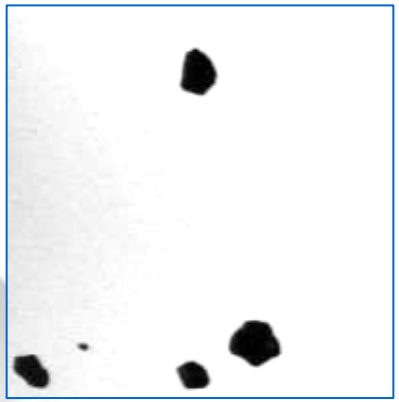
# SGN and UI values also with CAMSIZER

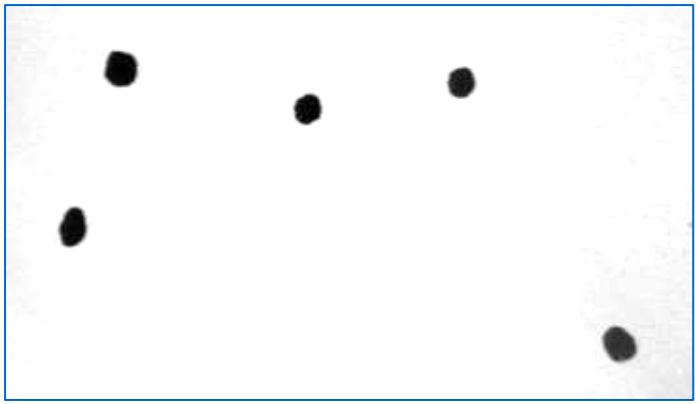


# CAMSIZER-Advantages Fertilizer

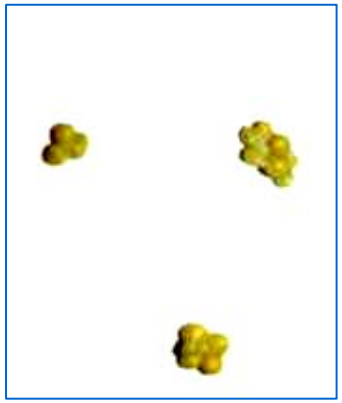
## Analysis of size and shape of



broken



granulated



coated

## fertilizers



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# Different Fertilizer Product Types, Different Production Methods, as well as Different Sizes and Shapes



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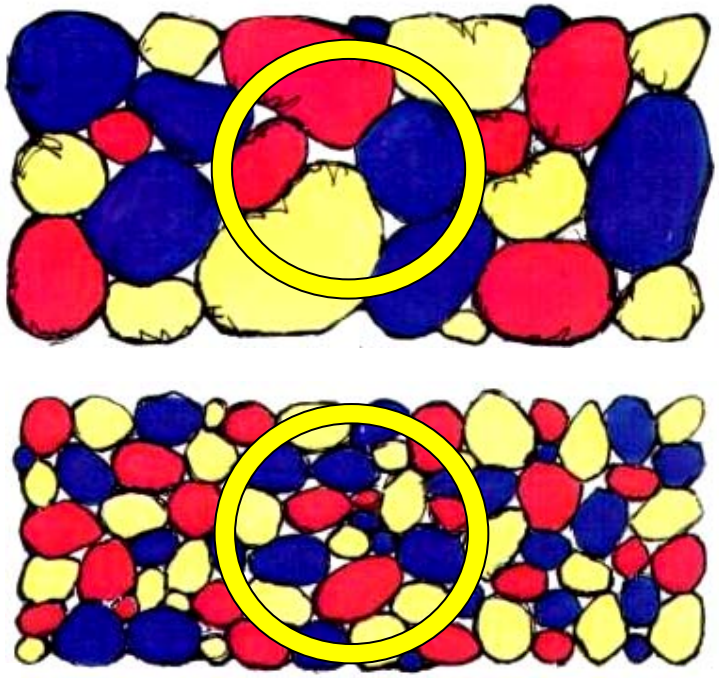
# Sample Quantity in Mass or Volume in Relation to the Particle Size

## ISO 13322-1

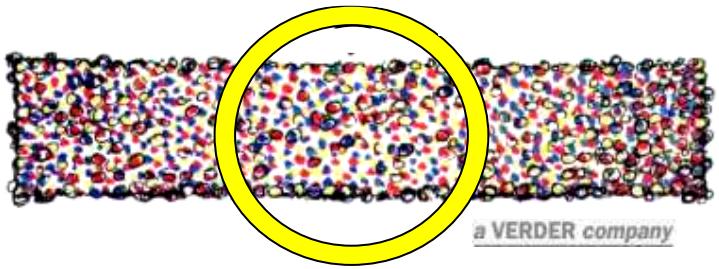
Tables 1 to 3 list, as a function of the geometric standard deviation $\sigma_g$ of the test powder, the number of particles to be measured, $N$, the median particle size of a cumulative mass distribution, $x_{50,3}$, the Sauter Diameter, $\bar{x}_{1,2}$, and the average mass diameter, $\bar{x}_{3,0}$, for the precision, respectively, $\delta$ = 0,05, 0,1 and 0,2. Here, the confidence coefficient probability, $P$, is equal to 0,95.

**Table 1- Number of particles to be measured $N$, $\delta$ = 0,05, $p$ = 0,95**

| $\delta$ | $\sigma_g$ | $x_{50,3}$ | $\bar{x}_{1,2}$ | $\bar{x}_{3,0}$ |
|---|---|---|---|---|
| 0,05 | 1,10 | 585 | 389 | 131 |
| | 1,15 | 1 460 | 934 | 294 |
| | 1,20 | 2 939 | 1 808 | 528 |
| | 1,25 | 5 223 | 3 103 | 843 |
| | 1,30 | 8 526 | 4 920 | 1 247 |
| | 1,35 | 13 059 | 7 355 | 1 750 |
| | 1,40 | 19 026 | 10 504 | 2 363 |
| | 1,45 | 26 617 | 14 457 | 3 096 |
| | 1,50 | 36 007 | 19 295 | 3 956 |
| | 1,55 | 47 358 | 25 093 | 4 952 |
| | 1,60 | 60 811 | 31 919 | 6 092 |



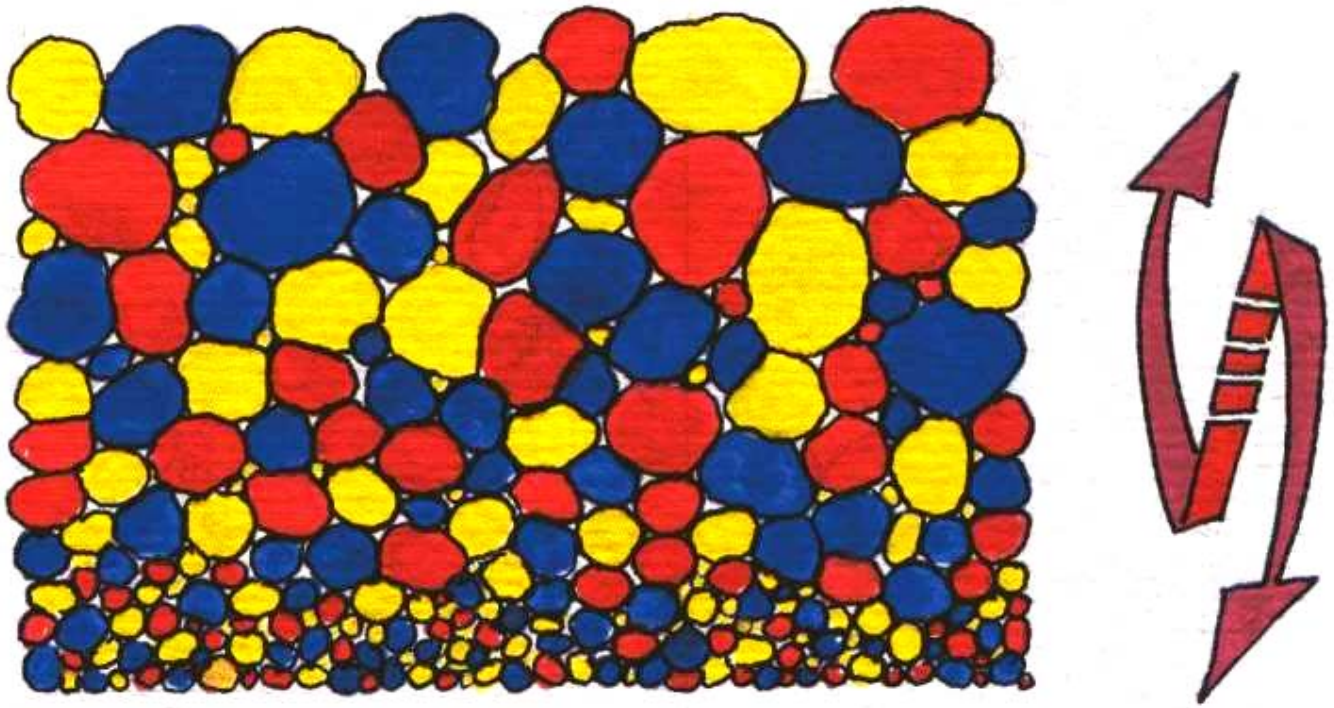
A Sufficient Sample Quantity is Based on the **Number** of Particles



# Separation of fine and coarse particles

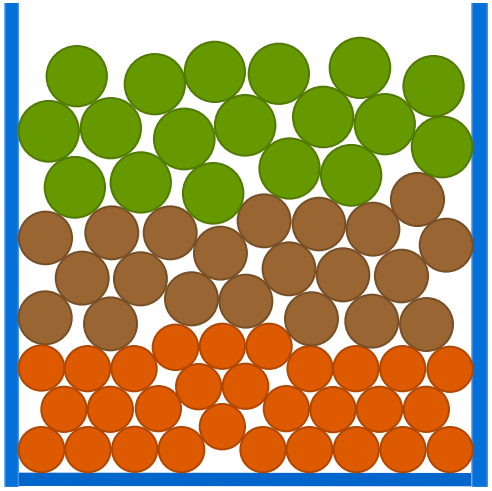
Separation happens during

- Transport processes (container, train and truck)
- Feeding processes (funnels, vibration feeders, belts)
- and Storage (bulk pile, silo)

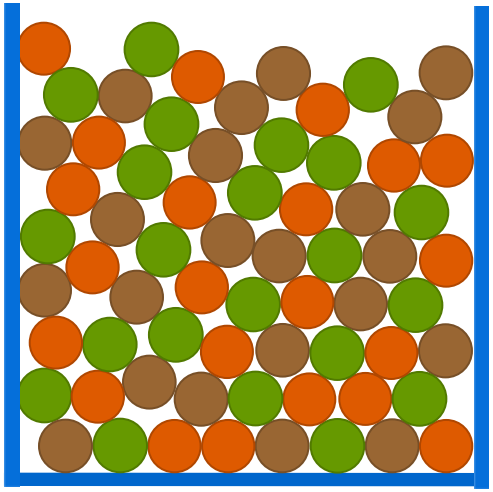


# Separation of fine and coarse particles

Separation happens because of different sizes



Particles having different sizes separate



Particles with similar sizes keep homogenety in mix



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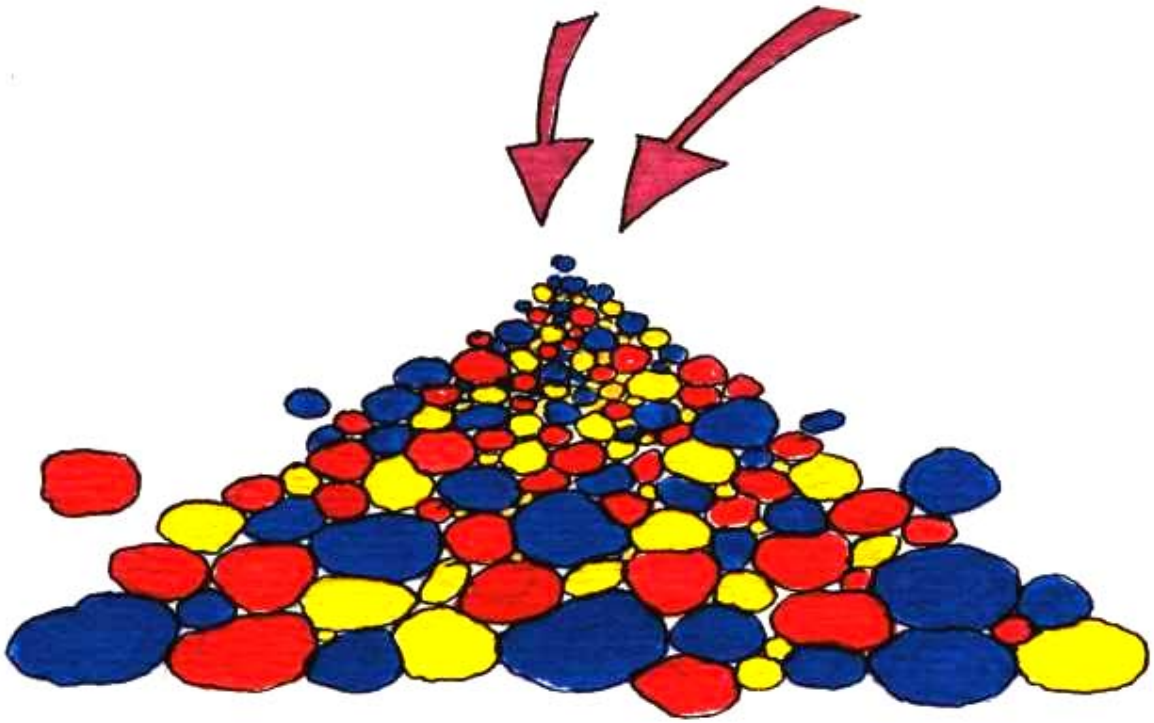
# Separation of fine and coarse particles

Segregation (separation by size) happens during

- Filling processes (silo)
- Feeding processes (bulk pile)
- Accumulation of fines in the middle of the pile

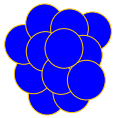


# How to attach small „Dust“ Particles to the larger Granules to get a „better“ Product?

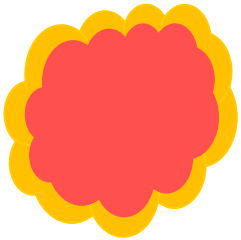
**Fertilizer producer coats the granules with petrochemical oil and amine to bind the dust to the granules for better handling to avoid accumulation of dust**

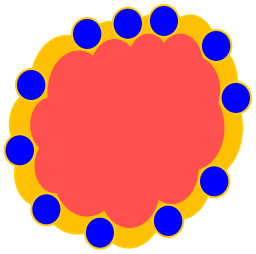
Large Granule

Dust Agglomerate



Large Granule with Coating





**Dust sticks to particle because of the Coating**



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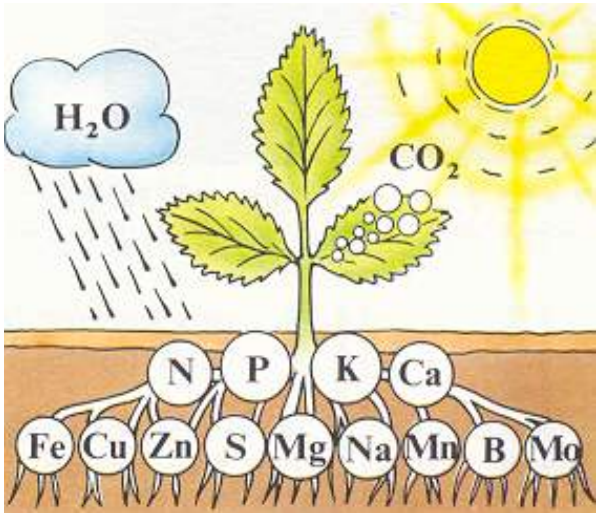
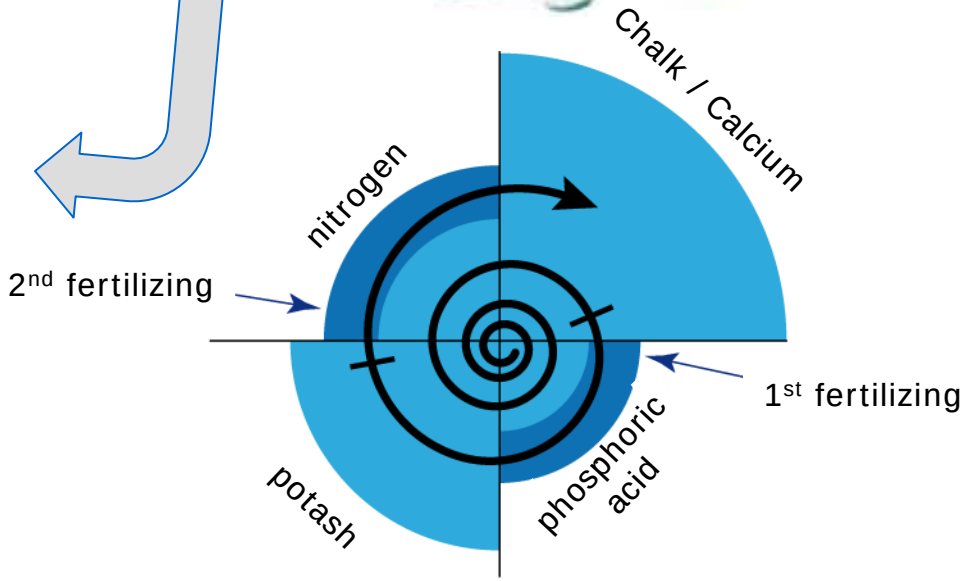
# Only complete Fertilizing provides good Nutrition

Plant availability




Optimum soil condition


**Anything else is wasted money and pollution of our environment**

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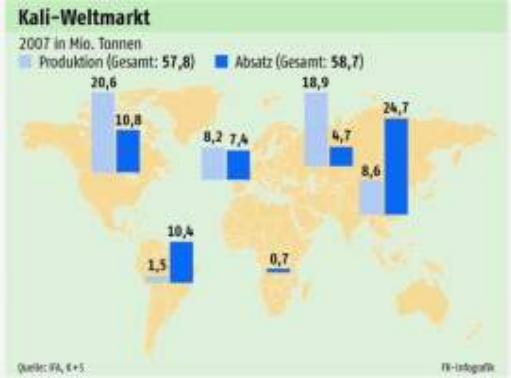
# A German Fertilizer Producer is mining and producing KCl, $MgSO_4$ and $KSO_4$




Before 2000 => Sieve analysis 0.05 – 2.5 mm
After 2000 => CAMSIZER analysis

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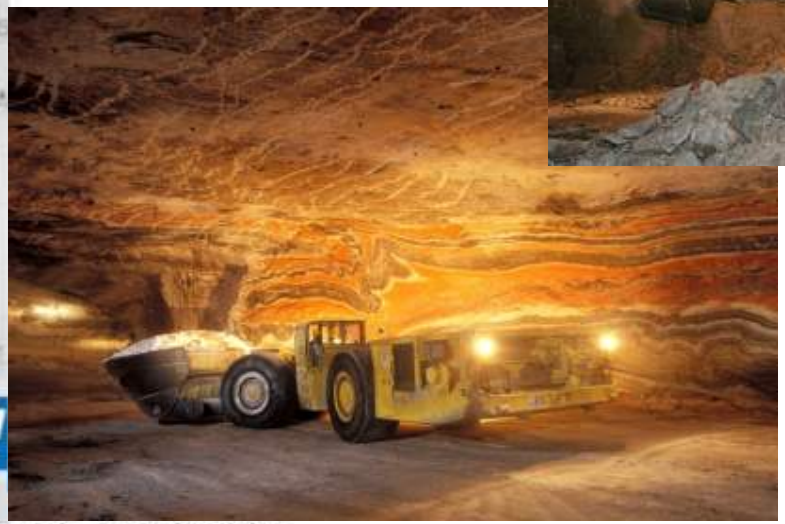
# Shaft Mining of Potash KCl



# Handling of Potash KCl



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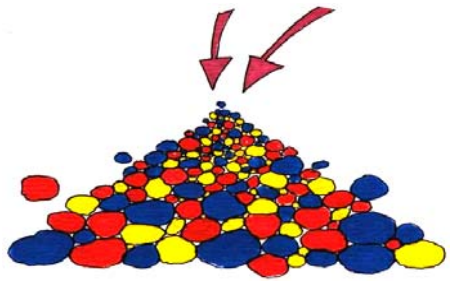
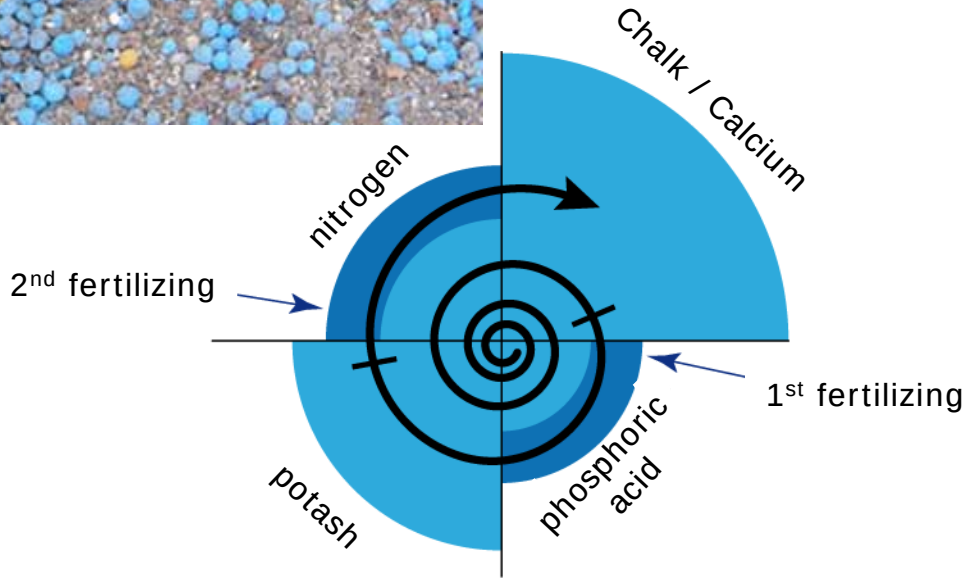
# Bulk Blending Companies and Farmers want the Same Size of the Components

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# Fertilizer in the Silo



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# Railcar Loading



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# Urea Fertilizer (also for AdBlue / BlueTec)



$OC(NH_2)_2$

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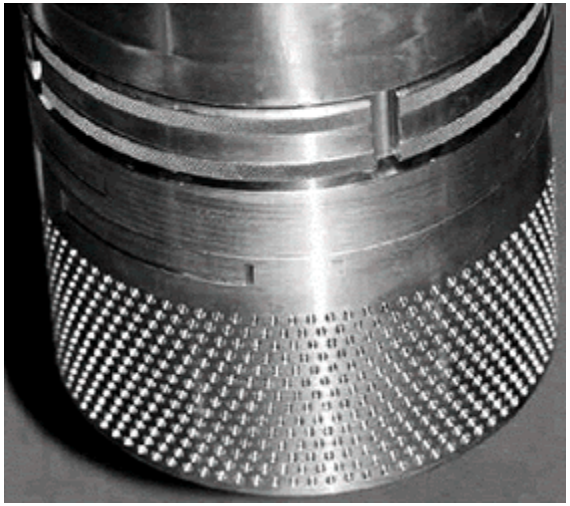
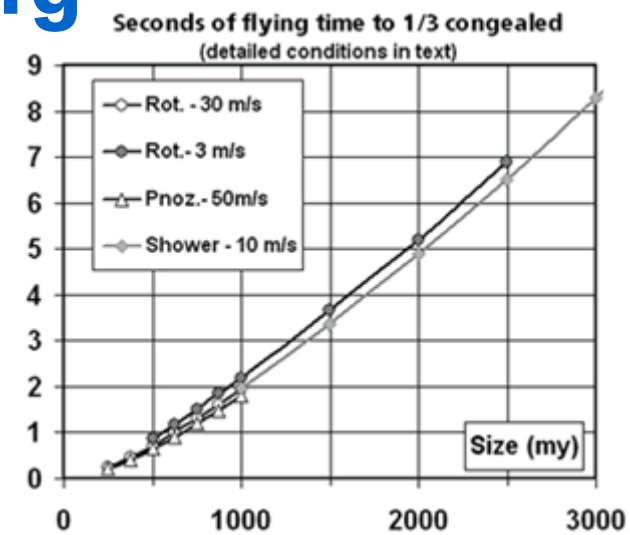
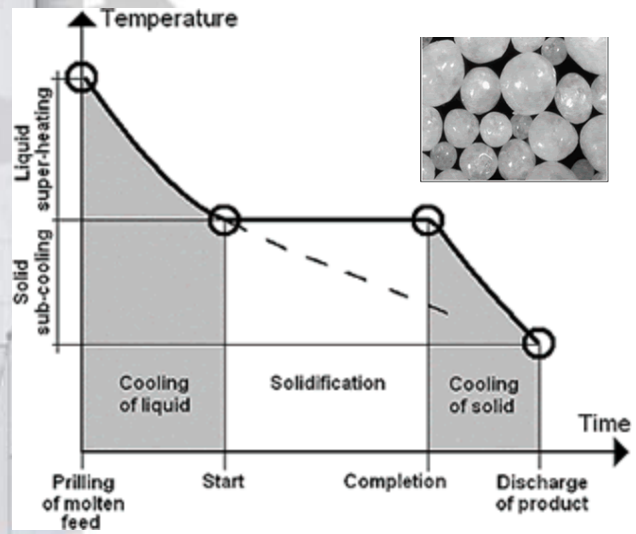
# Prilling

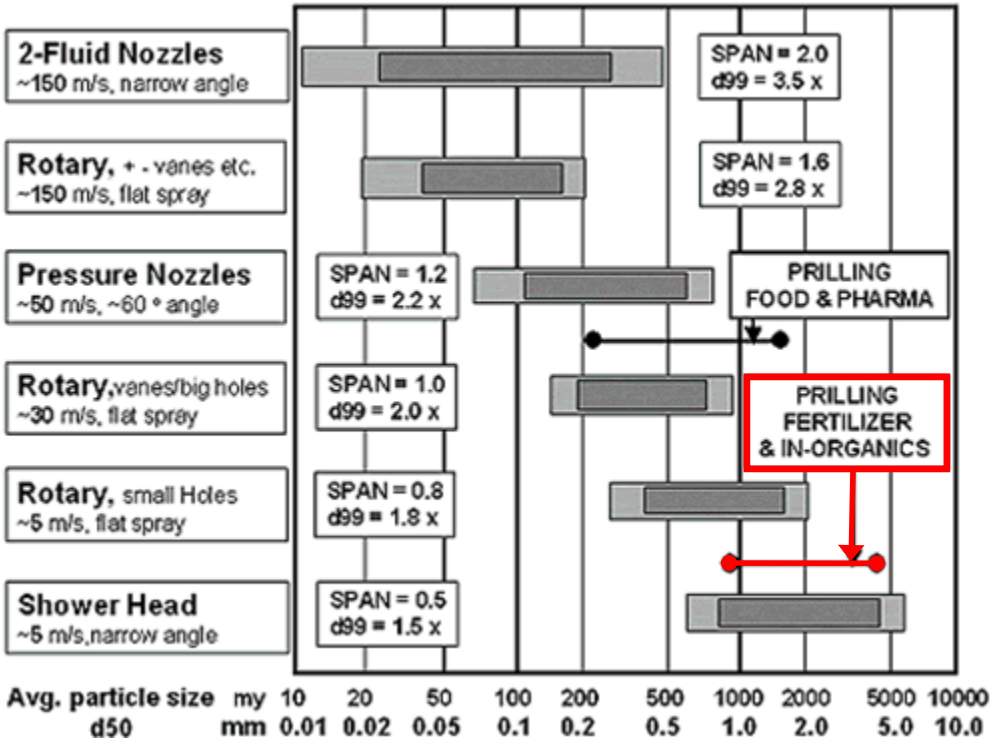


Prilling Wheel




Prilling = Multiple Core Encapsulation

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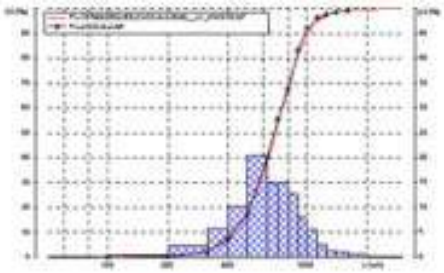
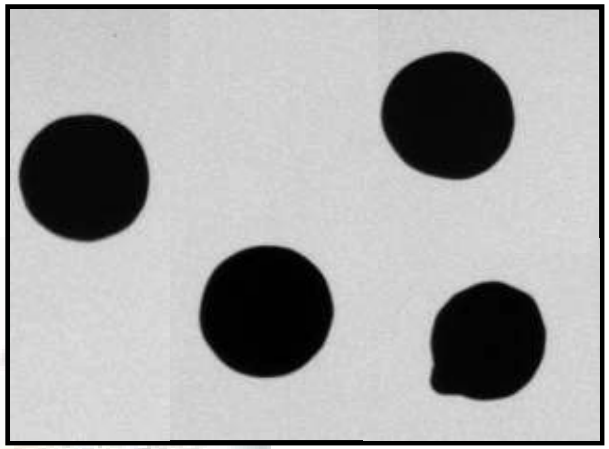
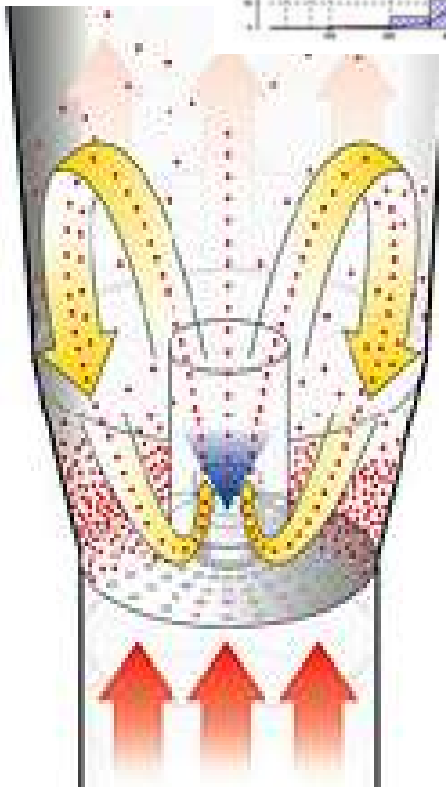
# Fluidized Bed Granulation

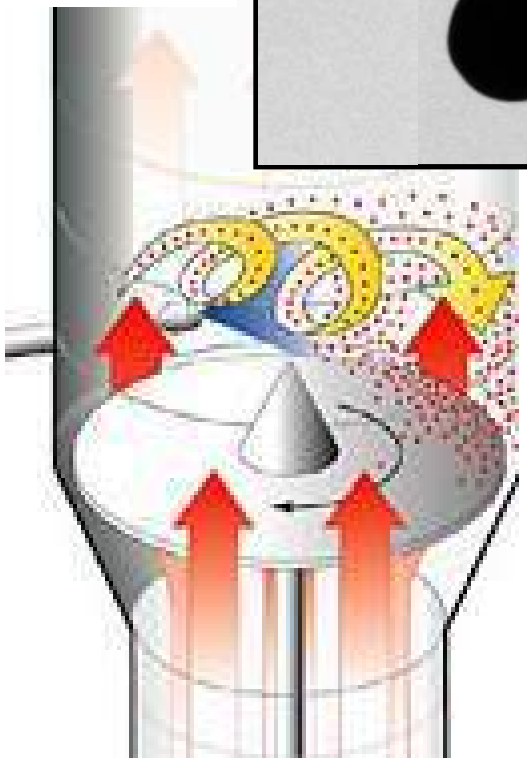




Top-Spray



Bottom-Spray



Tangential-Spray



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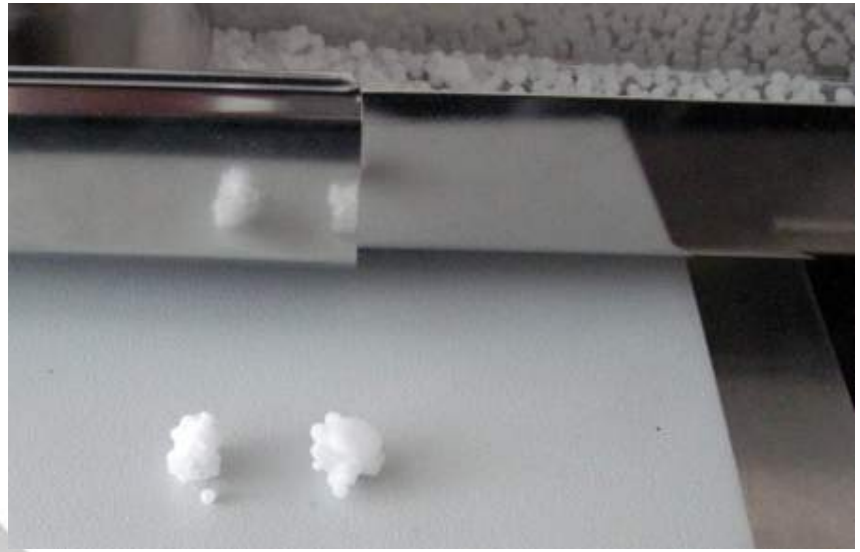
# Urea Agglomerates





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# CAMSIZER for Fertilizer




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# Previous
# Size Measurement System


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# Previous
# Roundness Measurement System



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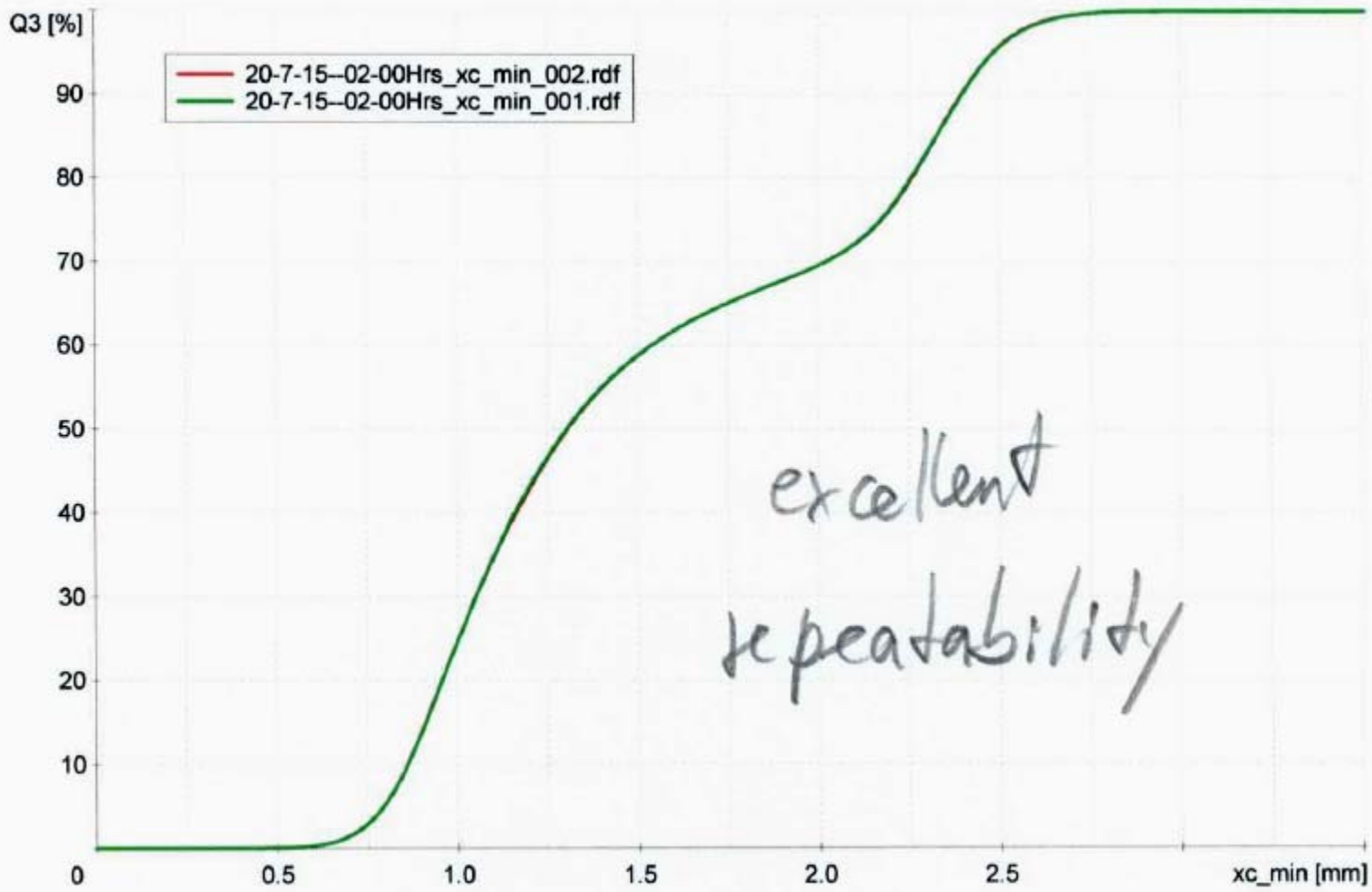
# CAMSIZER Test Fertilizer

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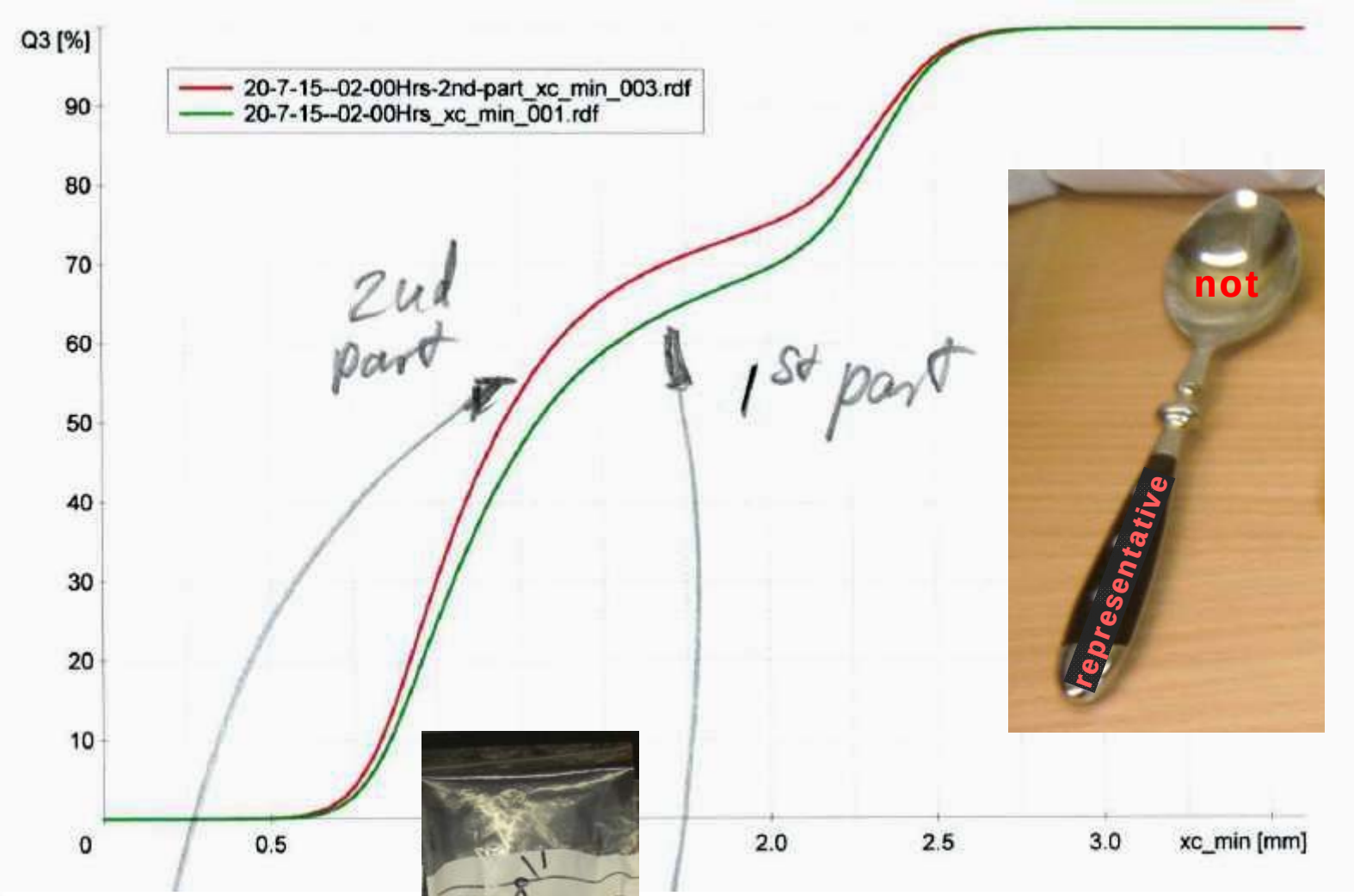
# Sample Taking Fertilizer







**with spoon from bag**

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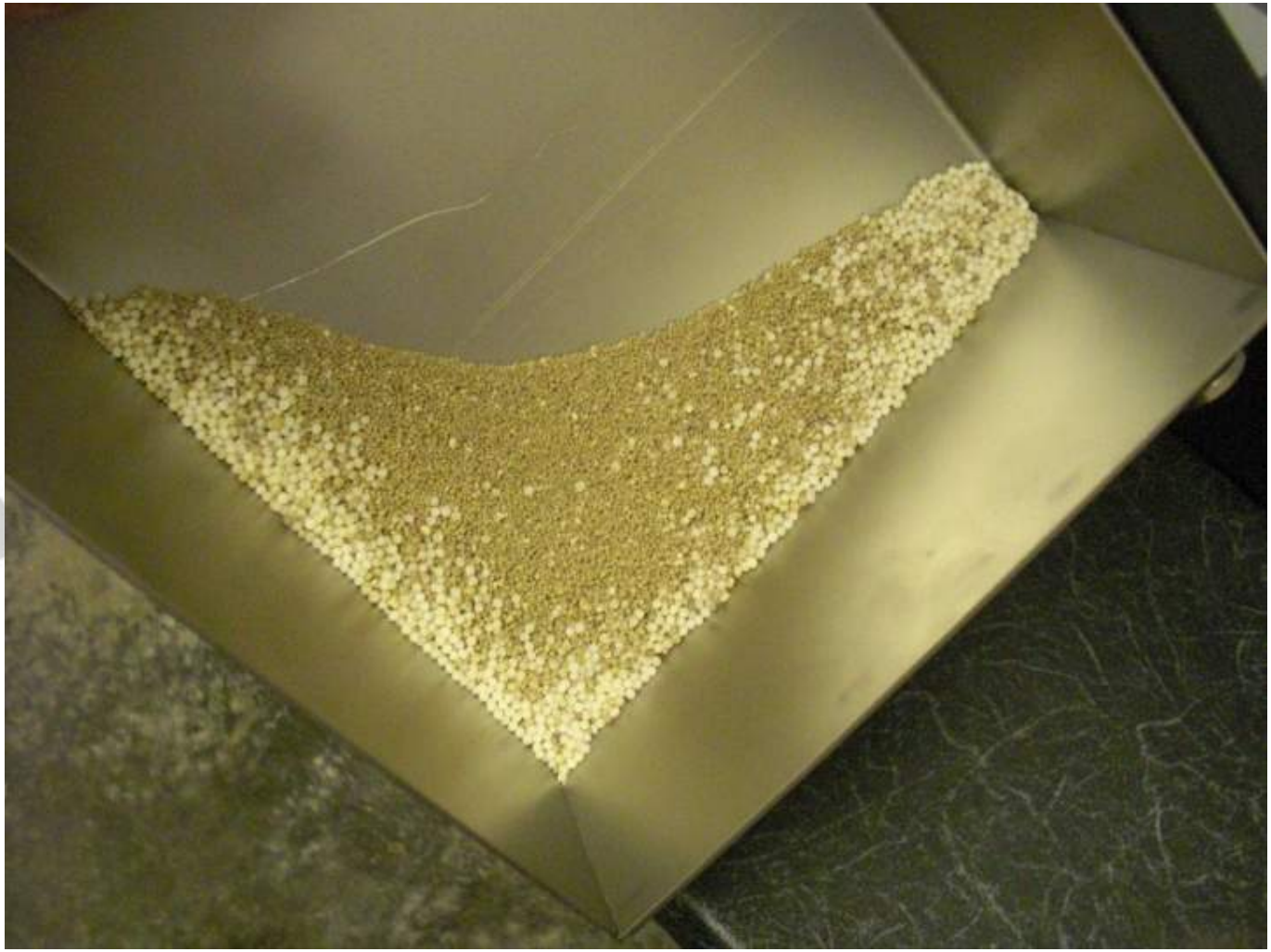
# Extreme Segregation because of Bimodal Size





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# Sample Splitting



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# Sample Splitting


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# Sample Splitting




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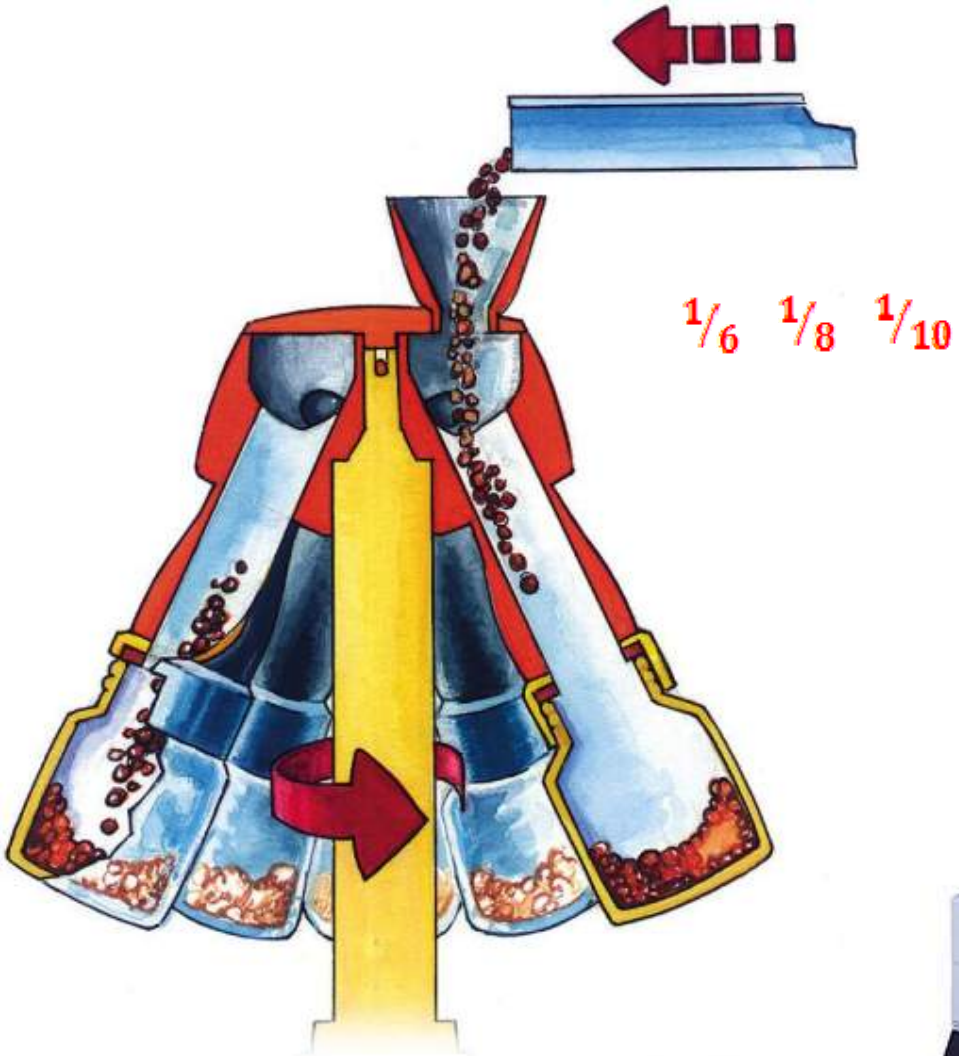
# Sample Splitting

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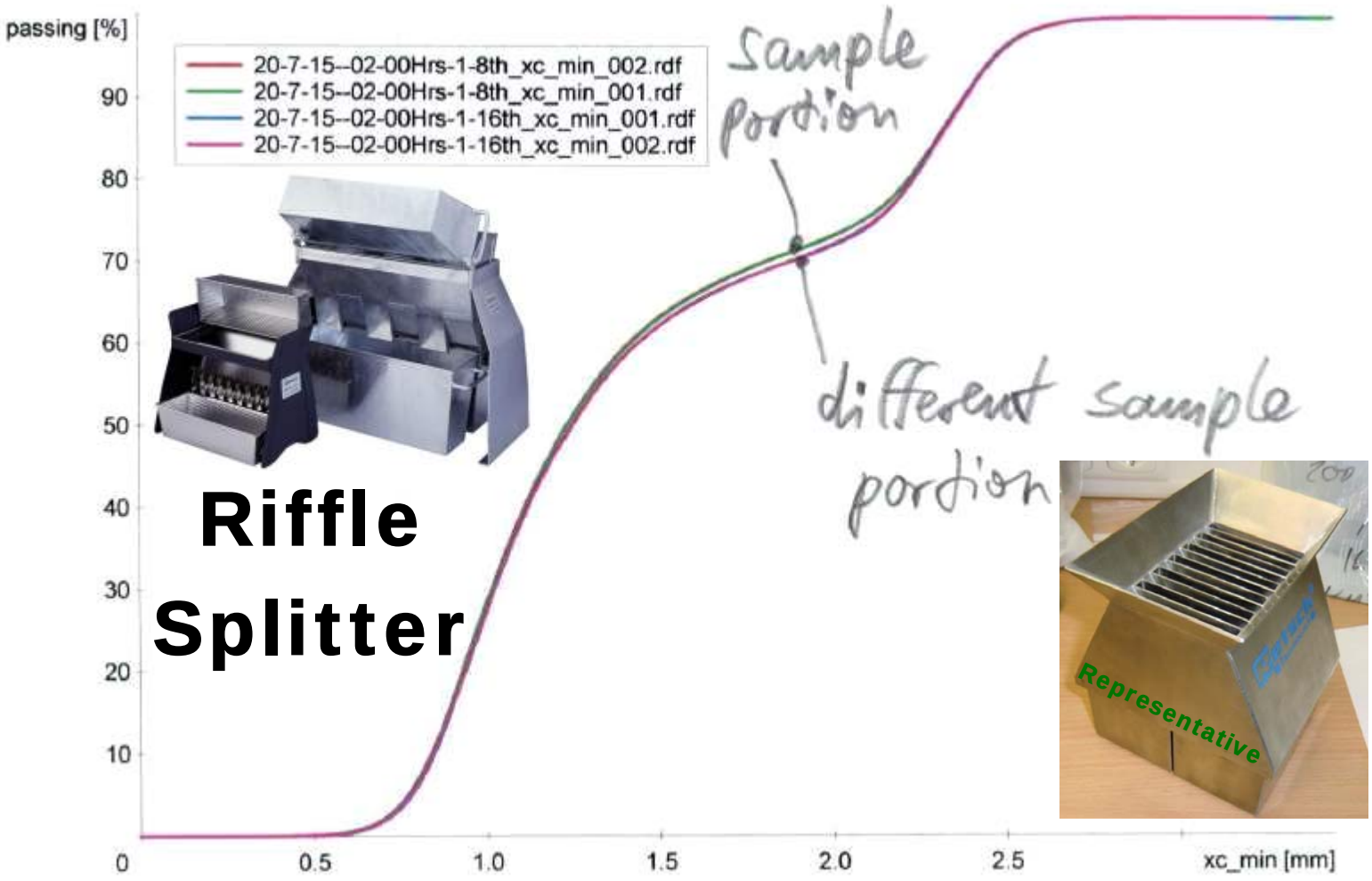
# Sample Splitting Fertilizer Mix



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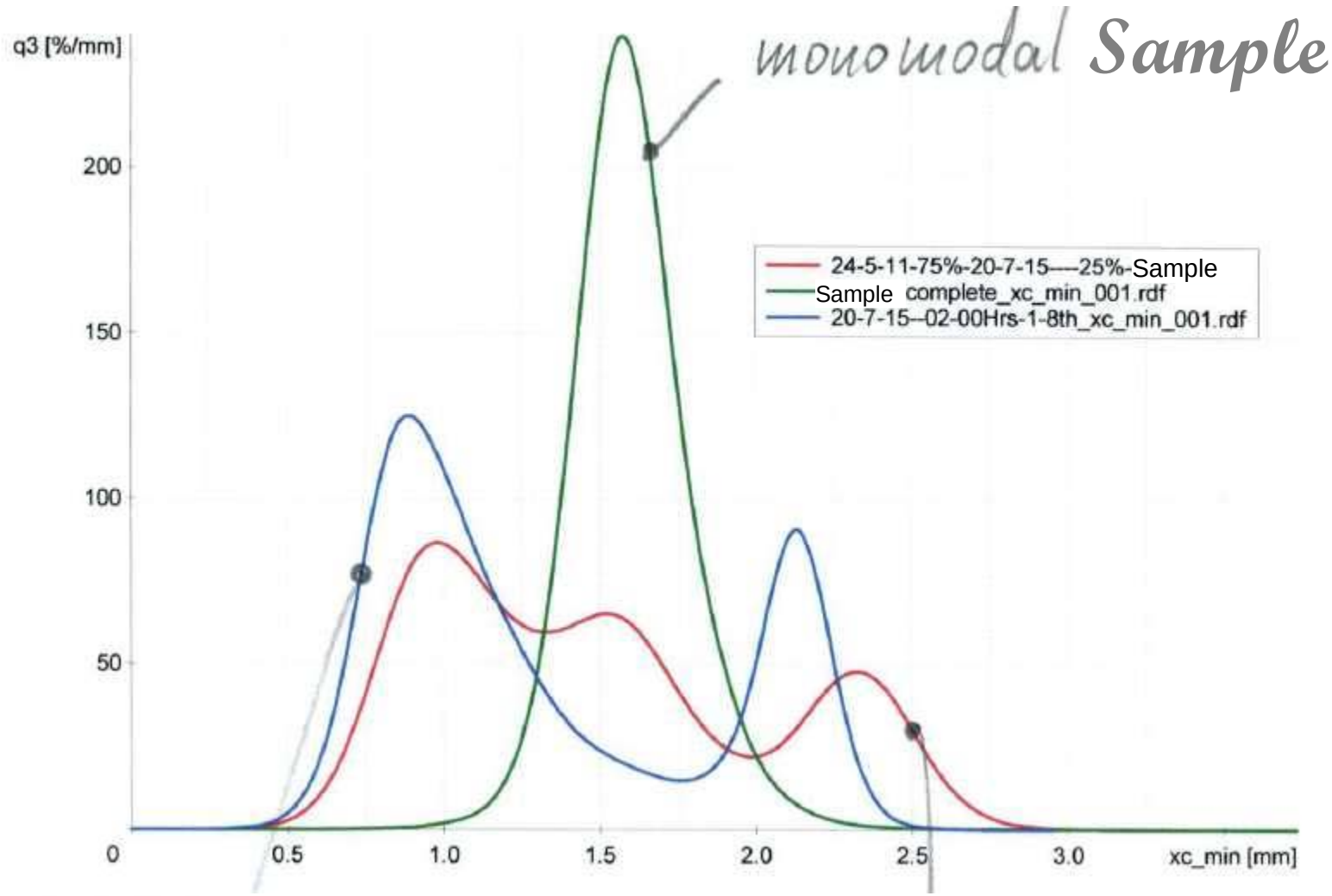
# Mixture of Fertilizers




**Blue** = bimodal sample

**Green** = monomodal Poly S

**Red** = 3-modal Mixture

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# 3-Size Mixture



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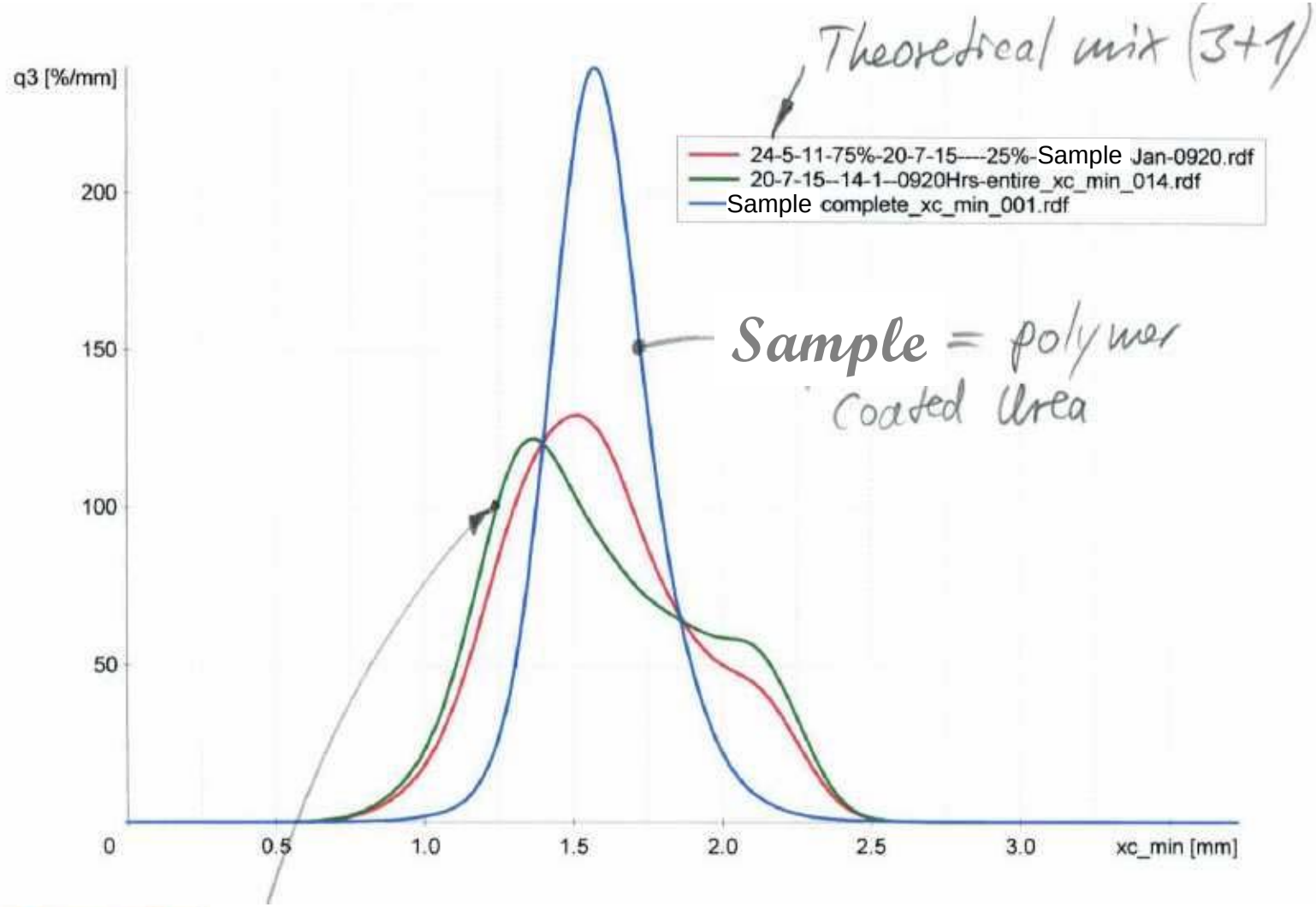
# Improved Product Fertilizer




**Blue** = monomodal Poly S

**Green** = "1.5"-modal Urea + Phosphate

**Red** = "1.5"- modal Mixture of 3 products

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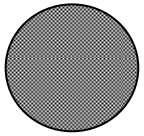
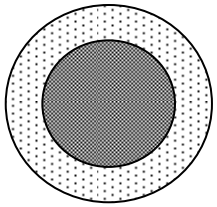
# Slow Release Fertilizer Particulates

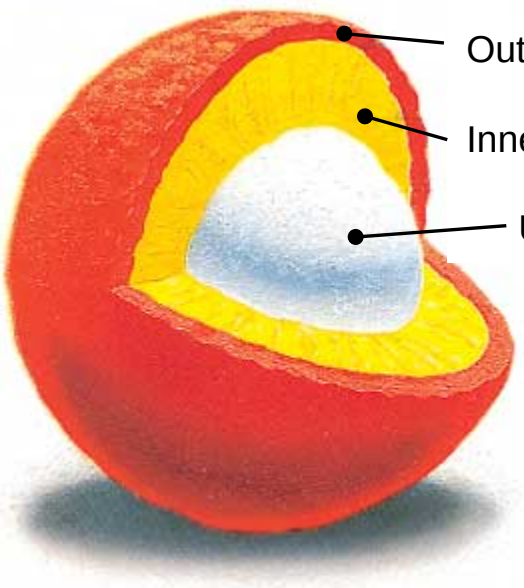
- Urea Core
- Sulfur Layer
- Polymer Coating

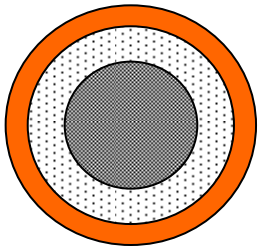


Urea Core



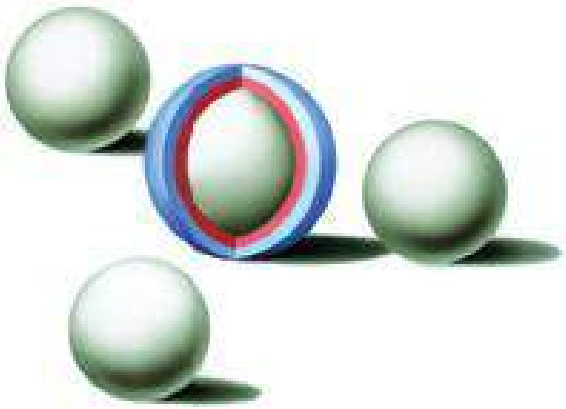
Urea Core
+ Sulfur layer



Urea Core
+ Sulfur layer
+ Polymer Coating

# Coating Layer Thickness



**Urea Pellets are a solid round form of nitrogen fertilizer. The individual pellets are almost spherical with diameters usually between 1000 µm and 3000 µm, sometimes 5000 µm.**

**Coatings can be measured with an accuracy of 2 µm layer coating thickness**

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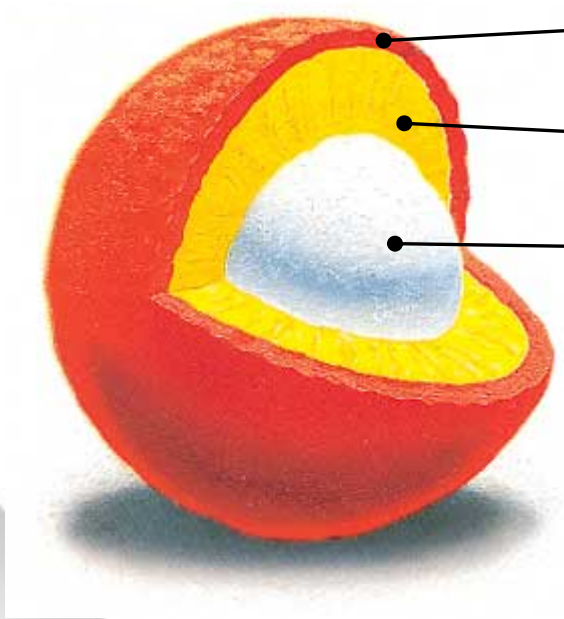
# Problems during Fluid Bed Processing

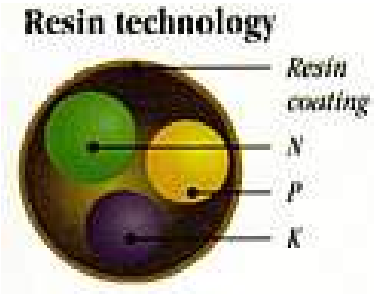


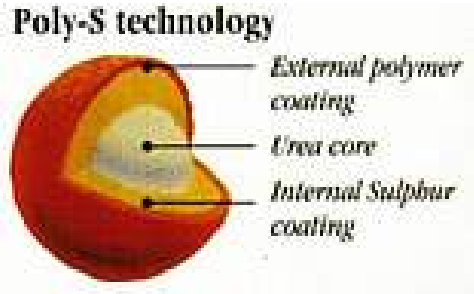
Outer External Polymer Coating

Inner Sulfur Coating

Urea Nutrient Core

Particles stick together during the coating and drying

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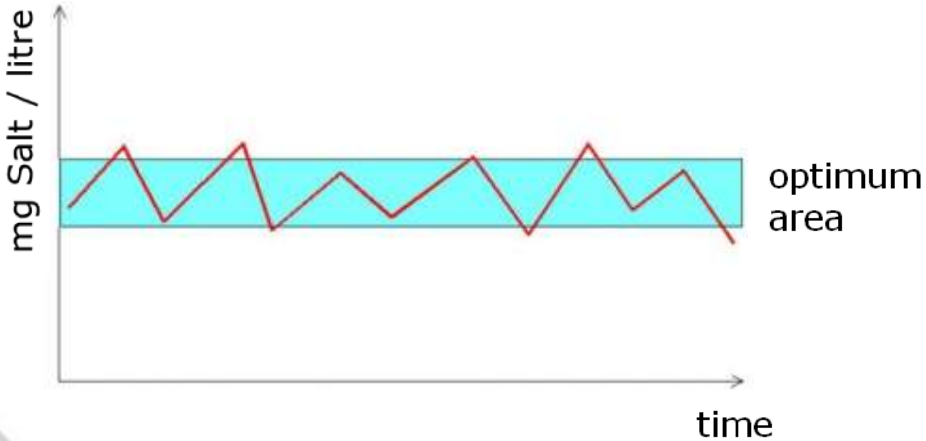
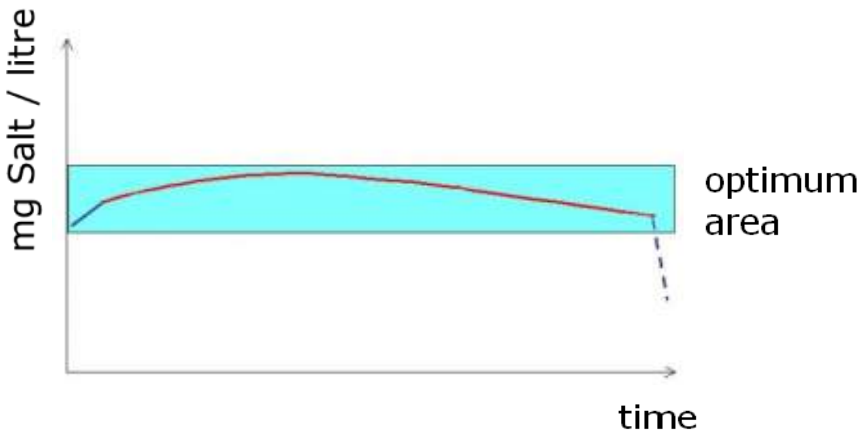
Time diagram of Salt Content during Interval Fertilizing
additional fluid fertilizing two times a week




**Type and amount of fertilizer is dependent on the soil content, the water quality, the type of plant, and the developing process of the specific plant,**

Time diagram of Salt Content after single Depot Fertilizing
basic fertilizing of standard soil




**30 % – 60 %**
**of nutrition application can be solved with depot fertilizer**



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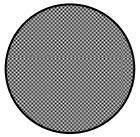
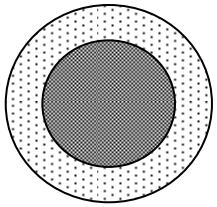
# Slow Release Fertilizer Particulates

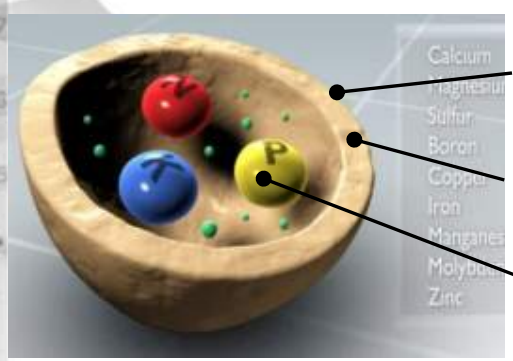
- NPK + Traces Core
- Polymer Resin Layer
- "Glue" Layer



NPK+
Core



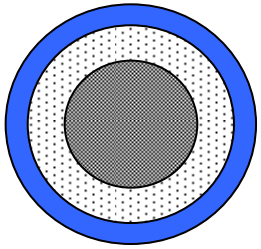
NPK+ Core
+ Polymer Resin Layer

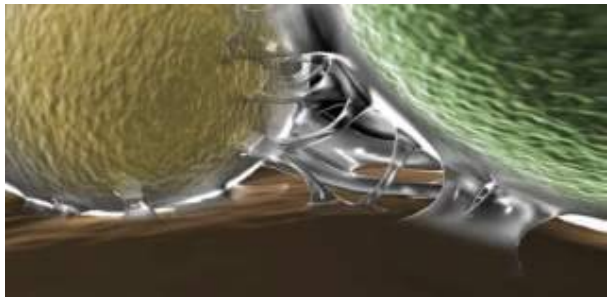



**Outer „Glue" Coating**

**Inner Polymer Resin Coating**

**NPK Nutrient Core**





NPK+ Core
+ Polymer Resin Layer
+ Glue Coating Layer

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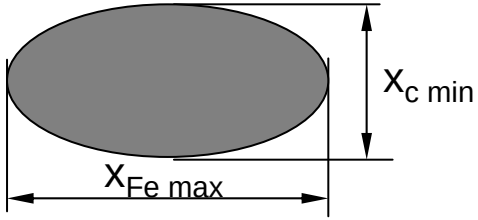
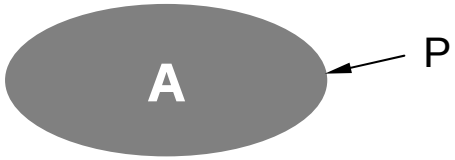
# Particle Shape

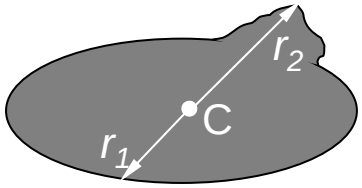
- **Breadth- / Length-ratio**



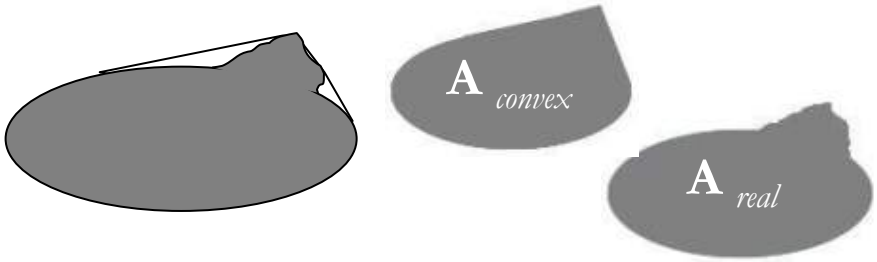

- **Roundness**




- **Symmetry**




- **Convexity**



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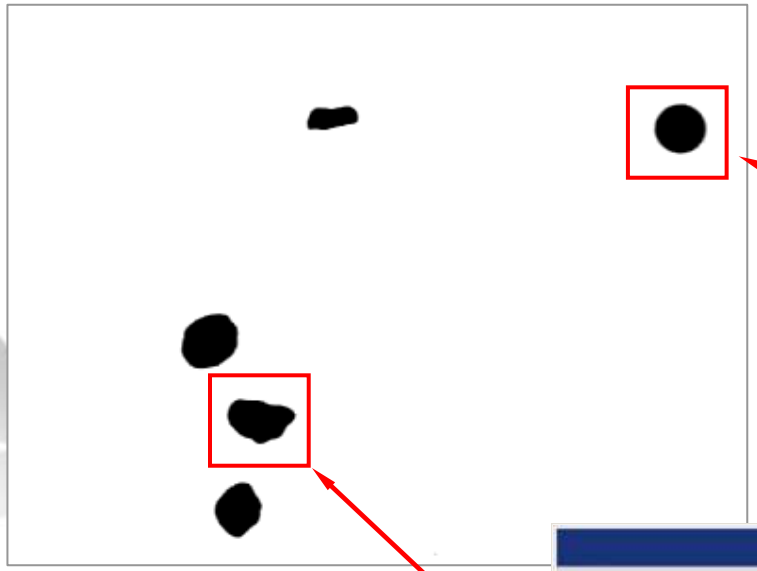
# Optical Process Control

## analysis for size and shape



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

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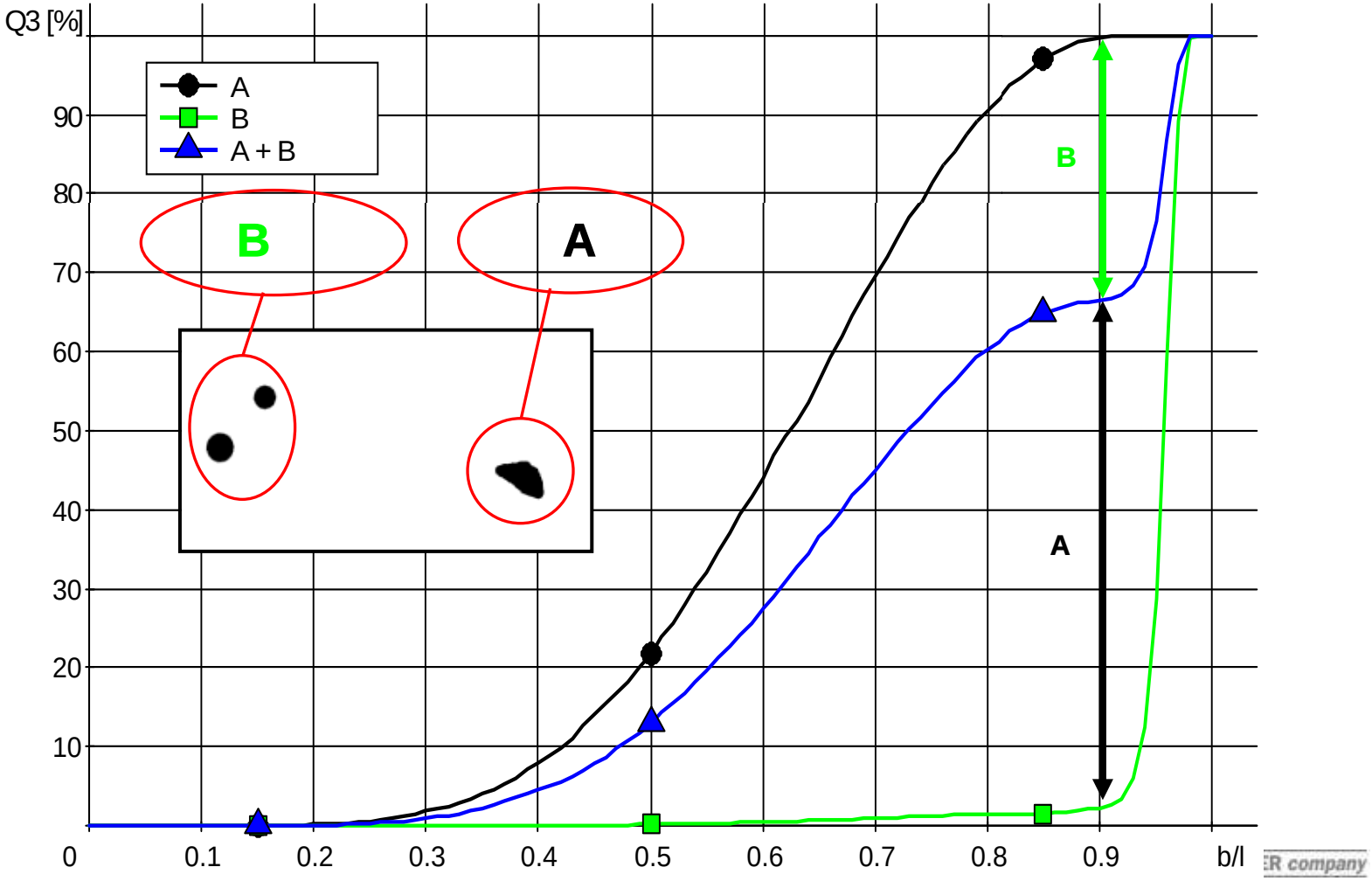
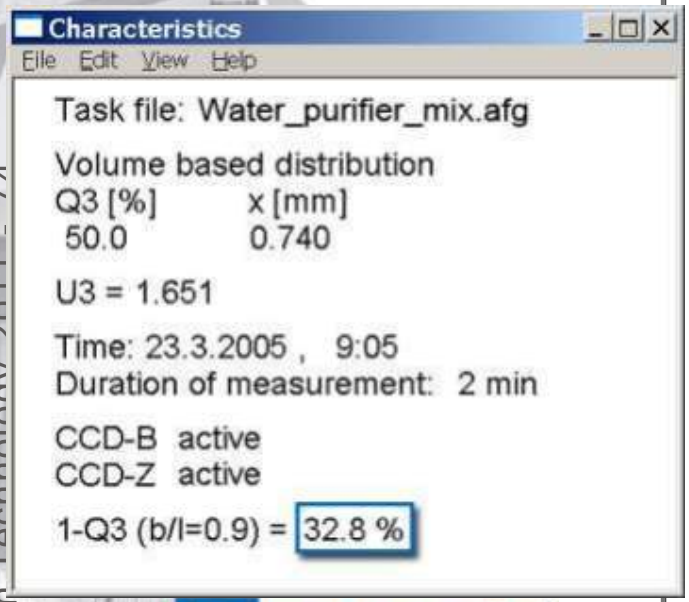
# Urea with Granular Potash

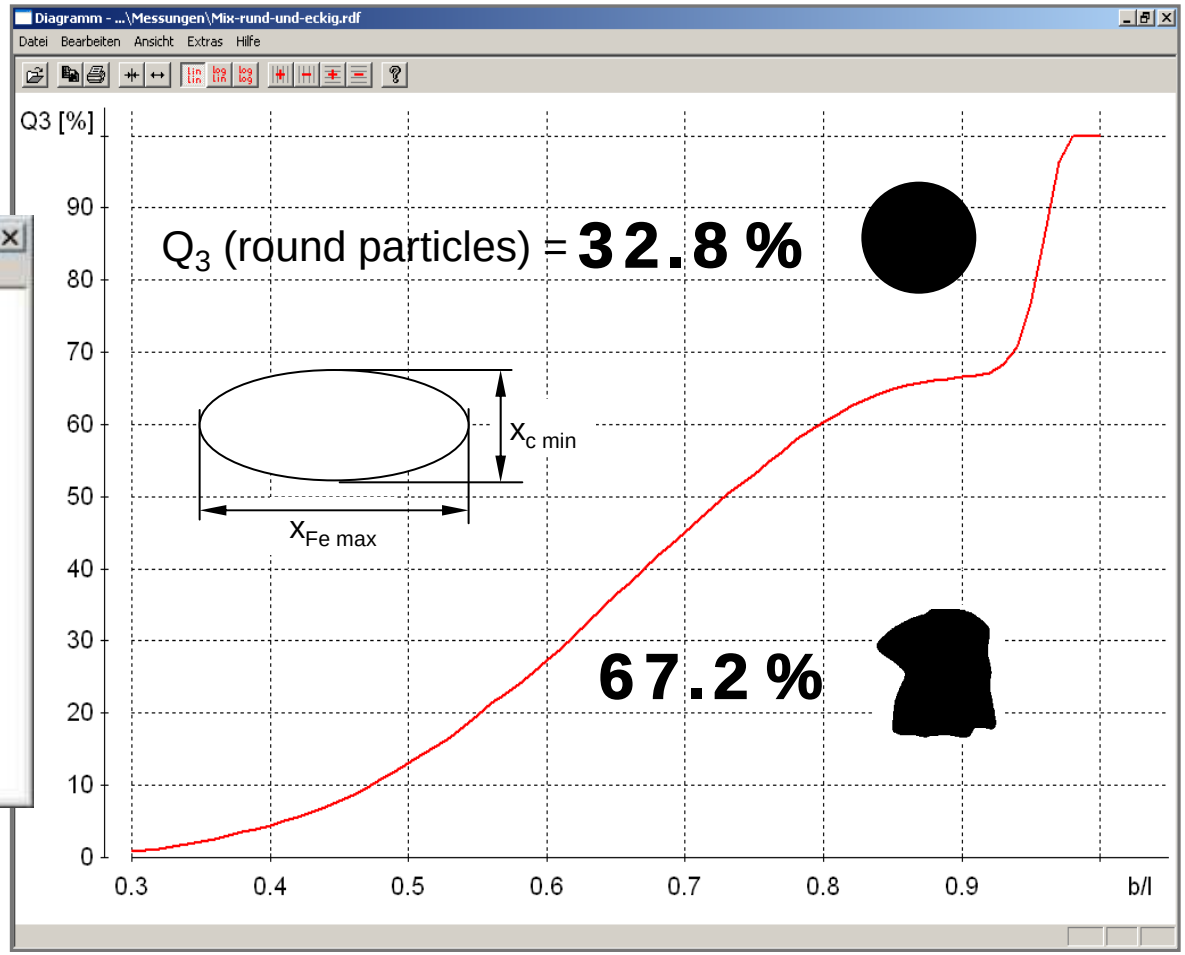
## Amounts of urea beads and pressed / crushed potash granules in a mixture



# Urea with Granular Potash

## CAMSIZER can find the mixing ratio of components of Urea Beads and Granular Potash

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# Traditional Measurement Pellets

- Sieve analysis → 1.0 – 3.0 mm
- Vibrating plate and weighting (amount of broken particles)

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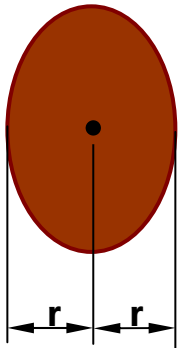
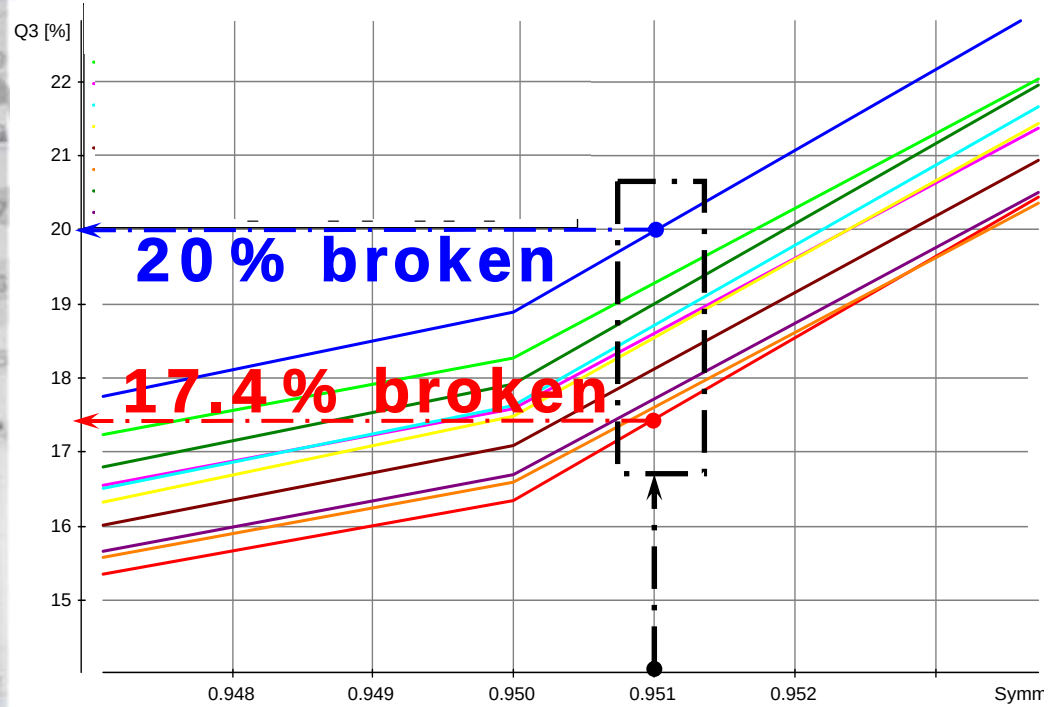
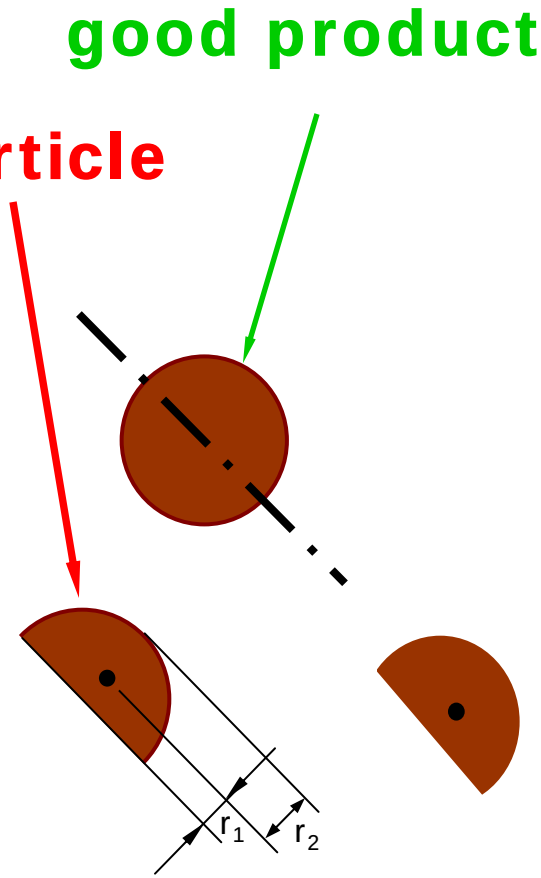
# CAMSIZER – Advantages Measuring Broken Beads




$$\text{Symmetry} \sim \min\left(\frac{r_1}{r_2}\right)$$

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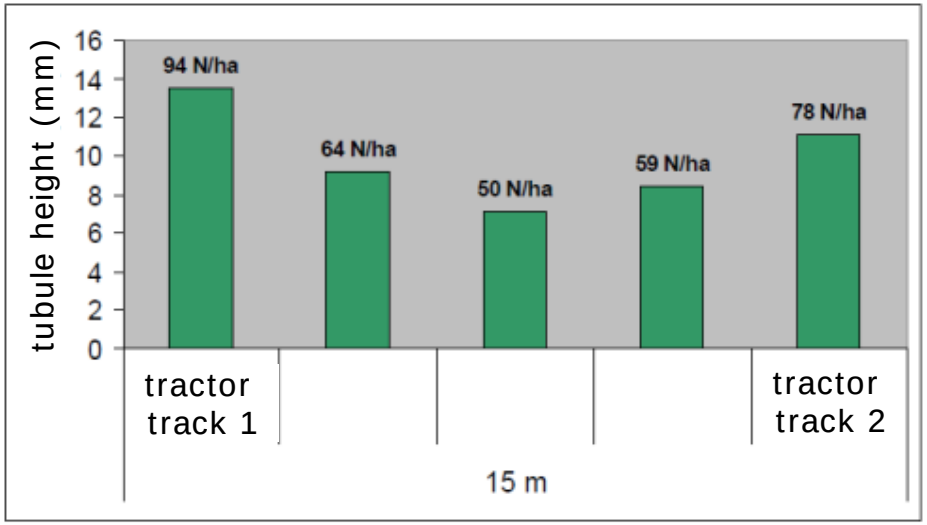
# Spreading Fertilizer Particulates

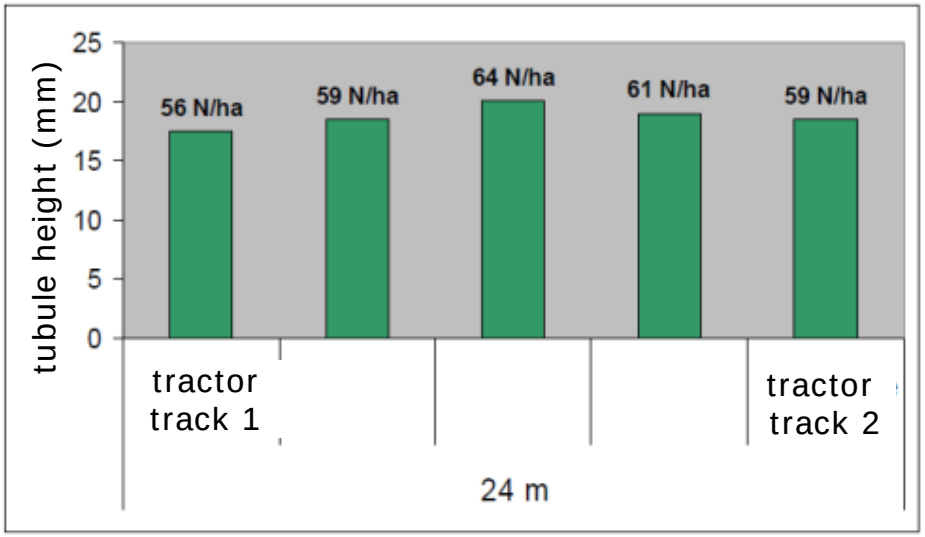


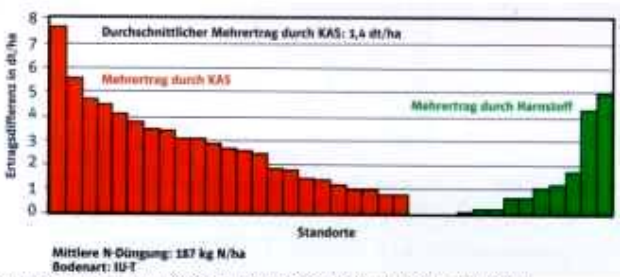
# Spreading Fertilizer Particulates

**Larger pellets can be thrown further than smaller pellets**

**The exact size is important to get controlled spreading conditions**




Grafik 1: Mehrerträge von KAS bzw. Harnstoff in 36 Versuchen der deutschen Offizialberatung, 1982 – 2000

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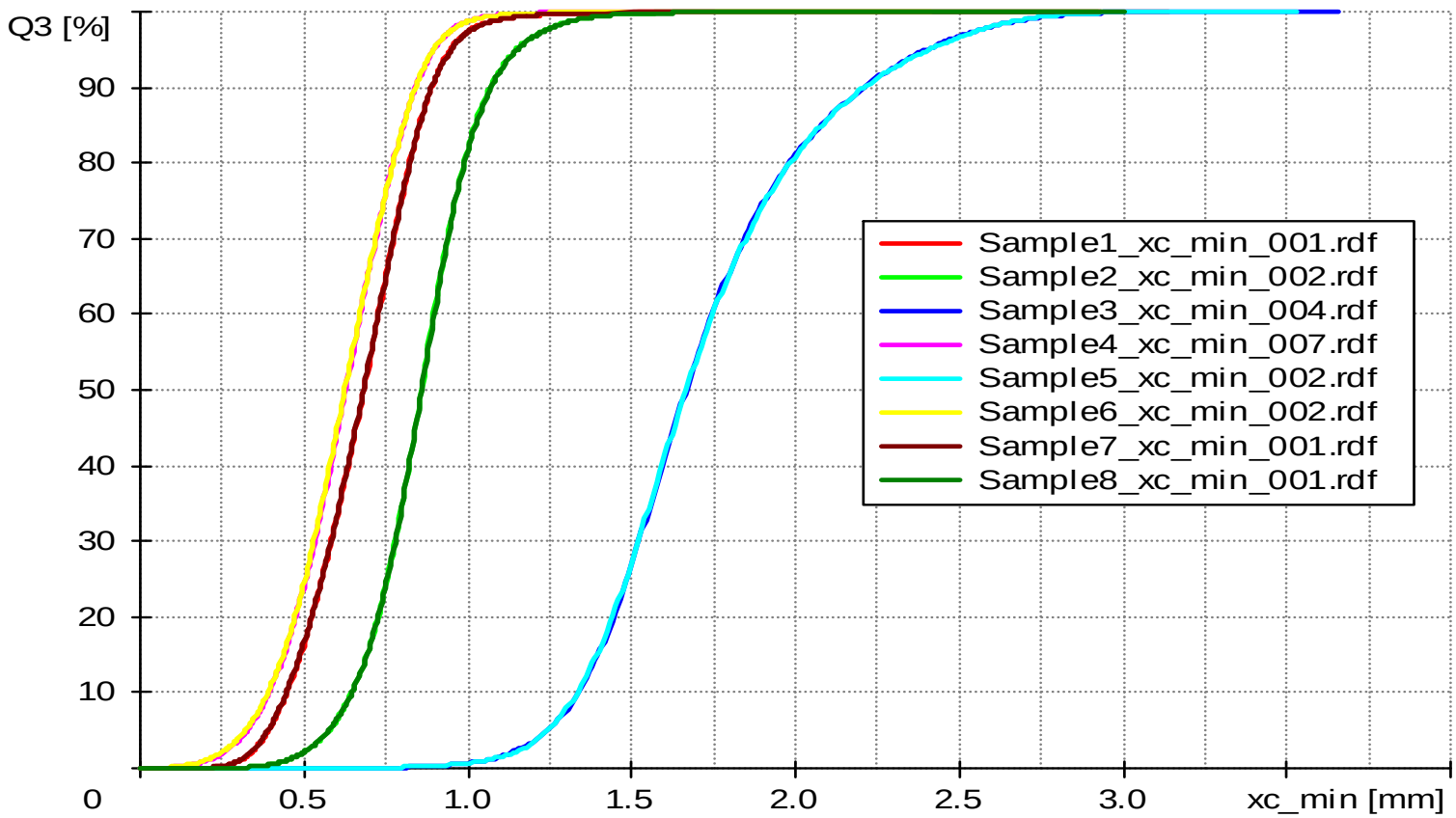
# Calcium Phosphate




**Samples 1-8, following samples show identical results (split samples ?)**

**1=7, 3=5, 2=8, 4=6**

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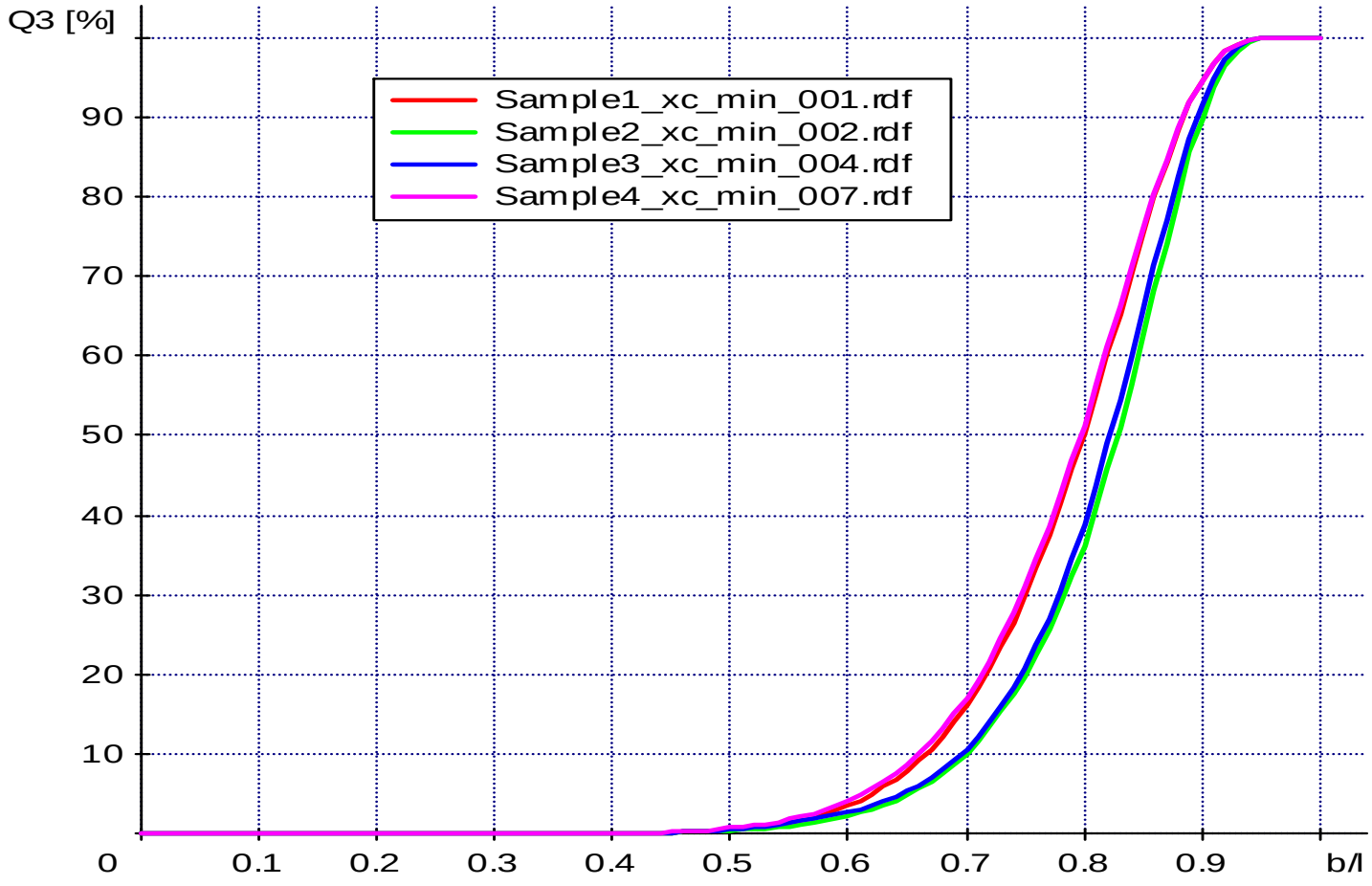
# Calcium Phosphate




**Samples 1-4 b/l = aspec ratio, 2 groups "1+4" and "2+3"**



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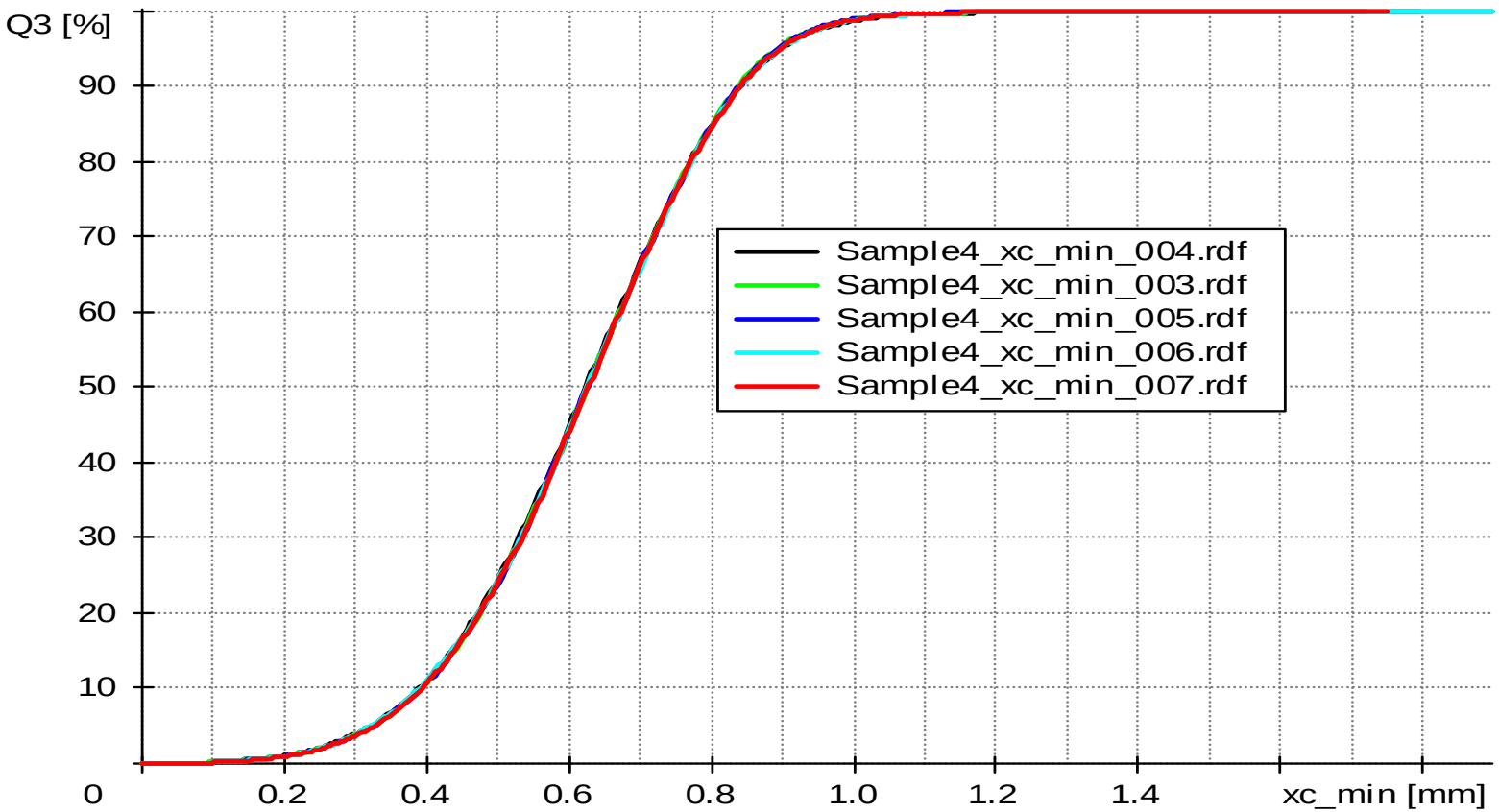
# Calcium Phosphate




**Repeatability: 5 consecutive measurements**

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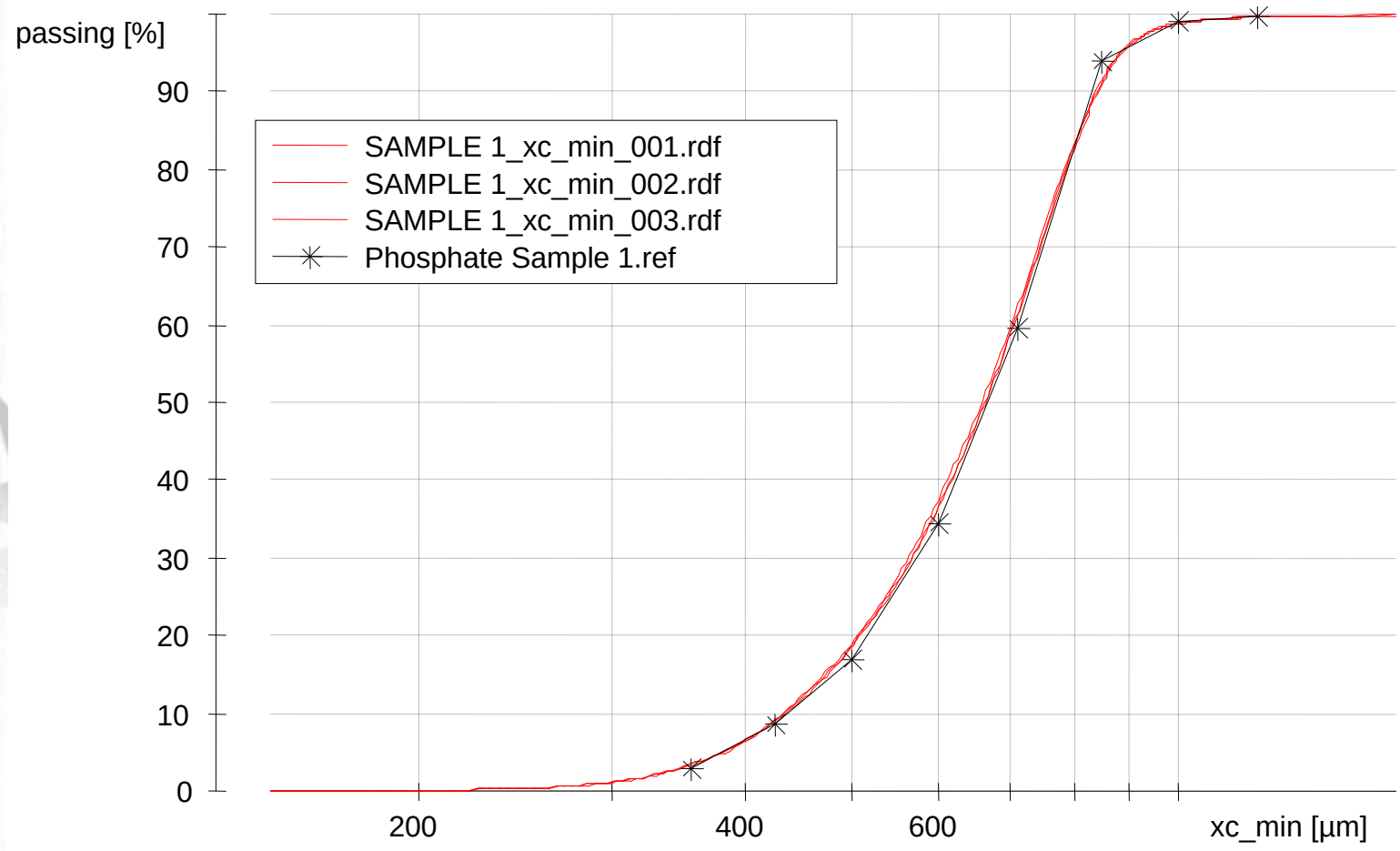
# Sieve Correlation Calcium Phosphate




**CAMSIZER sieve correlation using Elementary Fitting one single sieve class and entire sample**



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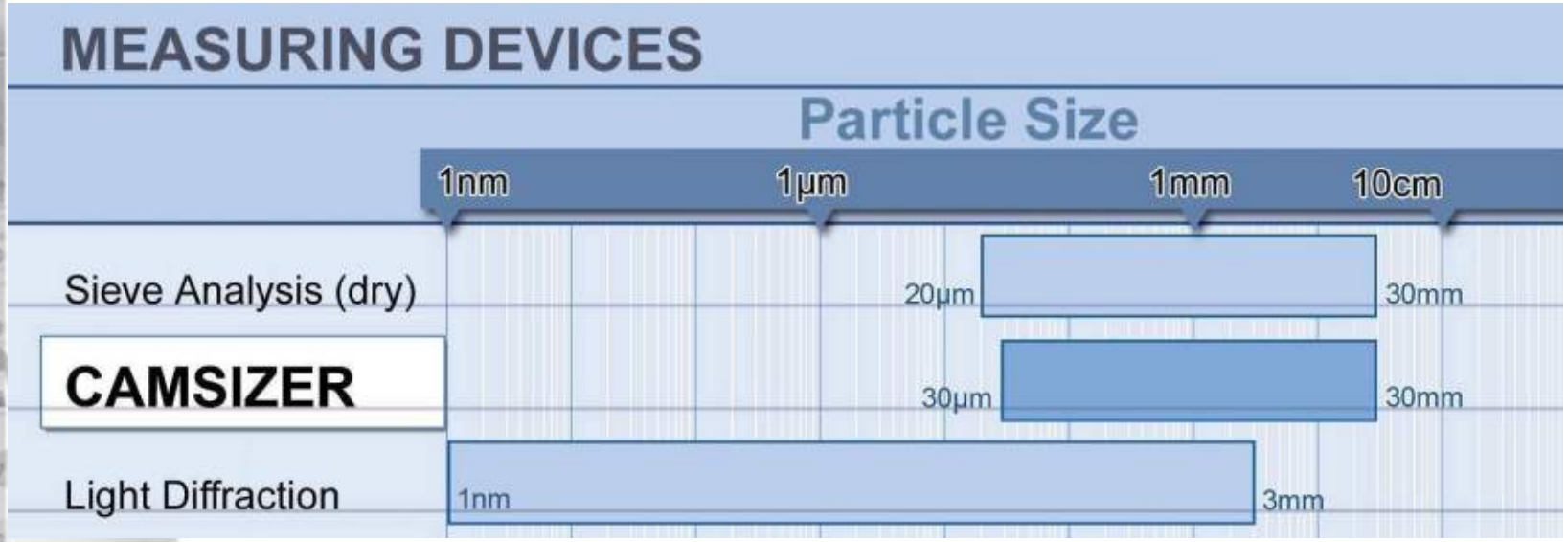
# Competing Measuring Methods





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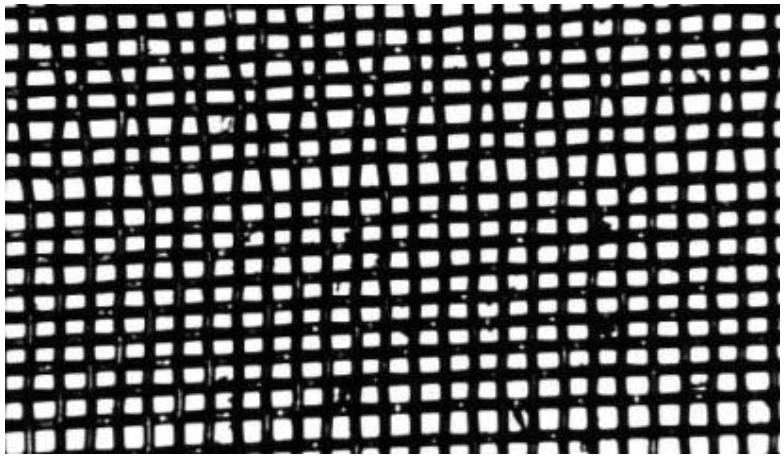
# Comparison of Methods: Sieving

## Advantages

- robust and industrial-suited
- easy handling
- references available from user



Worn out sieves

## Disadvantages

- high amount of time and work
- low resolution,
  small number of investigatable classes
- limited sample amount
- no shape analysis possible

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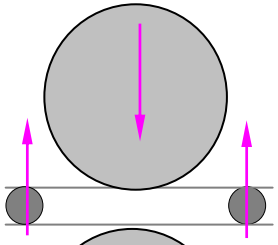
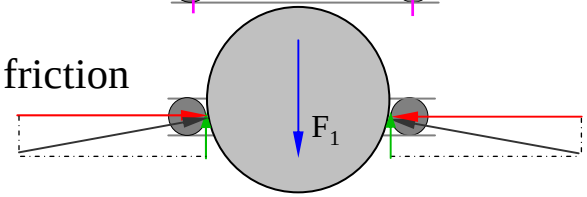
# Sieving problems

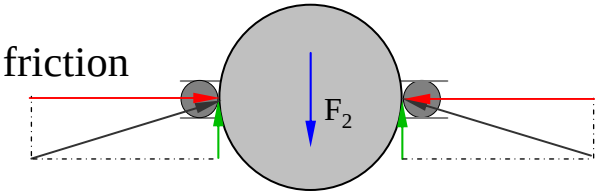
1. Move

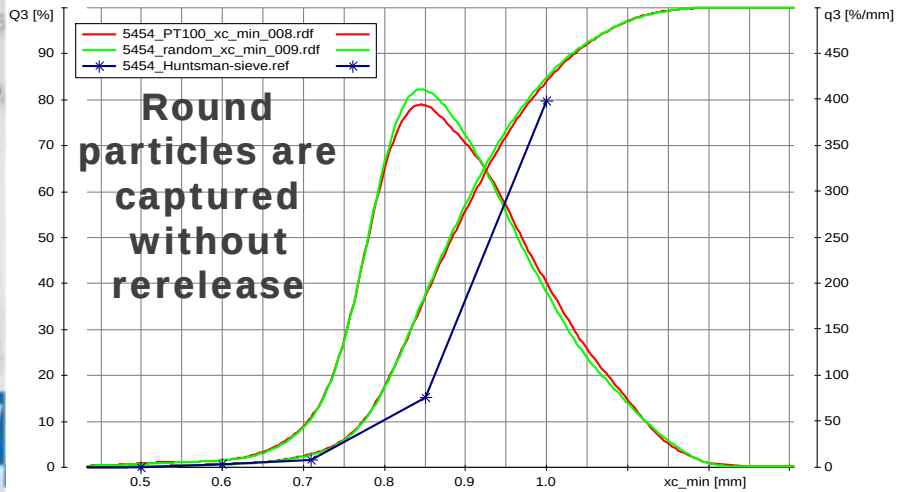


2. Sliding friction

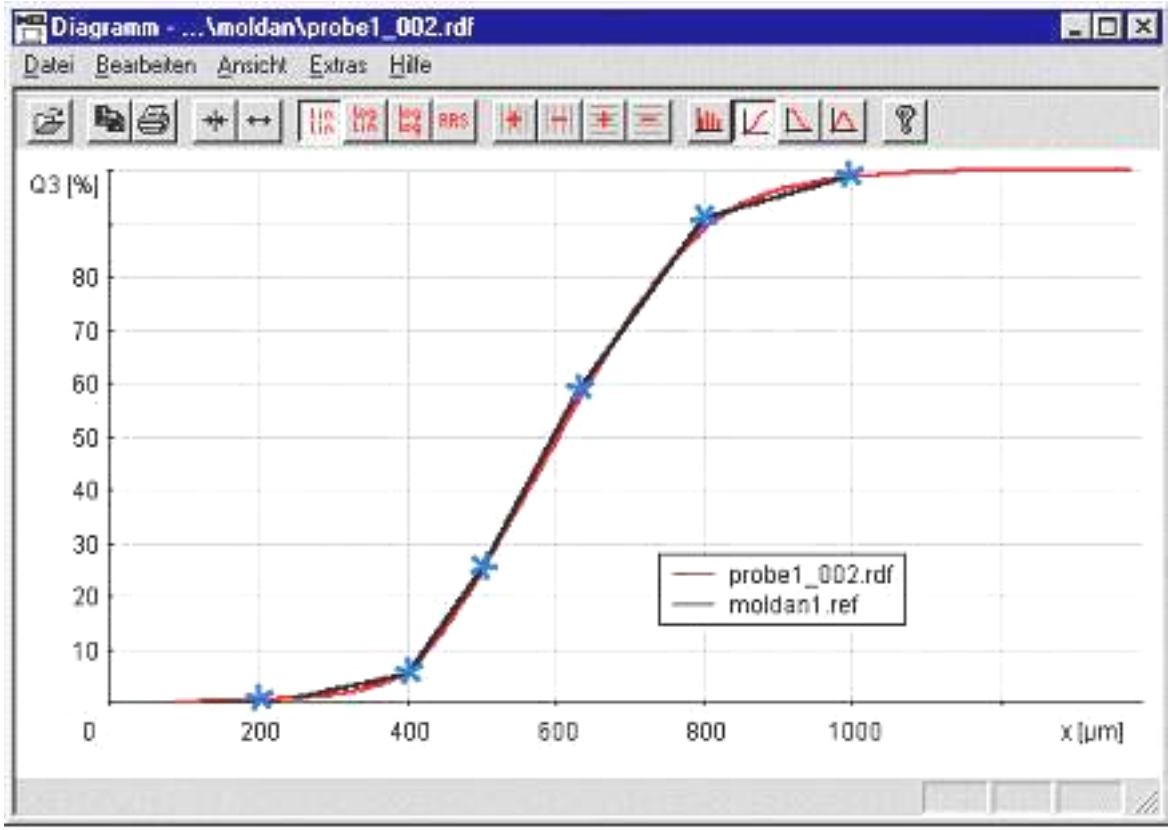



3. Static friction



# Fitting of CAMSIZER result to Sieving




**fitted result**

CAMSIZER-measurement **x** (**red**)
to sieving ***** (**blue**)



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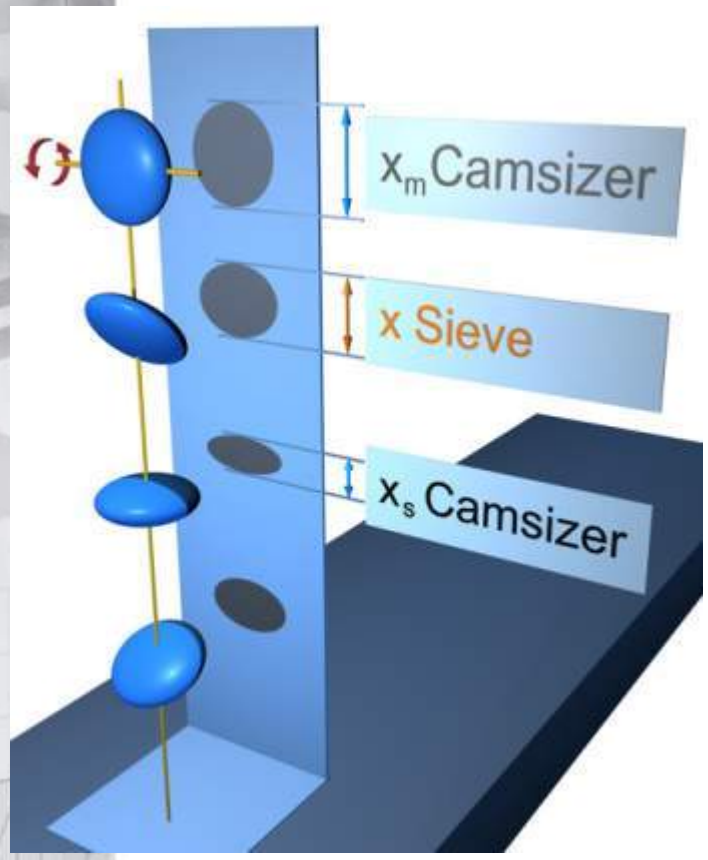
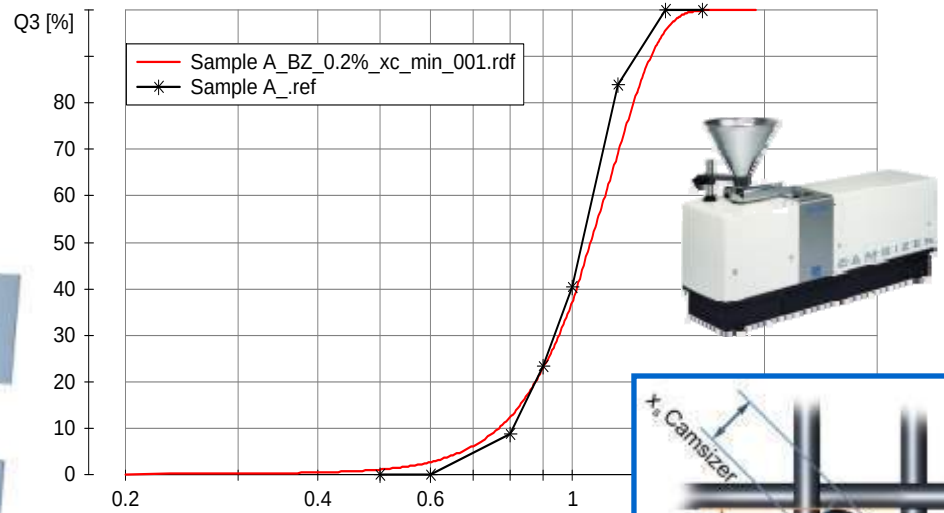
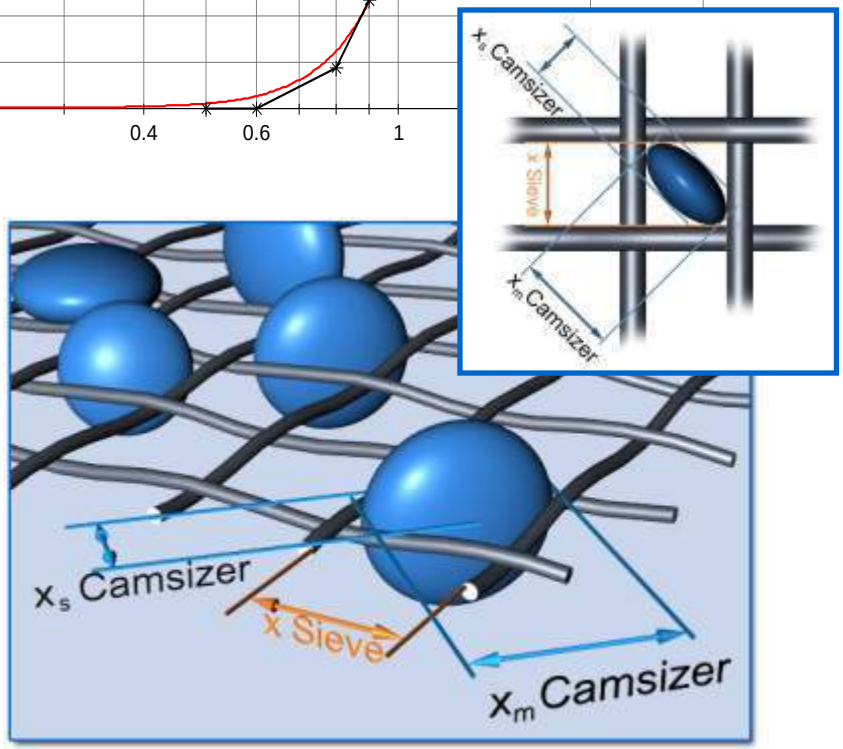
# Digitale Imaging ⇔ Sieving



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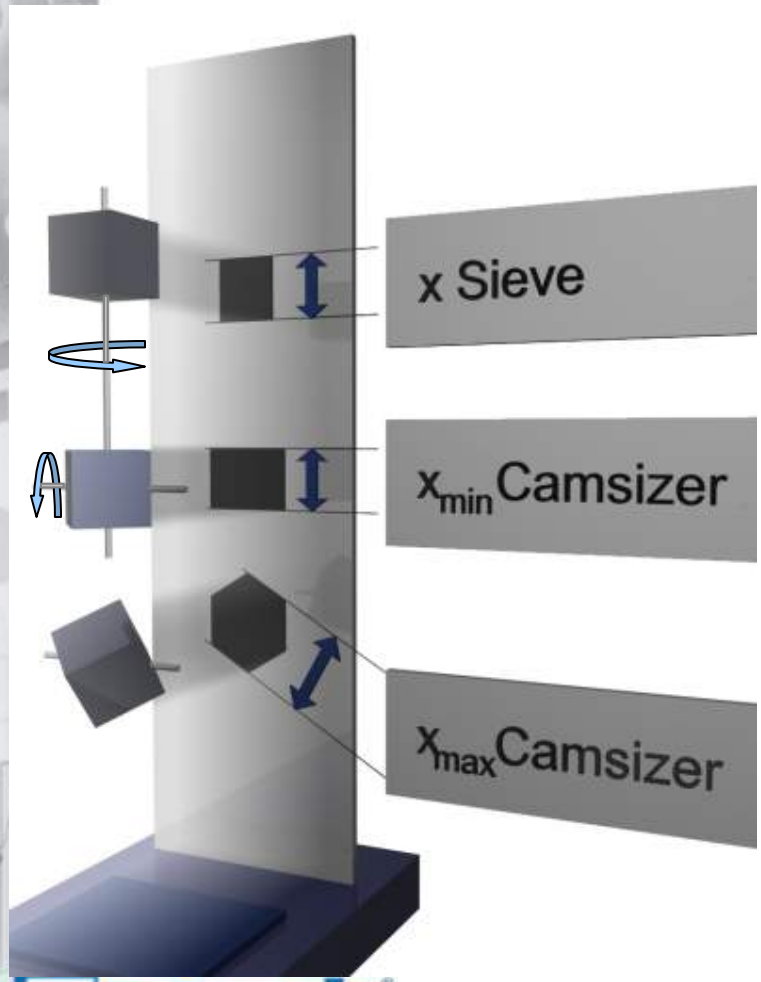
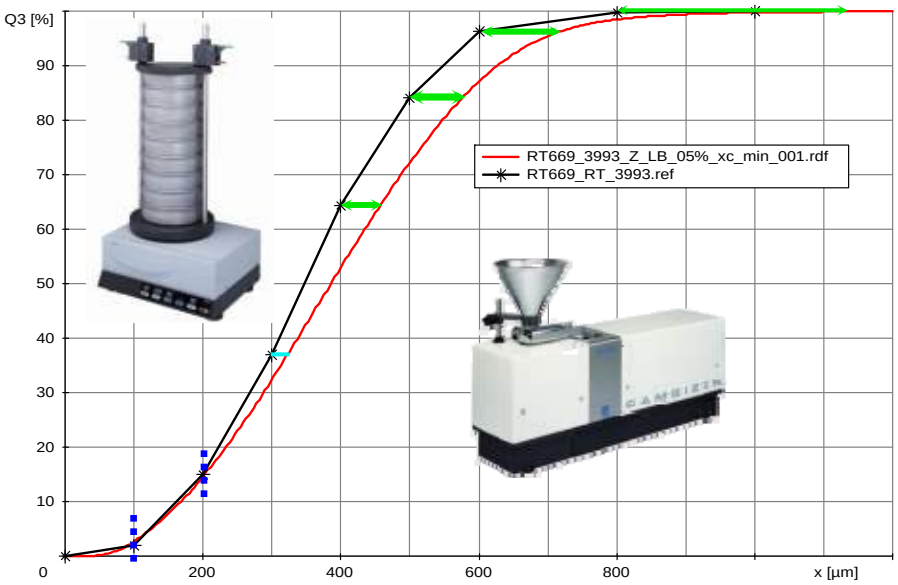
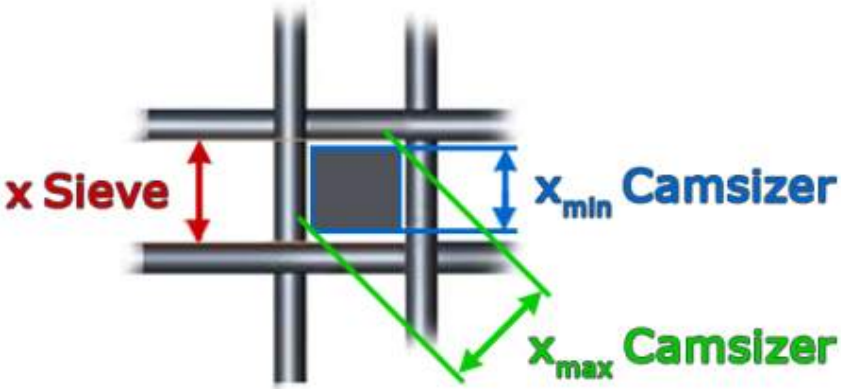
# Digital Imaging ⇔ Sieving
# Cubes / Angular Particles

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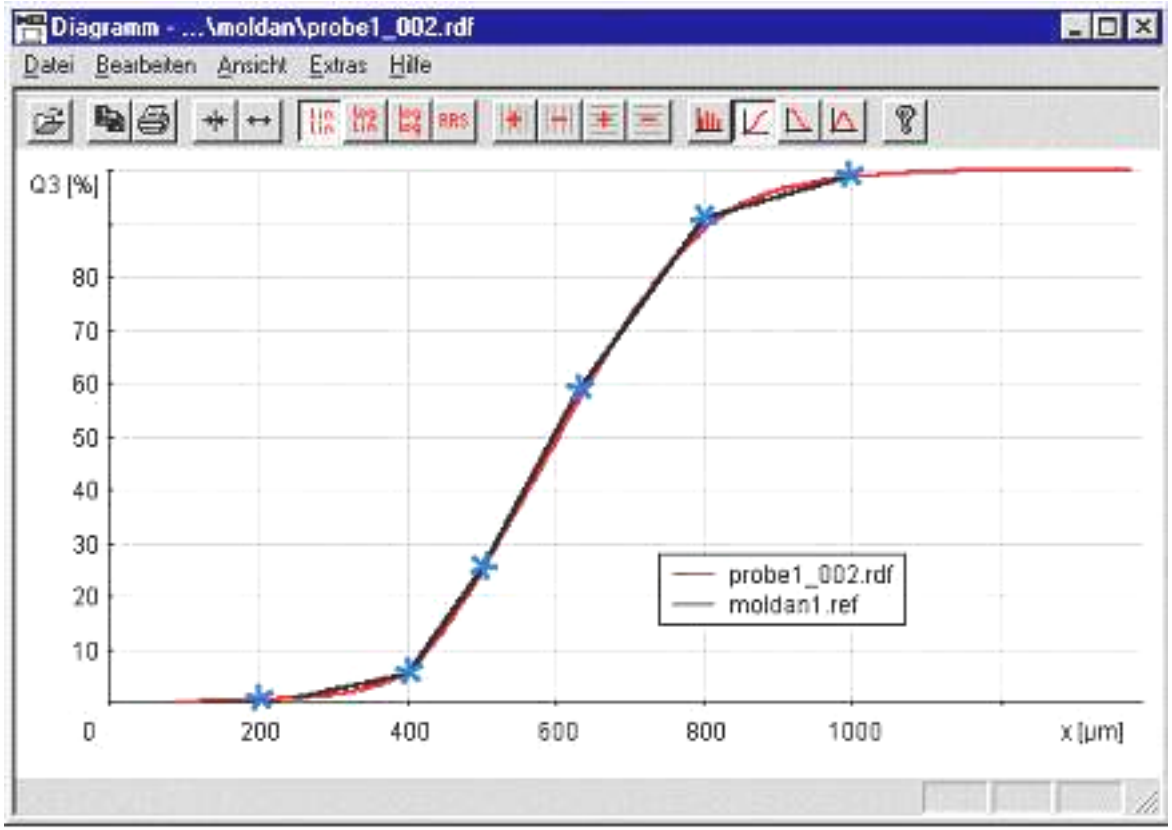
# Fitting of CAMSIZER result to Sieving




**fitted result**

CAMSIZER-measurement **x** (**red**)
to sieving ***** (**blue**)



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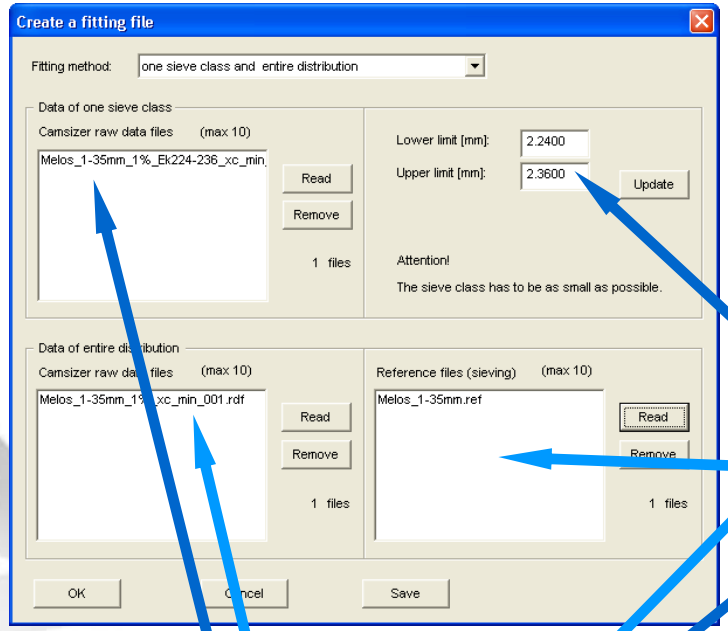
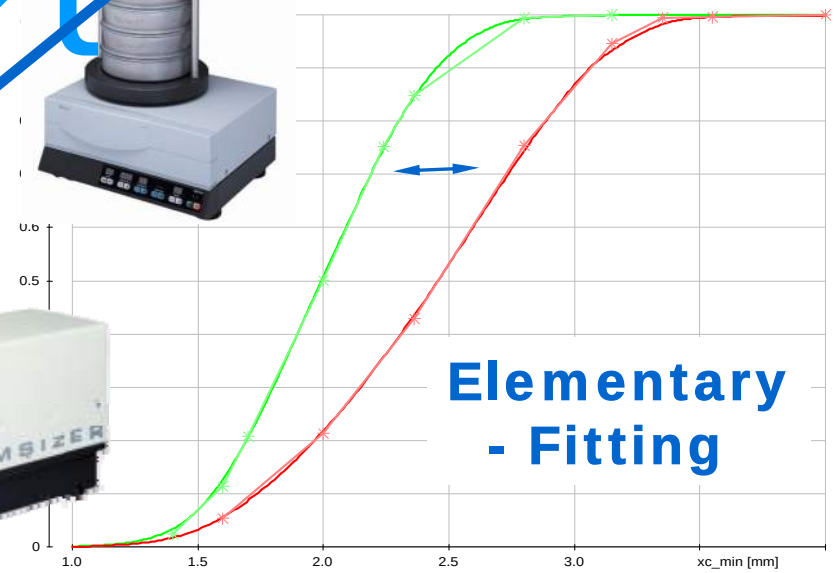
# Digital Imaging ⇔ Sieving




New elementary fitting with **single (narrow) sieve class** and **entire distribution**


**Elementary - Fitting**

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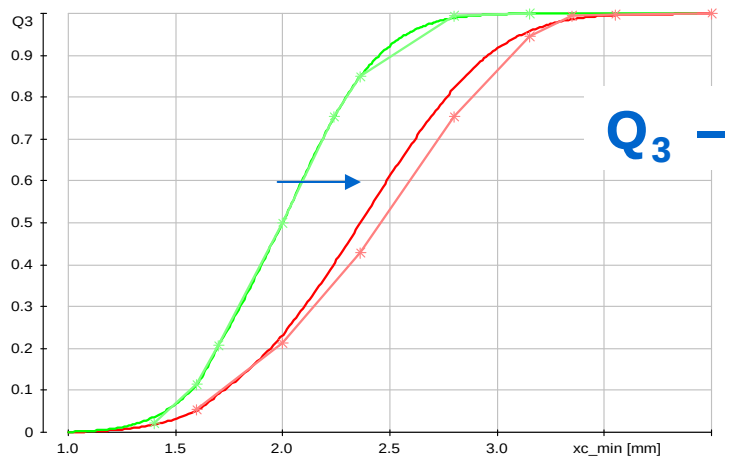
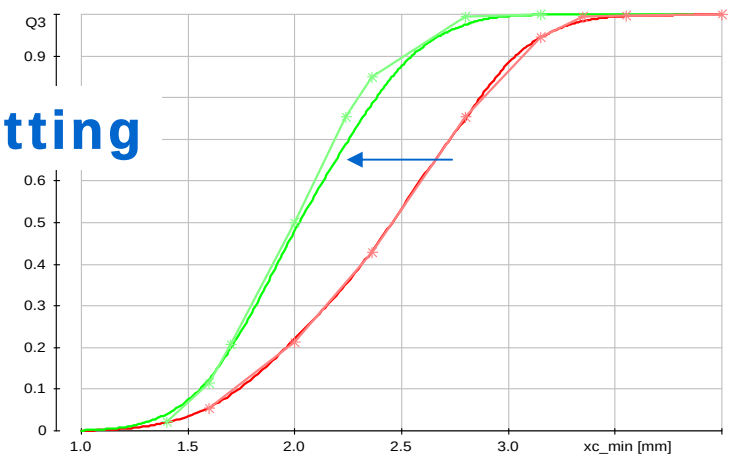
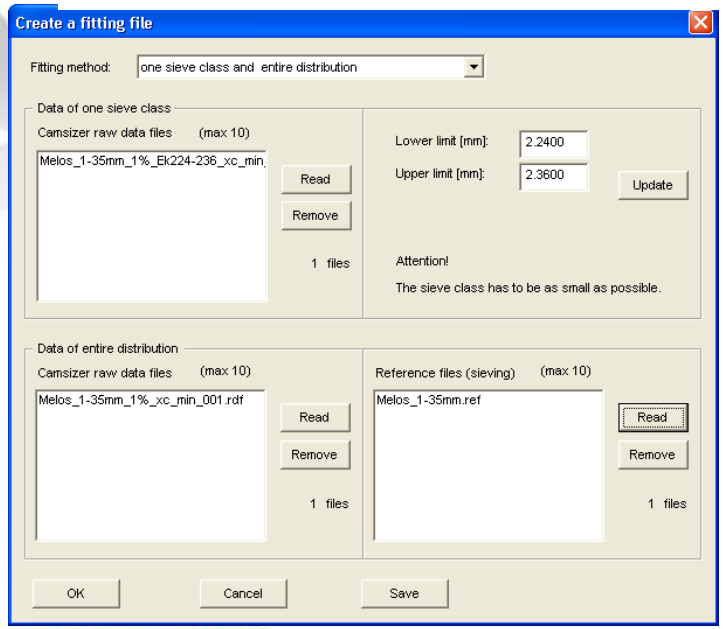
# Digital Imaging ⇔ Sieving

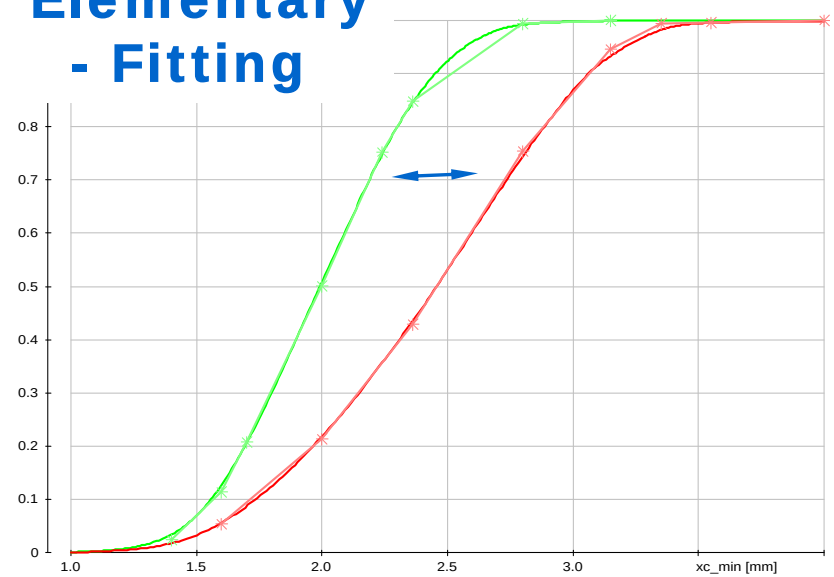



## $Q_3$ – Fitting




## Elementary - Fitting

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**550** installed Instruments worldwide:
Nearly on all continents
For many applications / industries:
**A**mmonium phosphate, **B**ulk blending companies, **C**rop protection, **D**icalcium phosphate, **E**xplosives **F**ertilizer.....



**RE**liable
**T**echnology &
**S**uperior
**CH**aracteristics

# Thank you for your attention!

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