
# NEW! Next Generation Dynamic Image Analysis: ANALYSETTE 28

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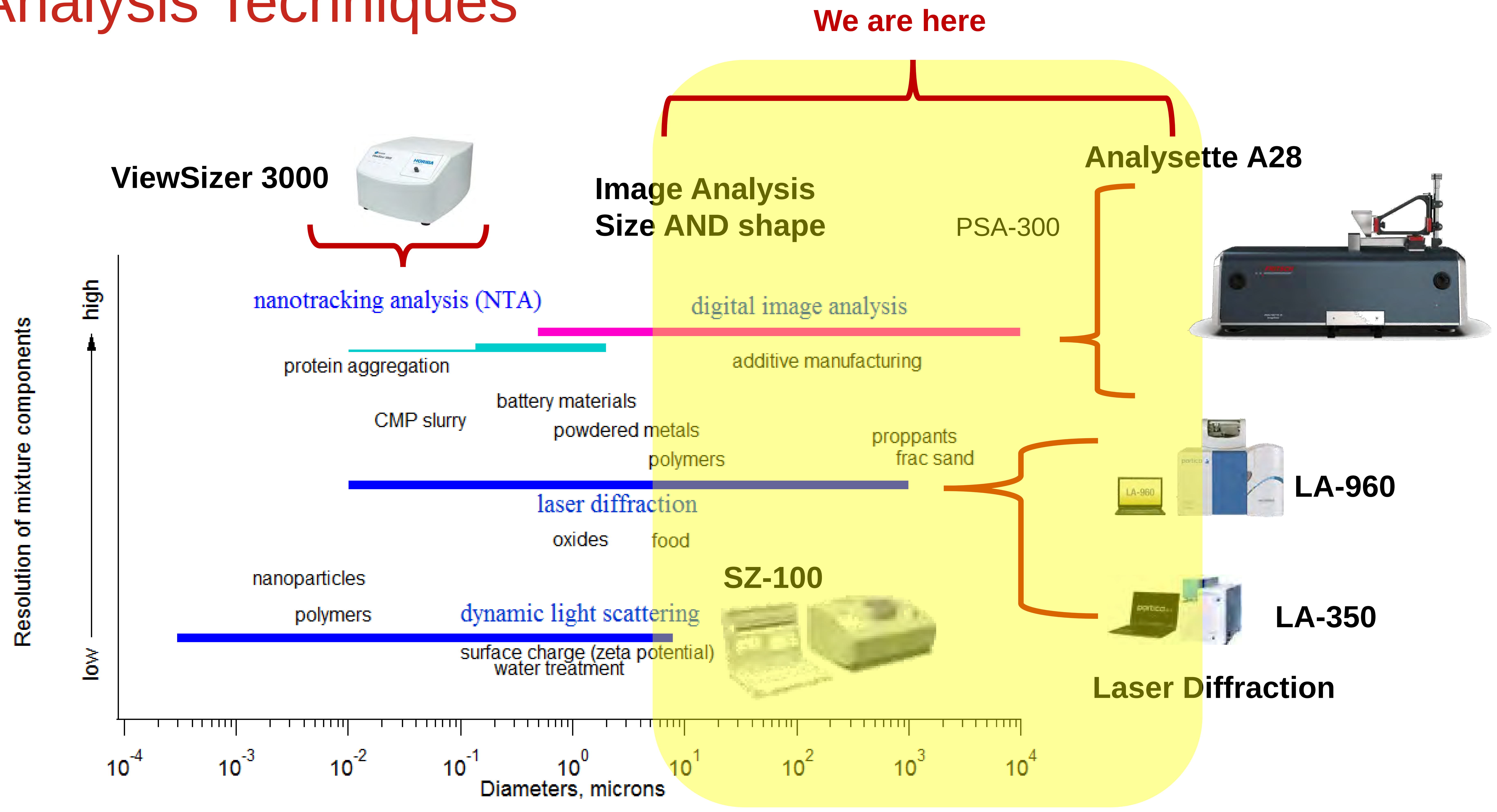
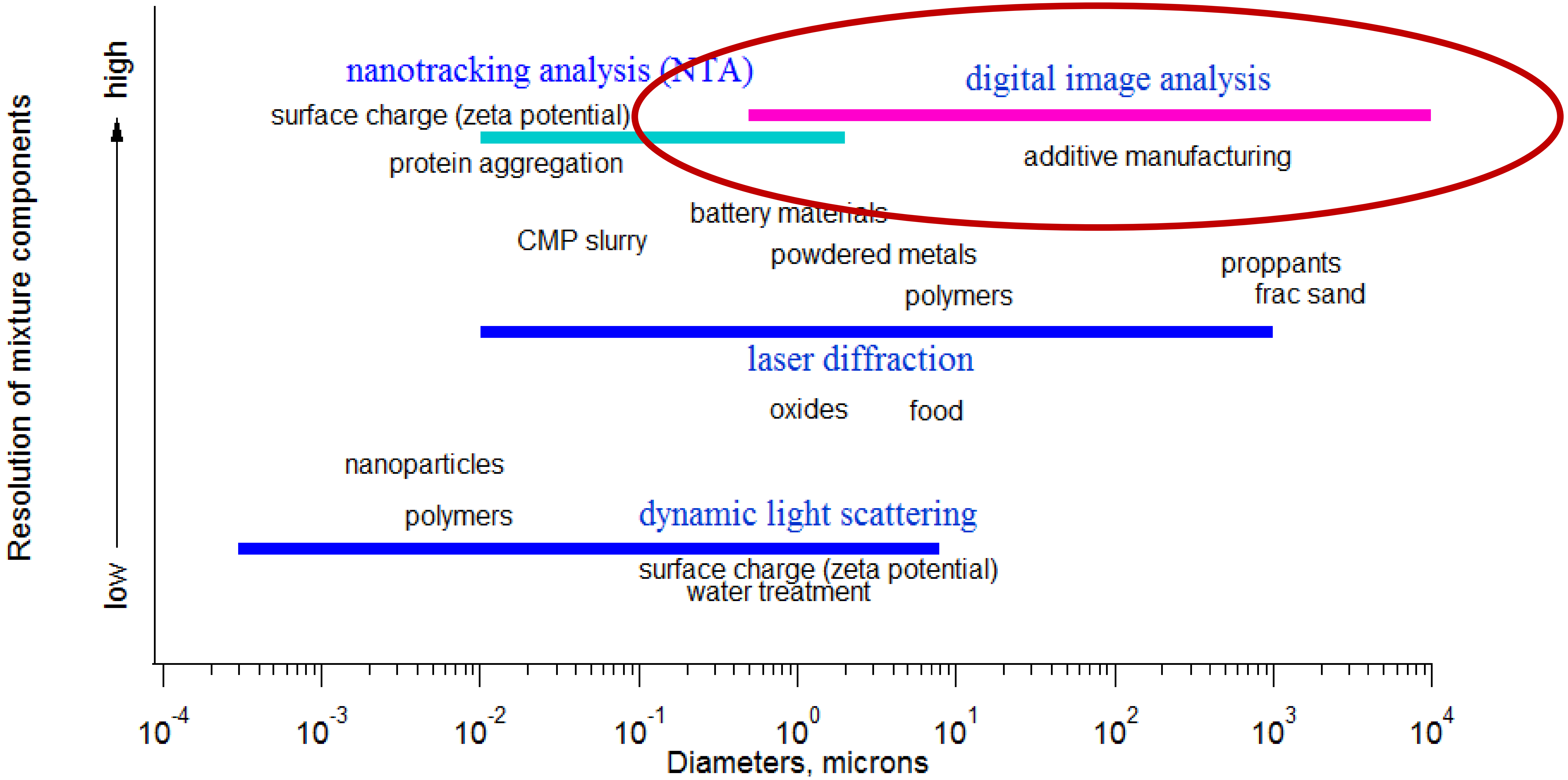
FRITSCH

# Analysis Techniques



# Sizing techniques



# Introduction

- **Günther Crolly (PhD)**
- Head of development particle sizer
- FRITSCH HQ Germany

**FRITSCH** is an internationally respected manufacturer of application-oriented laboratory instruments. Our instruments have been used for decades worldwide for Sample Preparation and Particle Sizing in industry and research laboratories. Trust FRITSCH quality made in Germany, our experience and our service.

# Why image analysis?

- Replace sieves (really!)
- Verify/supplement laser diffraction results (orthogonal technique).
- Access larger sizes than laser diffraction
- Need shape information, for example due to importance of powder flow
- Particle size is a critical quality attribute (CQA) in the realm of Oral solid dosage (OSD) manufacture.
- Particle size directly relates to:
    - Flowability
    - Blend uniformity
    - Compressibility
    - Dissolution rate
    - Bioavailability

same size (cross section), but behave very differently.

# Why image analysis?

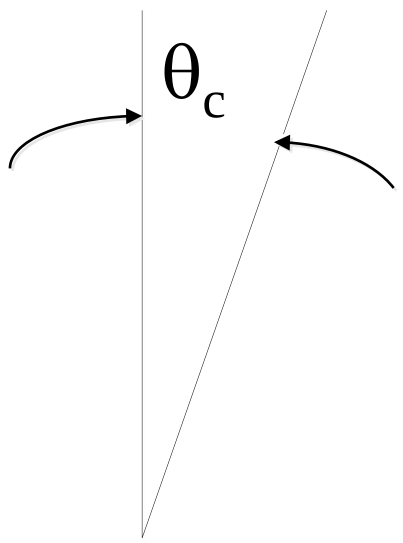
- Replace Sieves

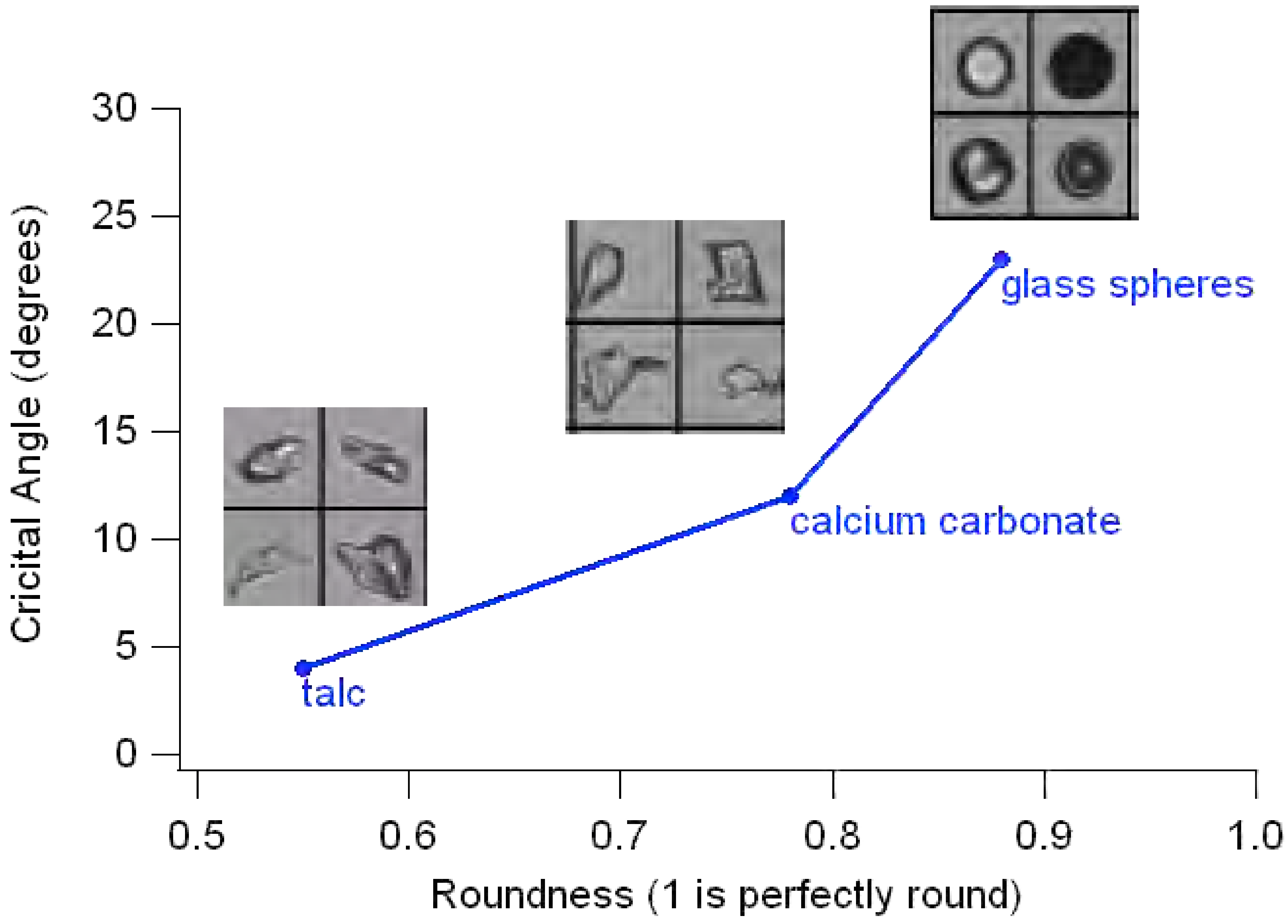
- Tend to wear over time. It is difficult to tell when sieve results are “drifting” due to wear
- Results depend on nature of shaking and loading leading to operator to operator variations in results.
- Small number of size classes

Courtesy Wikipedia user © BMK (de.wikipedia.org)“
//commons.wikimedia.org/wiki/File:Laboratory_sieves_BMK.jpg

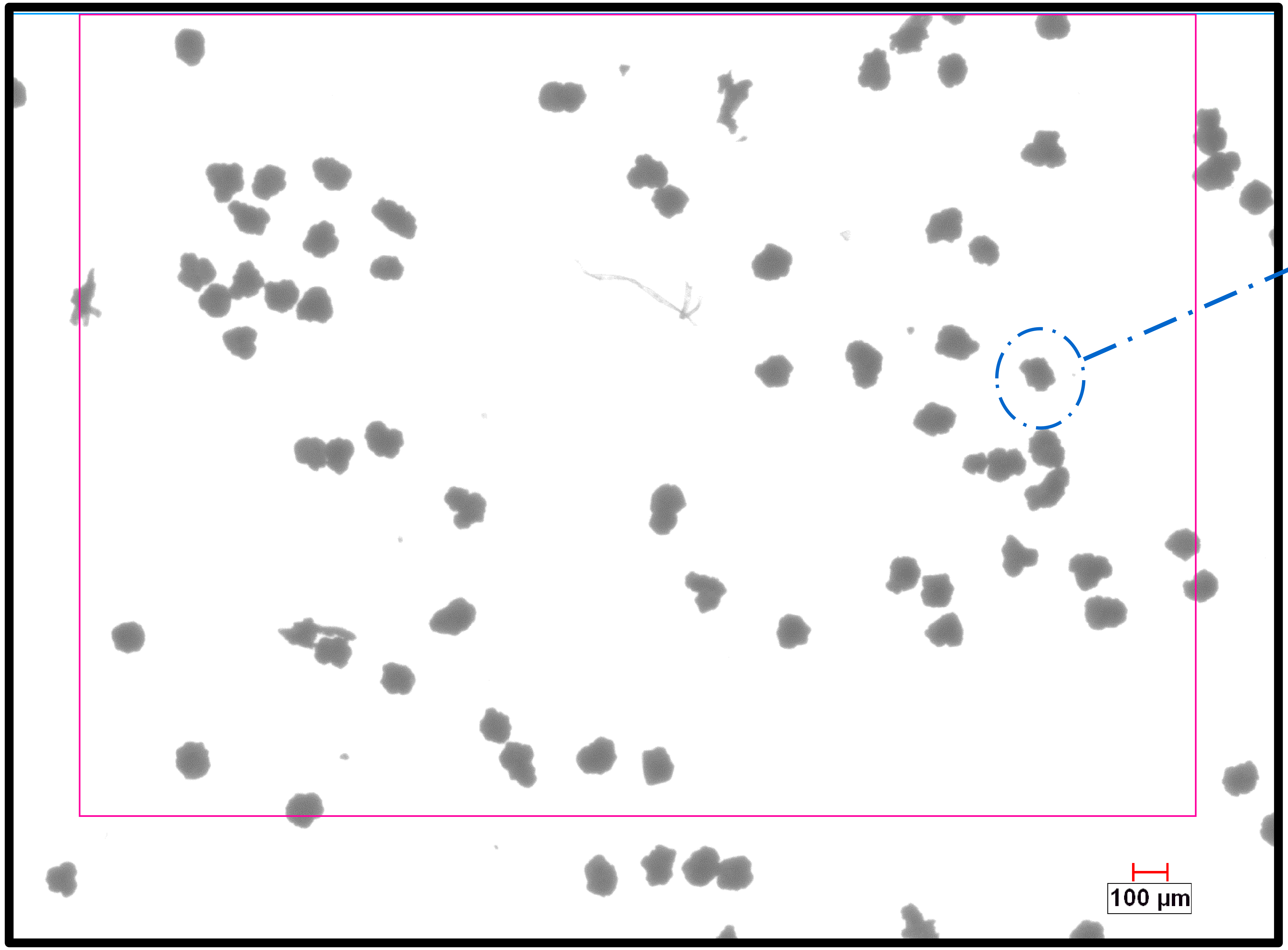
# Effect of shape on flow

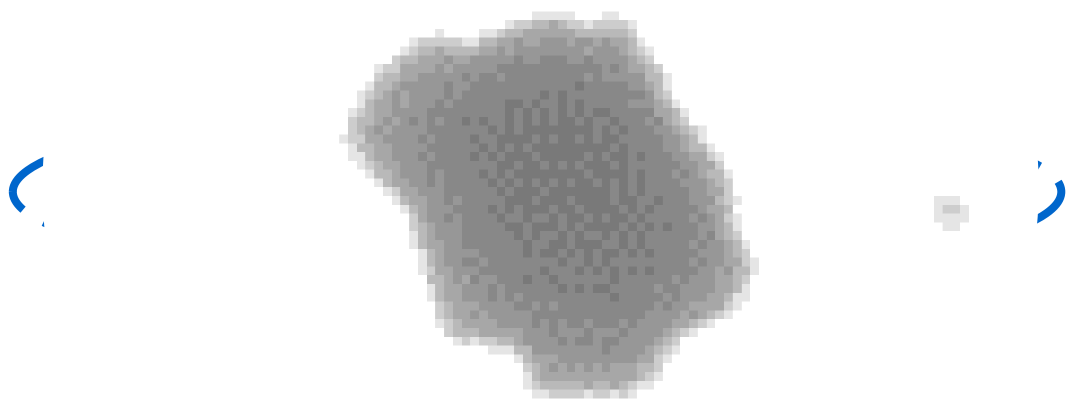
- Yes, I assumed density doesn't matter.
- Roundness is a measure based on particle perimeter.



# What is image analysis?

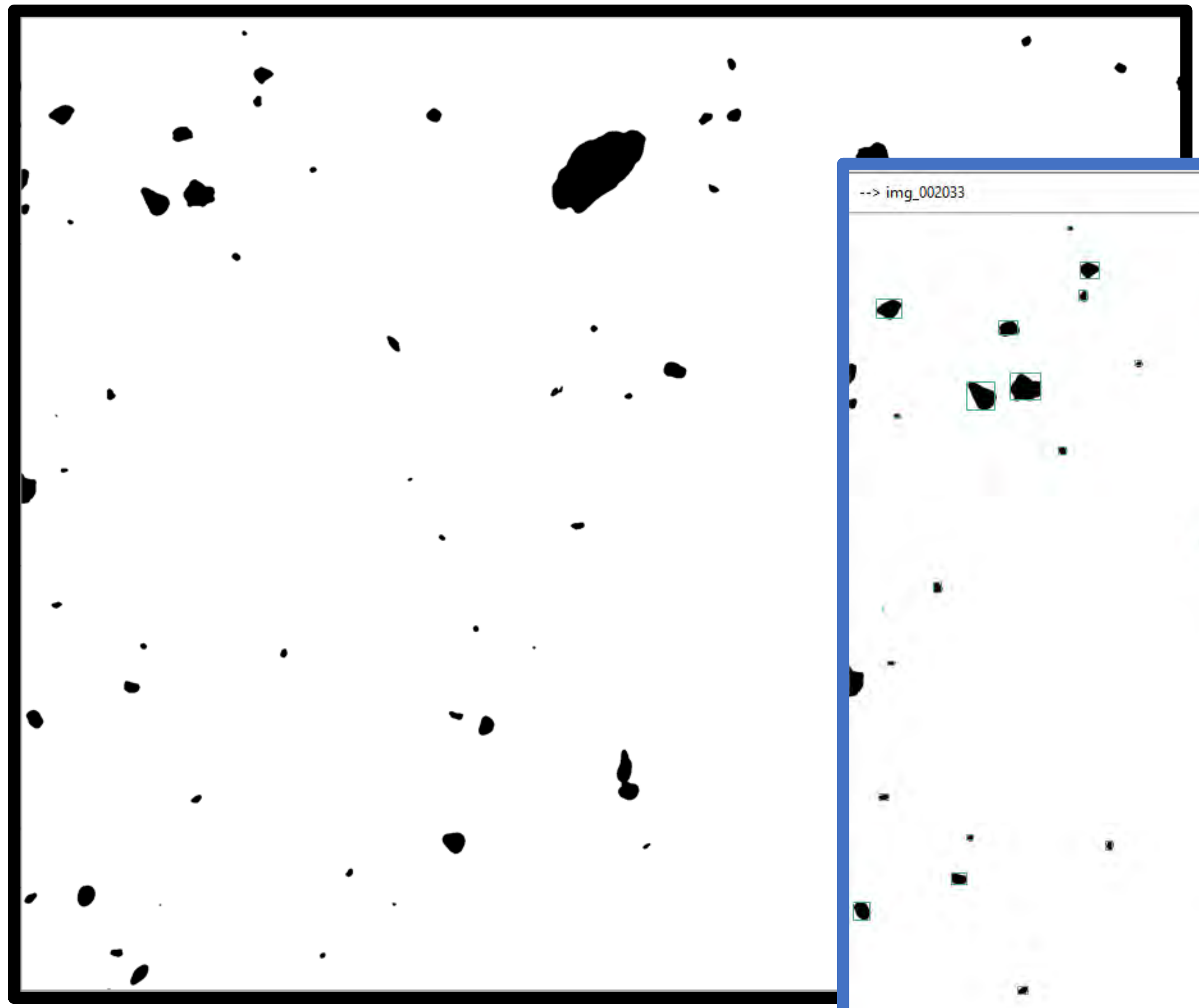


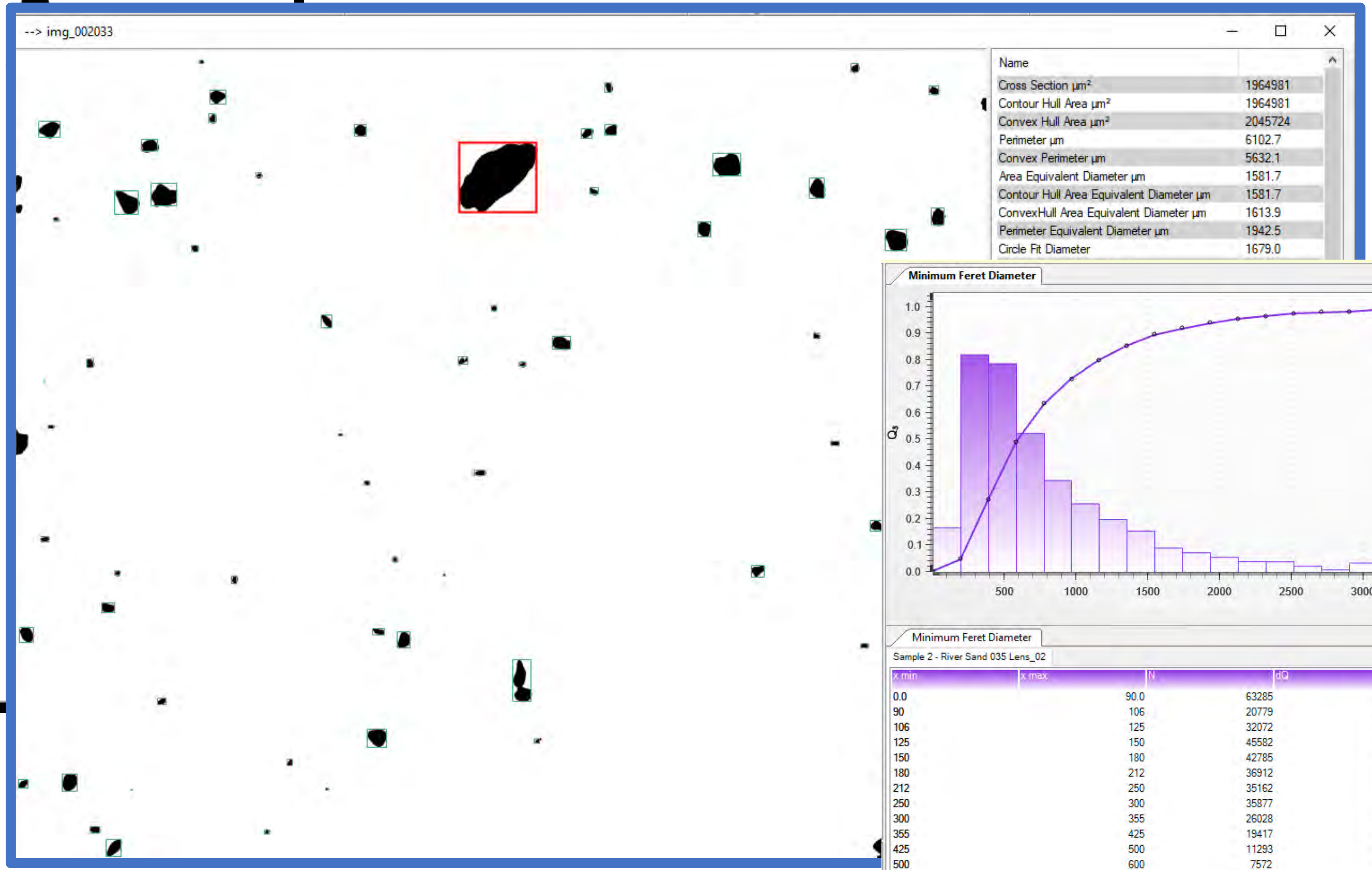
Take a picture, analyze for **size** and **shape**

# Image Analysis

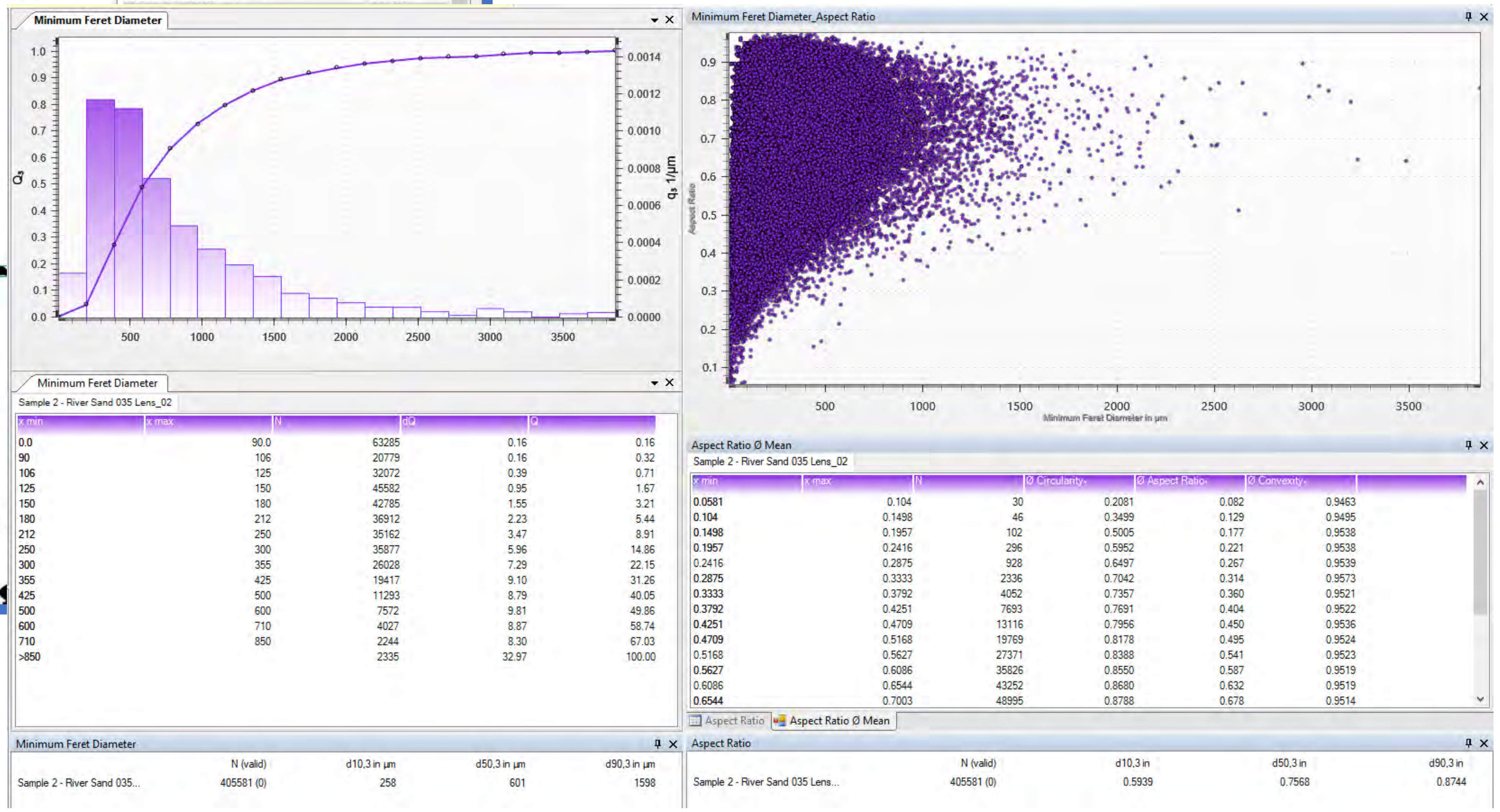
## Collect Image



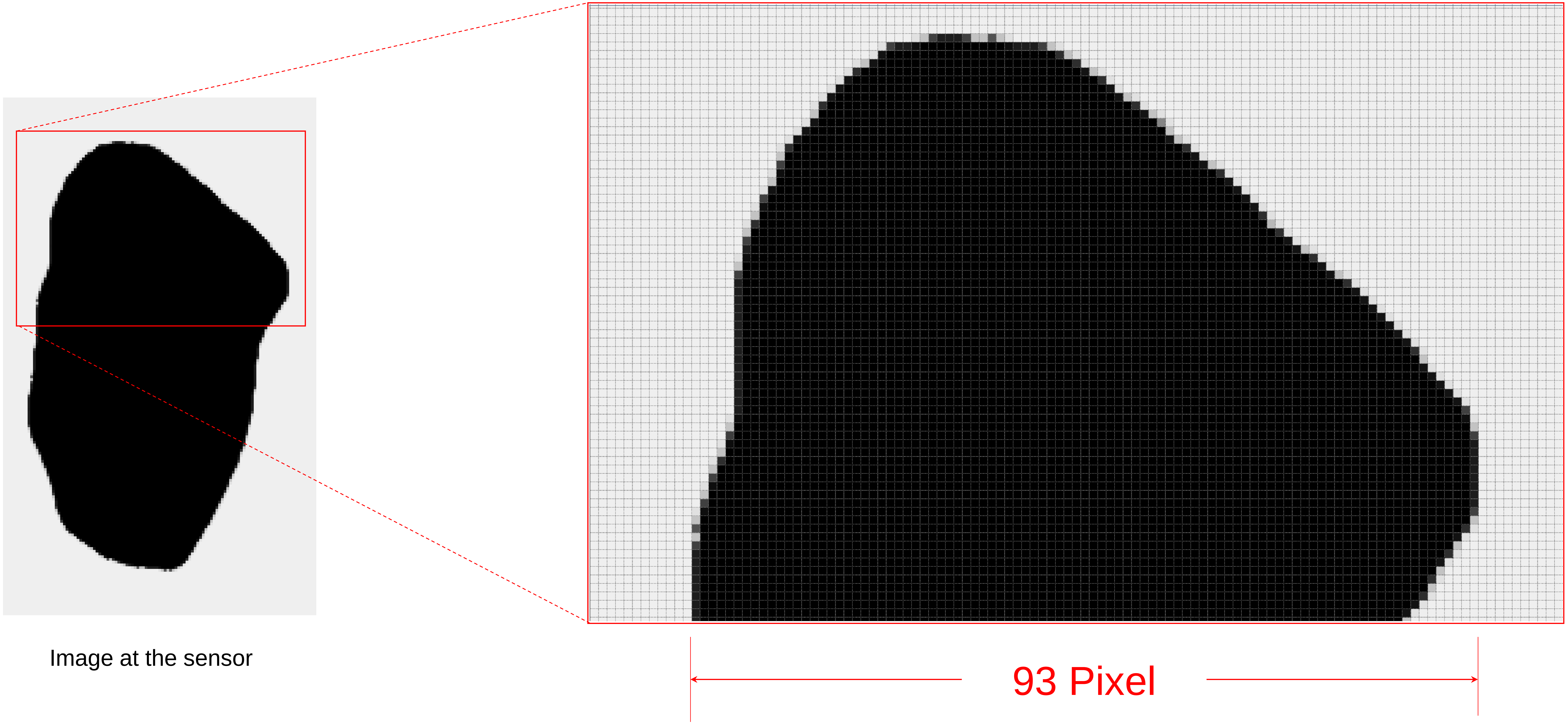
## Particle by particle parameters



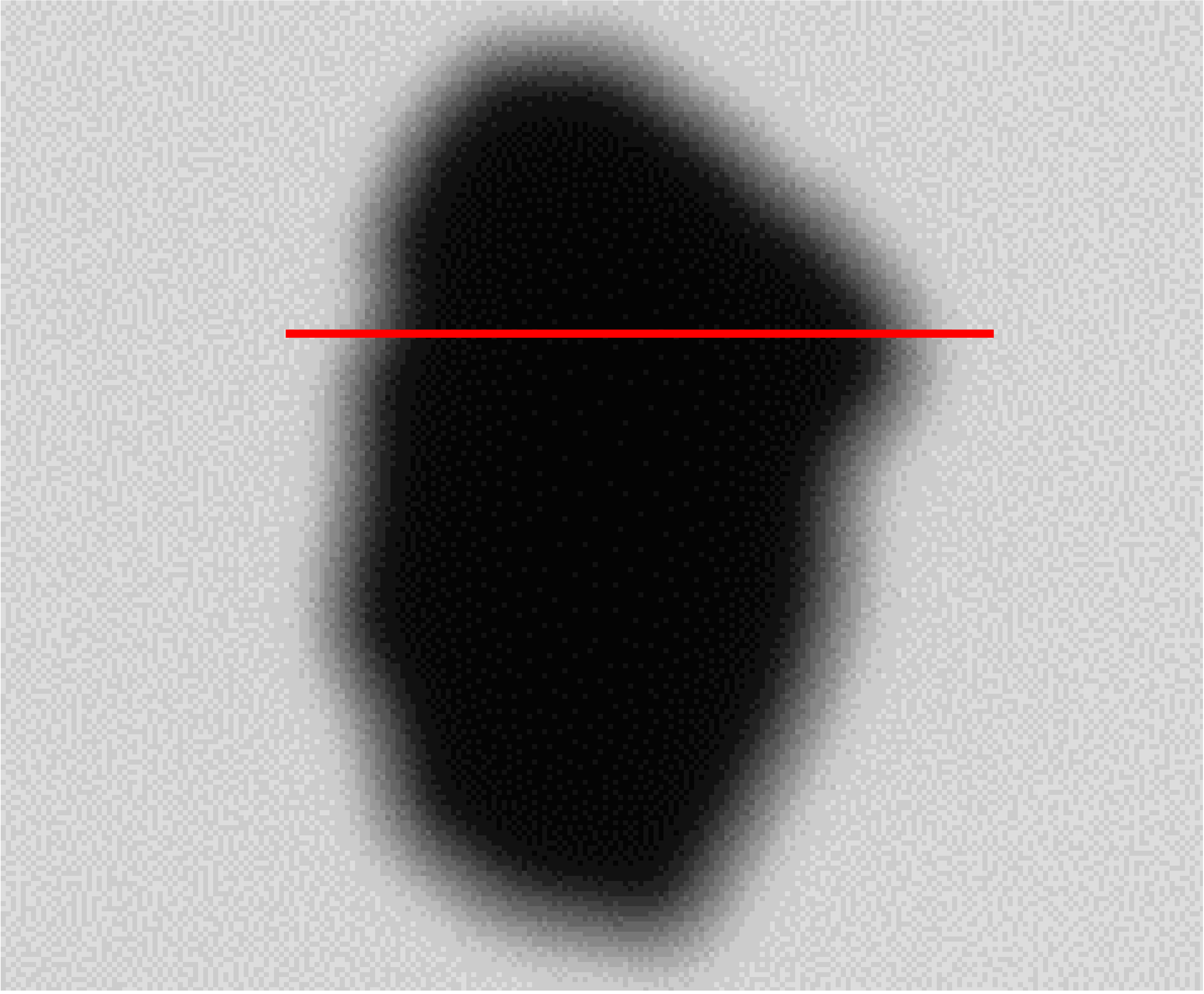

## Population statistics/ parameters



# How images get processed

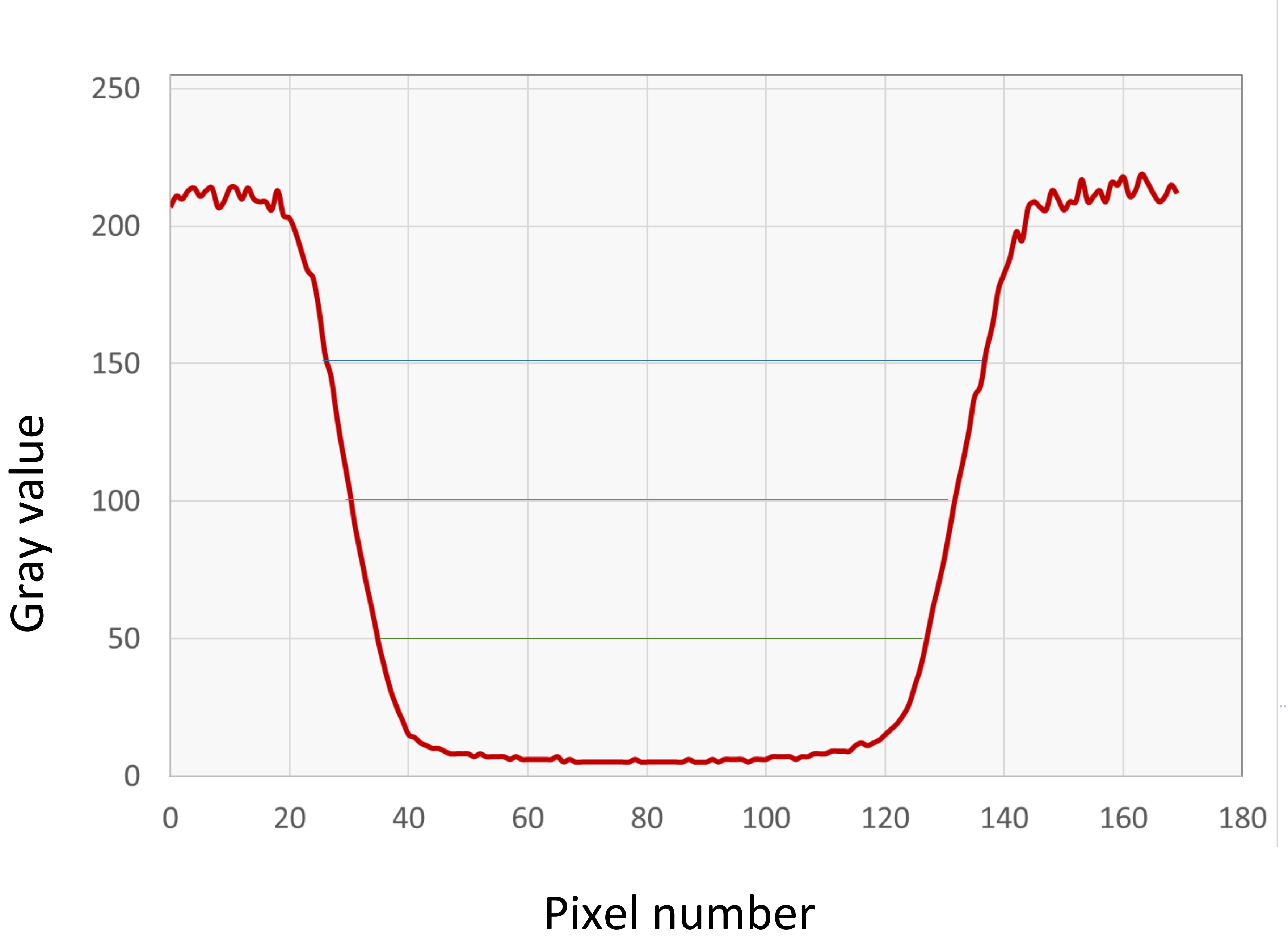


# How real images get processed



**Threshold**

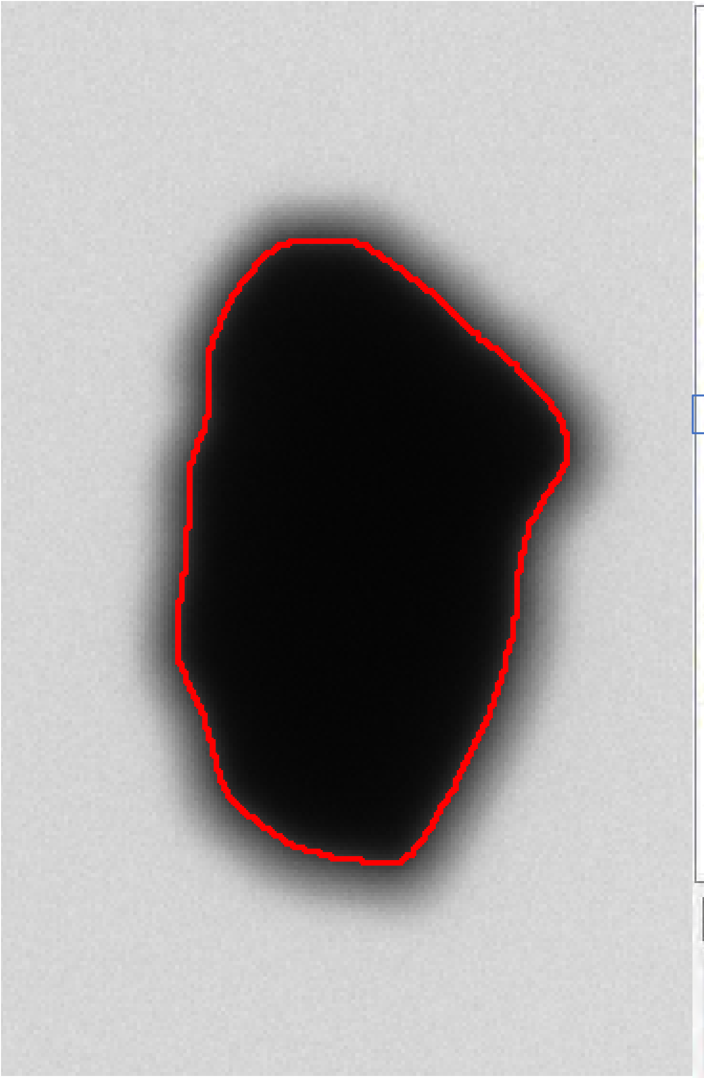
Gray value along a given line



# Binarization threshold

## Threshold

### 50

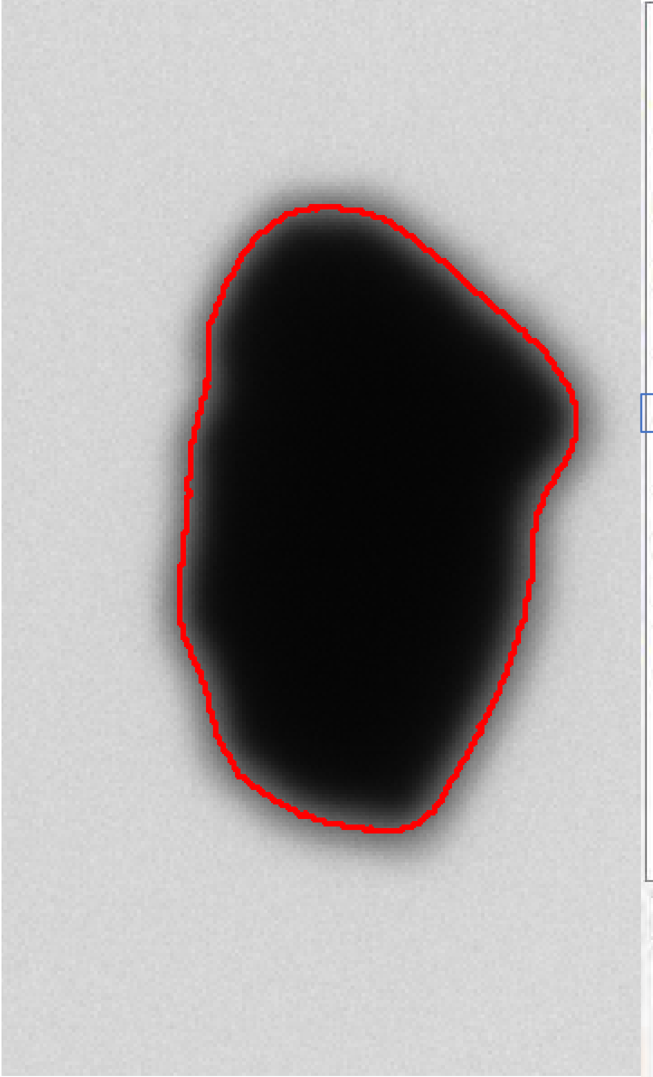


| Name | |
|---|---|
| Cross Section µm² | 266615 |
| Contour Hull Area µm² | 266615 |
| Convex Hull Area µm² | 273346 |
| Perimeter µm | 2115,0 |
| Convex Perimeter µm | 1990,2 |
| Area Equivalent Diameter µm | 582,6 |
| Contour Hull Area Equivalent Diameter µm | 582,6 |
| ConvexHull Area Equivalent Diameter µm | 589,9 |
| Perimeter Equivalent Diameter µm | 673,2 |
| Circle Fit Diameter | 616,4 |
| Minimum Feret Diameter µm | 459,4 |
| Maximum Feret Diameter µm | 776,5 |
| Aspect Ratio | 0,592 |
| Major Ellipsis Axis µm | 780,9 |
| Minor Ellipsis Axis µm | 451,1 |
| Ellipsis Aspect Ratio | 0,578 |
| Solidity | 1,000 |
| Convexity | 0,941 |
| Circularity | 0,865 |
| Focus Parameter | 0,144 |

--> img_000001

| Name | Value |
|---|---|
| Image width (pixel) | 2448 |
| Image height (pixel) | 2048 |
| Pixelsize | 4,69 µm |

### 100

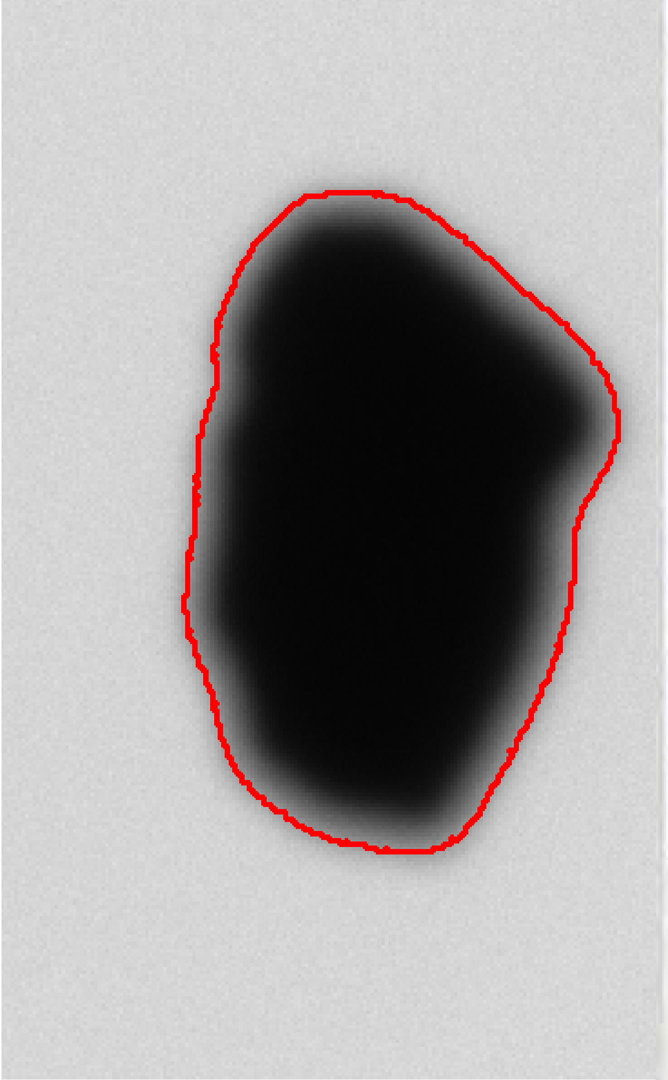


| Name | |
|---|---|
| Cross Section µm² | 321979 |
| Contour Hull Area µm² | 321979 |
| Convex Hull Area µm² | 329370 |
| Perimeter µm | 2359,3 |
| Convex Perimeter µm | 2162,1 |
| Area Equivalent Diameter µm | 640,3 |
| Contour Hull Area Equivalent Diameter µm | 640,3 |
| ConvexHull Area Equivalent Diameter µm | 647,6 |
| Perimeter Equivalent Diameter µm | 751,0 |
| Circle Fit Diameter | 660,4 |
| Minimum Feret Diameter µm | 507,8 |
| Maximum Feret Diameter µm | 833,5 |
| Aspect Ratio | 0,609 |
| Major Ellipsis Axis µm | 829,4 |
| Minor Ellipsis Axis µm | 505,2 |
| Ellipsis Aspect Ratio | 0,609 |
| Solidity | 1,000 |
| Convexity | 0,916 |
| Circularity | 0,853 |
| Focus Parameter | 0,158 |

--> img_000001

| Name | Value |
|---|---|
| Image width (pixel) | 2448 |
| Image height (pixel) | 2048 |
| Pixelsize | 4,69 µm |

### 150



| Name | |
|---|---|
| Cross Section µm² | 374913 |
| Contour Hull Area µm² | 374913 |
| Convex Hull Area µm² | 383139 |
| Perimeter µm | 2549,3 |
| Convex Perimeter µm | 2317,3 |
| Area Equivalent Diameter µm | 690,9 |
| Contour Hull Area Equivalent Diameter µm | 690,9 |
| ConvexHull Area Equivalent Diameter µm | 698,4 |
| Perimeter Equivalent Diameter µm | 811,5 |
| Circle Fit Diameter | 723,6 |
| Minimum Feret Diameter µm | 558,0 |
| Maximum Feret Diameter µm | 886,8 |
| Aspect Ratio | 0,629 |
| Major Ellipsis Axis µm | 886,6 |
| Minor Ellipsis Axis µm | 549,6 |
| Ellipsis Aspect Ratio | 0,620 |
| Solidity | 1,000 |
| Convexity | 0,909 |
| Circularity | 0,851 |
| Focus Parameter | 0,153 |

--> img_000001

| Name | Value |
|---|---|
| Image width (pixel) | 2448 |
| Image height (pixel) | 2048 |
| Pixelsize | 4,69 µm |

# Binarization threshold

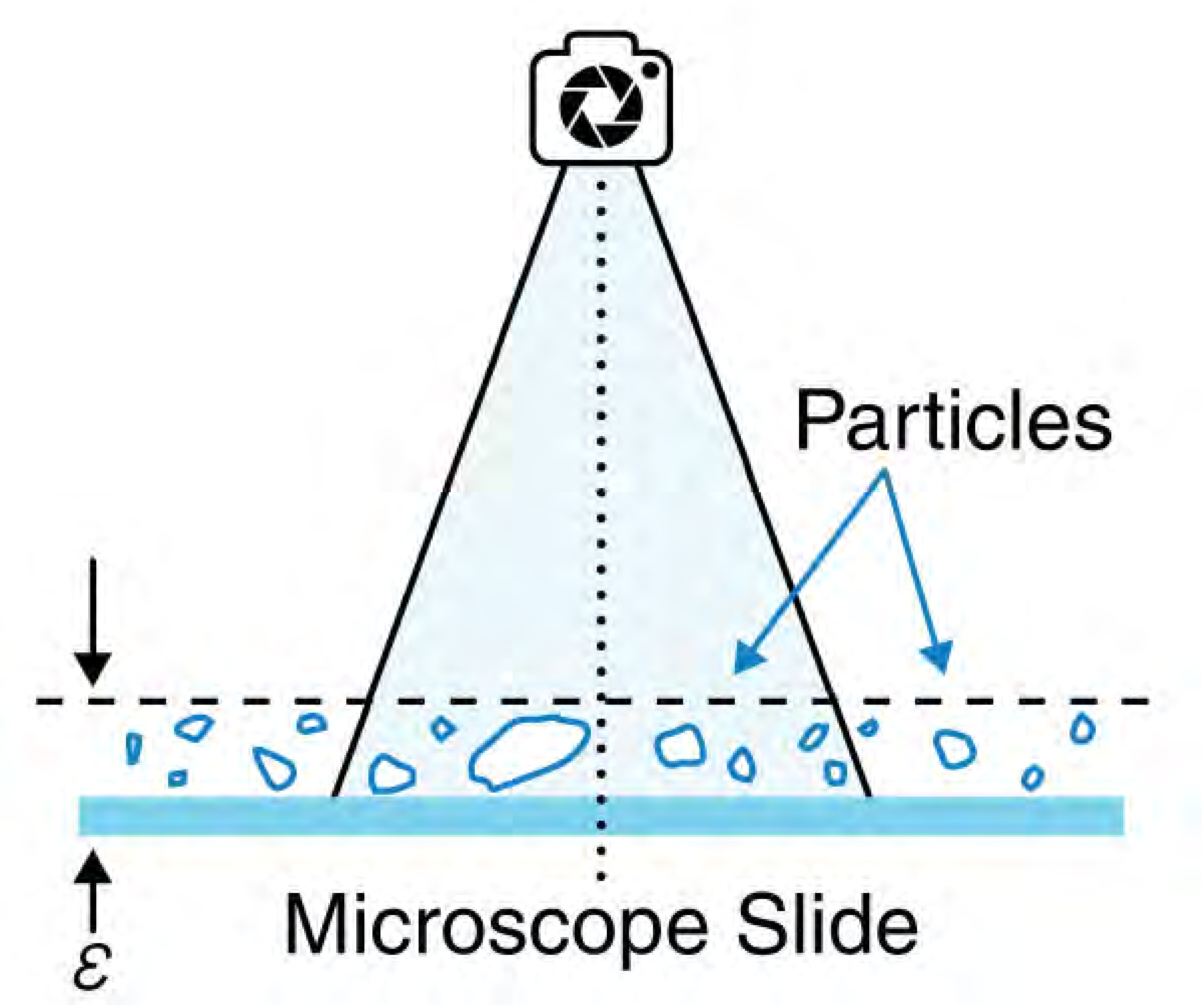
**Setting the binarization threshold**

- Automatic
- Manual setting

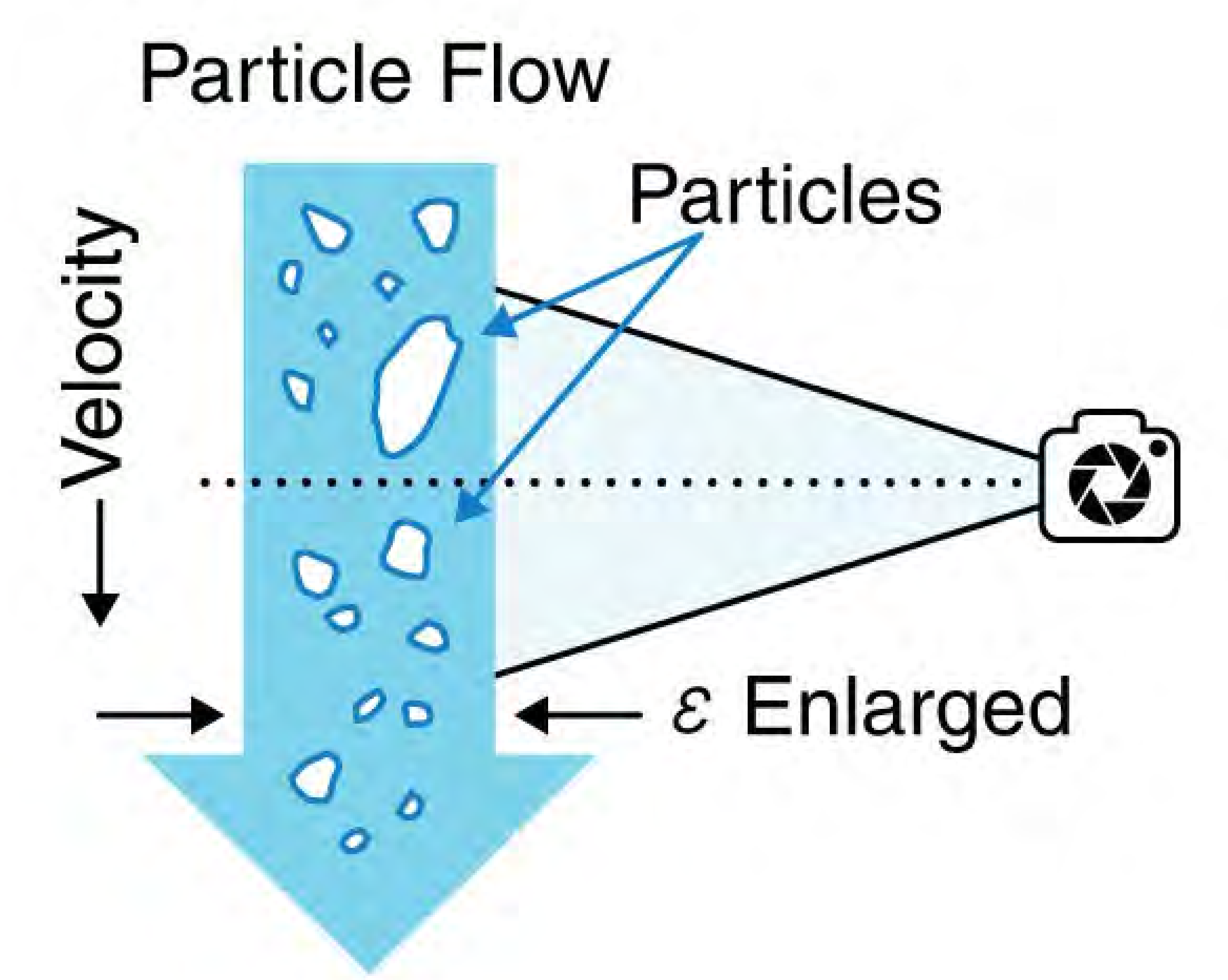
**Superposition** of the
established **particle boundaries**
(or the binarized images)
and the **original (raw) images**
should always be possible!

# Dynamic vs Static?

Static Image Analysis
Particle are not moving.

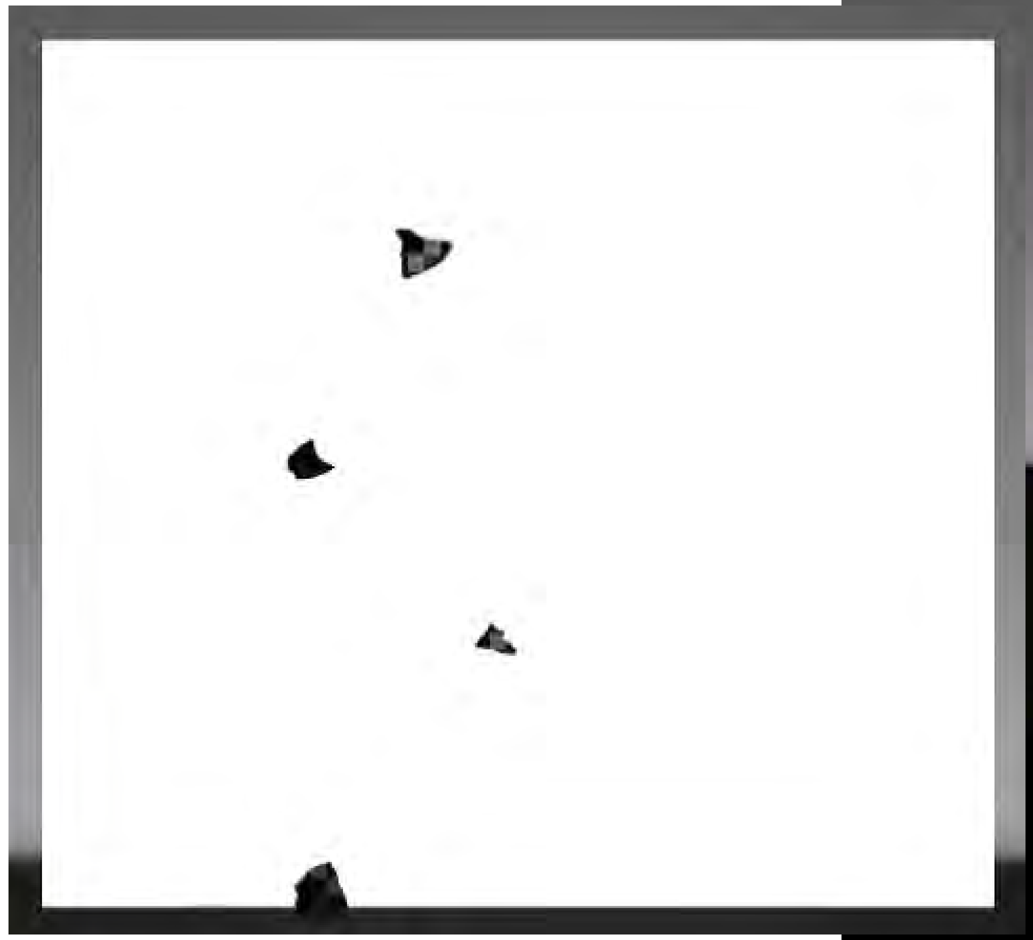


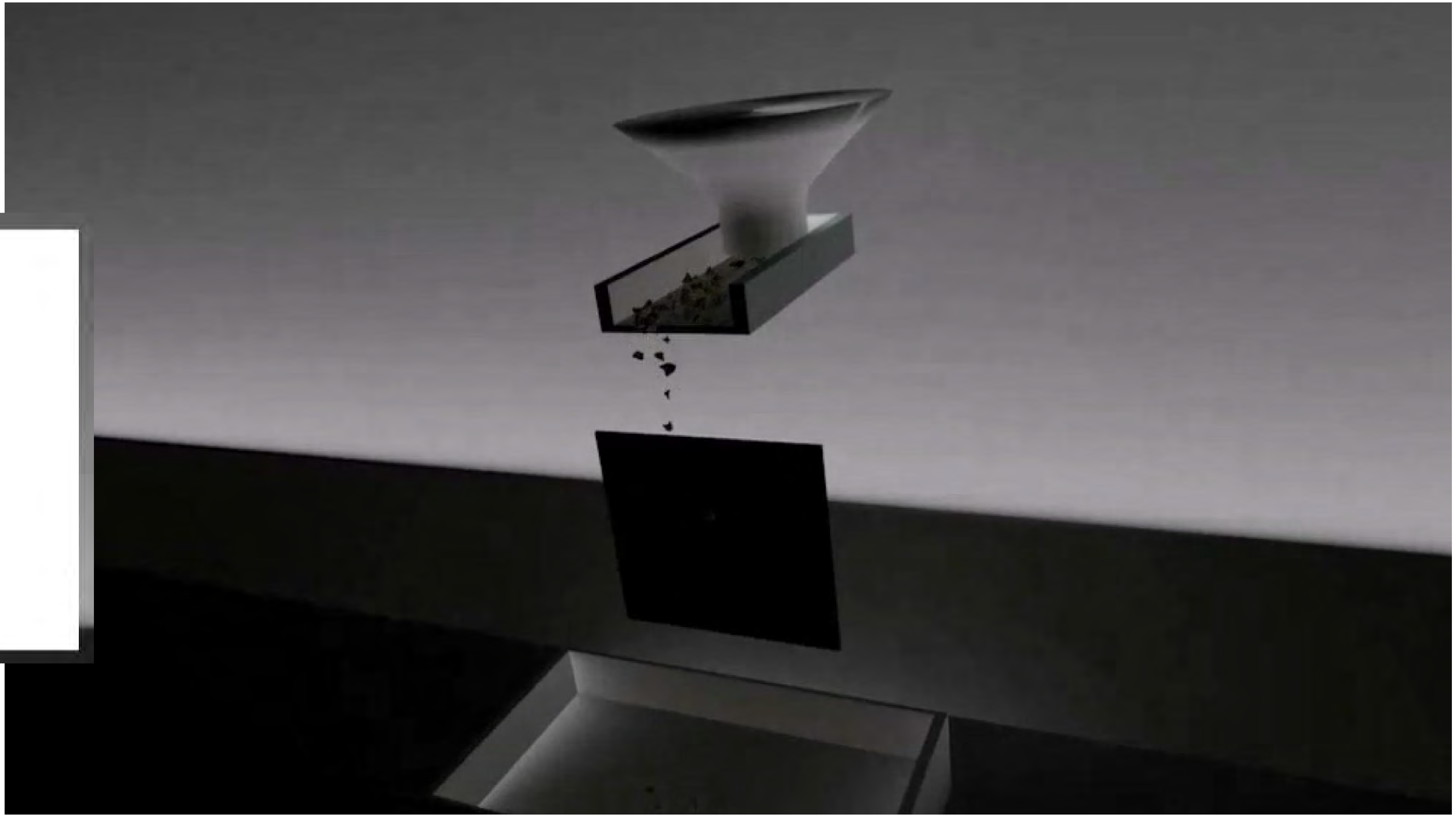

Dynamic Image Analysis
Particles are moving.



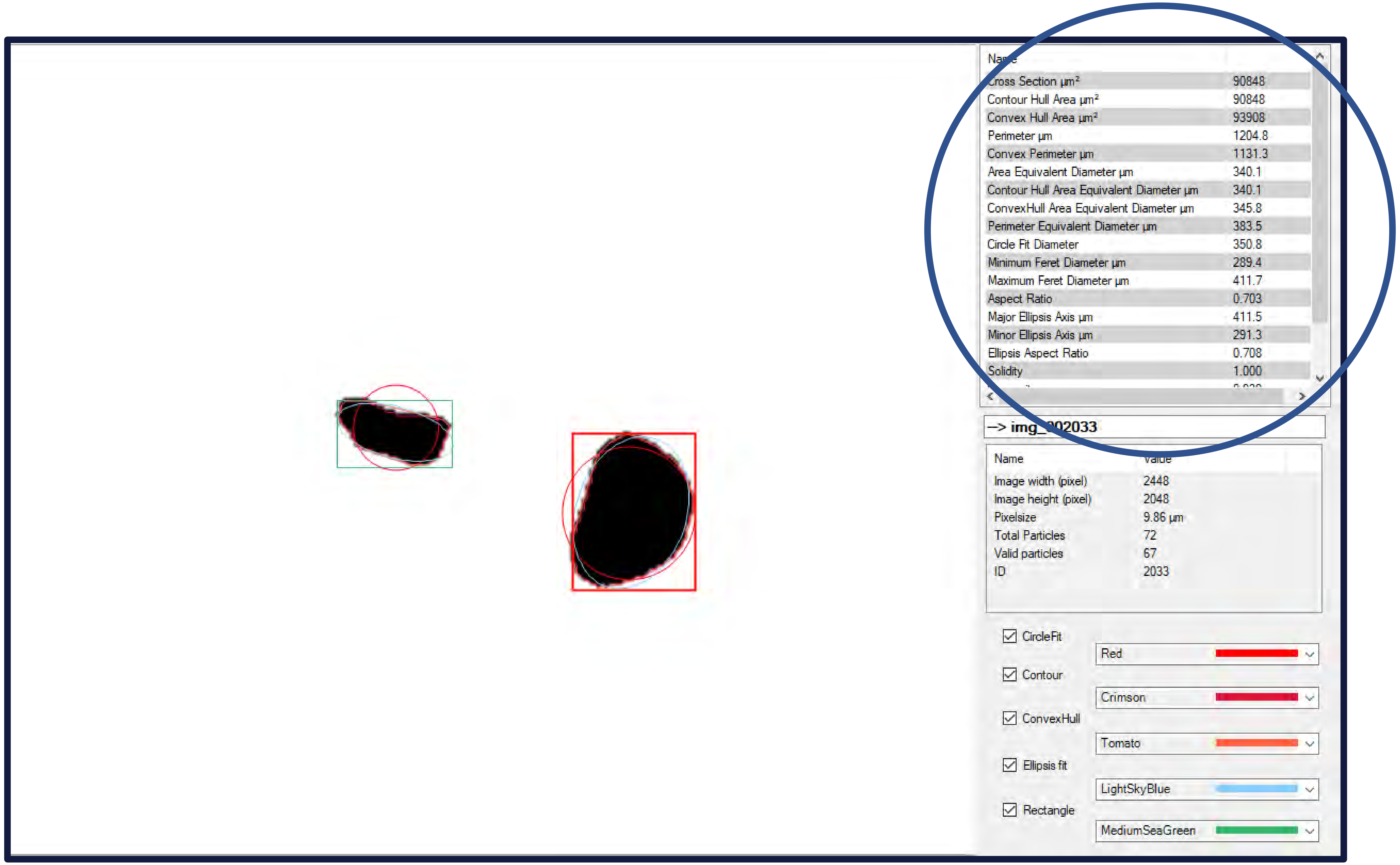
# Making particles move

Send them off the edge of a hopper.





# Result Parameters




What are all of these numbers from a single particle?

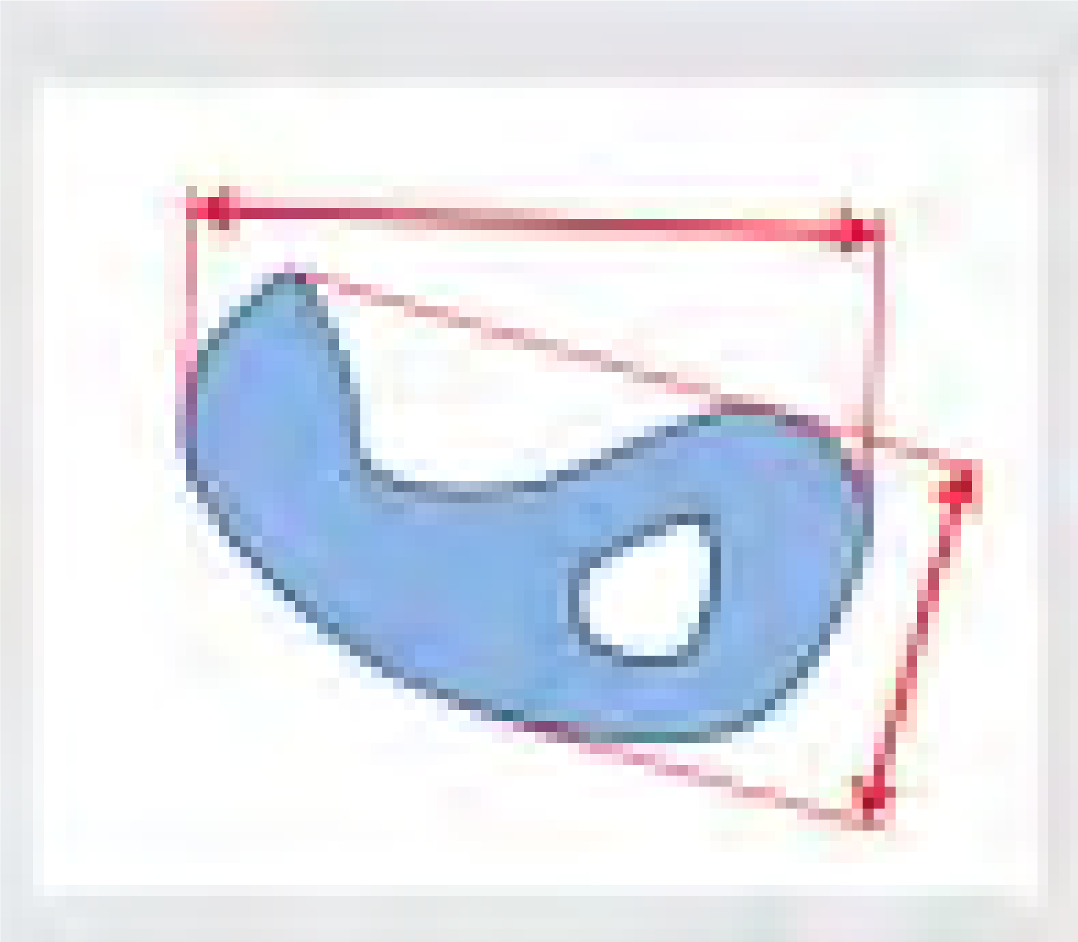
Need to check your manual for the definitions.

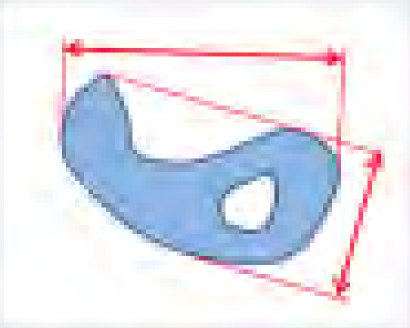
Examples follow.

# Feret (size) and Aspect ratio

Feret: size measured with calipers.

Aspect ratio: Here it is max size over min size



| No. | Name | Symbol | Definition | Example |
|---|---|---|---|---|
| 6 | Feret diameter (in µm) | $d_F$ | The Feret diameter is the distance between two parallel tangents, that are touching the particle projection on opposite sides of it. The tangents must not cross the edge of the particle projection at any point. The tangents can have any orientation. | |
| | ***Minimum** and **maximum** **Feret diameter*** *(in µm)* | $d_{F\,min}$ $d_{F\,max}$ | If the tangents illustrated above are rotated from 0 - 360° (relative to any direction) the Feret diameter adopts values between the minimum and the maximum Feret diameter. Minimum and maximum Feret diameters are usually not measured in directions that are at right angles to each other. |  |
| | **Aspect ratio** | $AV_F$ | The ratio of maximum to minimum Feret diameter $$AV_F = \frac{d_{F\,max}}{d_{F\,min}}$$ | |

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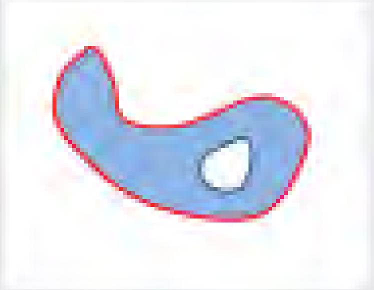
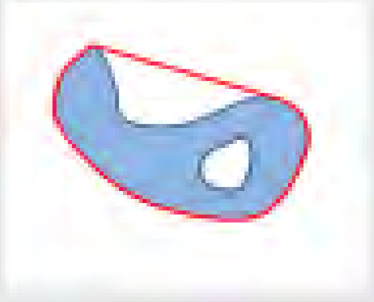
# Convexity (shape)

Convexity: Convex perimeter over particle perimeter

For circle (or other ellipse) result is 1.

As particle surface has more variation, value decreases.

| | | | |
|---|---|---|---|
| 8 | ***Convexity*** | *K* | Ratio of convex perimeter (no. 5) to the particle perimeter (no. 4): $$K = \frac{U_{konvex}}{U}$$ |

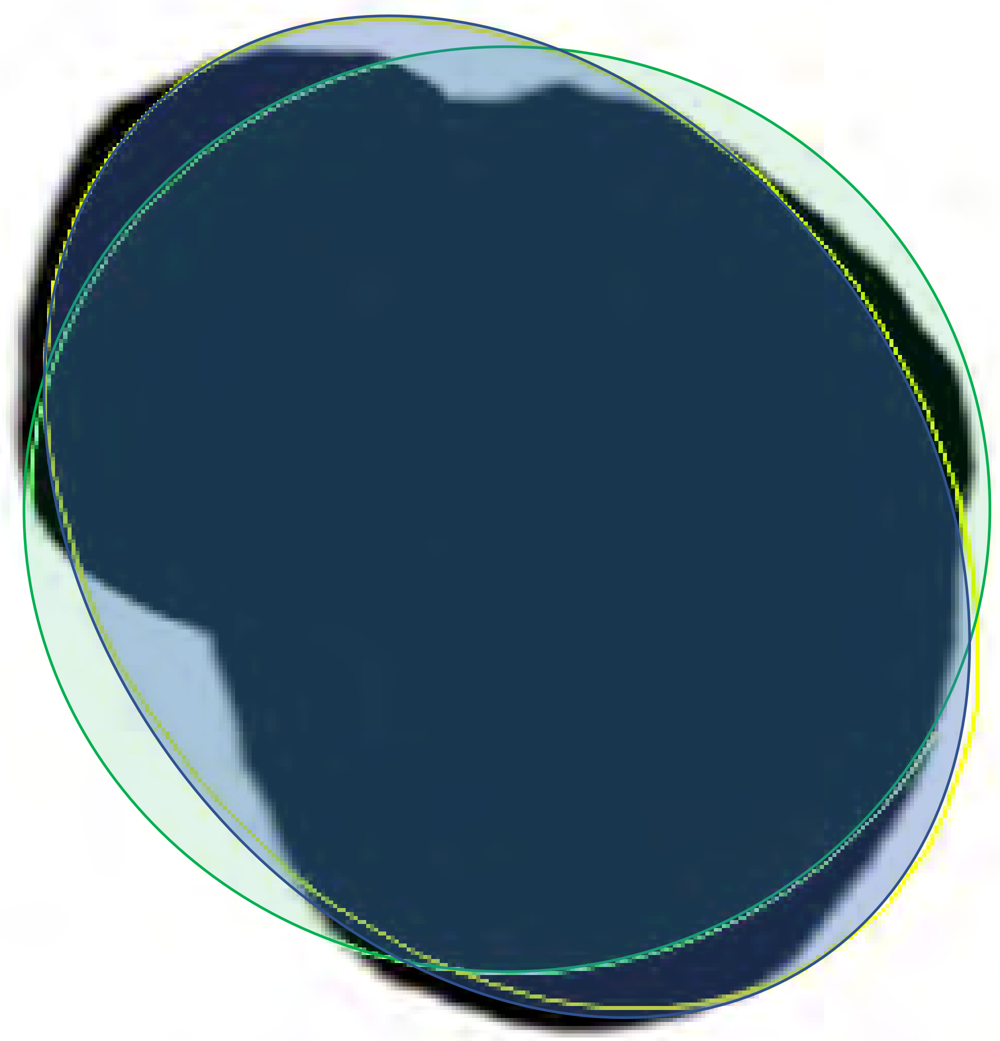
| | | | | |
|---|---|---|---|---|
| 4 | Perimeter (in µm) | *U* | Perimeter of particle projection |  |
| | **Equivalent diameter of the perimeter** (in µm) ***Perimeter-equivalent diameter*** | *d(U)* | The diameter of a spherical particle, whose projection has the same perimeter as the particle $$d(U) = \frac{U}{\pi}$$ | |
| 5 | ***Convex perimeter (in µm)*** | $U_{konvex}$ | Convex perimeter of particle projection |  |
| | **Equivalent diameter of the convex perimeter** (in µm) | $d(U_{konvex})$ | The diameter of a spherical particle, whose perimeter is the same as the convex perimeter of the particle | |

# Some fits

Circular fit (green) used for some size parameters.

Elliptical fit (blue) used for some size and shape parameters.

Less sensitive to pixelation.

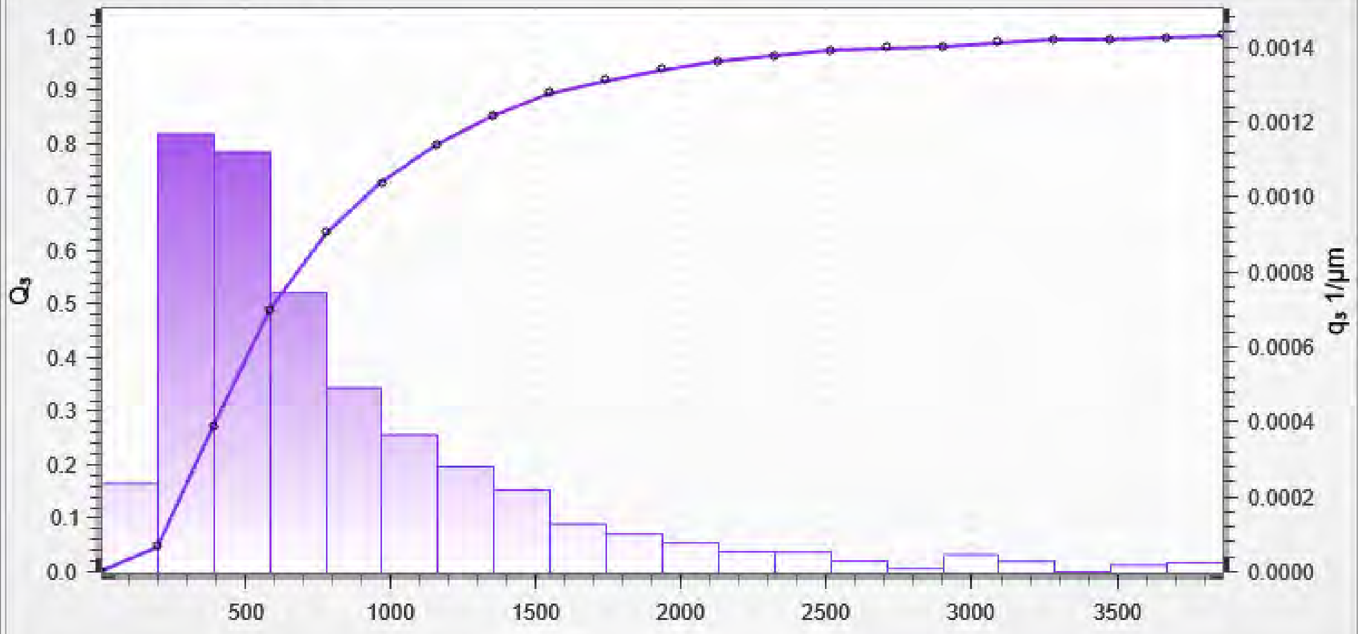


Convex hull…This gets longer than the perimeter…but it matches the perimeter for an ellipse.

# Massive results…

One particle has a lot of results…now we repeat over many particles.

Minimum Feret Diameter




Minimum Feret Diameter

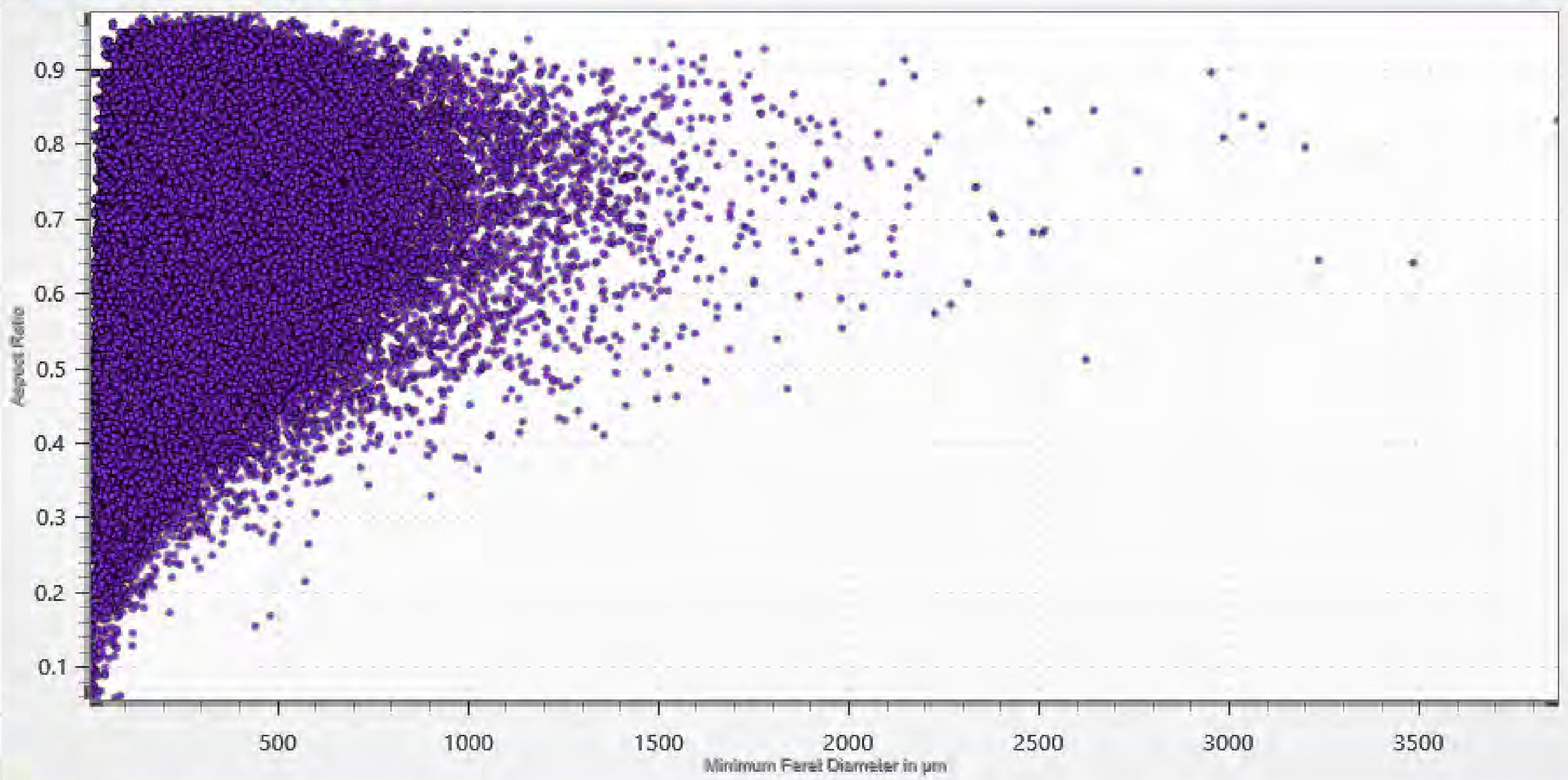
Sample 2 - River Sand 035 Lens_02

| x min | x max | N | dQ | Q |
|---|---|---|---|---|
| 0.0 | 90.0 | 63285 | 0.16 | 0.16 |
| 90 | 106 | 20779 | 0.16 | 0.32 |
| 106 | 125 | 32072 | 0.39 | 0.71 |
| 125 | 150 | 45582 | 0.95 | 1.67 |
| 150 | 180 | 42785 | 1.55 | 3.21 |
| 180 | 212 | 36912 | 2.23 | 5.44 |
| 212 | 250 | 35162 | 3.47 | 8.91 |
| 250 | 300 | 35877 | 5.96 | 14.86 |
| 300 | 355 | 26028 | 7.29 | 22.15 |
| 355 | 425 | 19417 | 9.10 | 31.26 |
| 425 | 500 | 11293 | 8.79 | 40.05 |
| 500 | 600 | 7572 | 9.81 | 49.86 |
| 600 | 710 | 4027 | 8.87 | 58.74 |
| 710 | 850 | 2244 | 8.30 | 67.03 |
| >850 | | 2335 | 32.97 | 100.00 |

Minimum Feret Diameter

| | N (valid) | d10,3 in µm | d50,3 in µm | d90,3 in µm |
|---|---|---|---|---|
| Sample 2 - River Sand 035... | 405581 (0) | 258 | 601 | 1598 |

Minimum Feret Diameter_Aspect Ratio




Aspect Ratio Ø Mean

Sample 2 - River Sand 035 Lens_02

| x min | x max | N | Ø Circularity | Ø Aspect Ratio | Ø Convexity |
|---|---|---|---|---|---|
| 0.0581 | 0.104 | 30 | 0.2081 | 0.082 | 0.9463 |
| 0.104 | 0.1498 | 46 | 0.3499 | 0.129 | 0.9495 |
| 0.1498 | 0.1957 | 102 | 0.5005 | 0.177 | 0.9538 |
| 0.1957 | 0.2416 | 296 | 0.5952 | 0.221 | 0.9538 |
| 0.2416 | 0.2875 | 928 | 0.6497 | 0.267 | 0.9539 |
| 0.2875 | 0.3333 | 2336 | 0.7042 | 0.314 | 0.9573 |
| 0.3333 | 0.3792 | 4052 | 0.7357 | 0.360 | 0.9521 |
| 0.3792 | 0.4251 | 7693 | 0.7691 | 0.404 | 0.9522 |
| 0.4251 | 0.4709 | 13116 | 0.7956 | 0.450 | 0.9536 |
| 0.4709 | 0.5168 | 19769 | 0.8178 | 0.495 | 0.9524 |
| 0.5168 | 0.5627 | 27371 | 0.8388 | 0.541 | 0.9523 |
| 0.5627 | 0.6086 | 35826 | 0.8550 | 0.587 | 0.9519 |
| 0.6086 | 0.6544 | 43252 | 0.8680 | 0.632 | 0.9519 |
| 0.6544 | 0.7003 | 48995 | 0.8788 | 0.678 | 0.9514 |

Aspect Ratio | Aspect Ratio Ø Mean

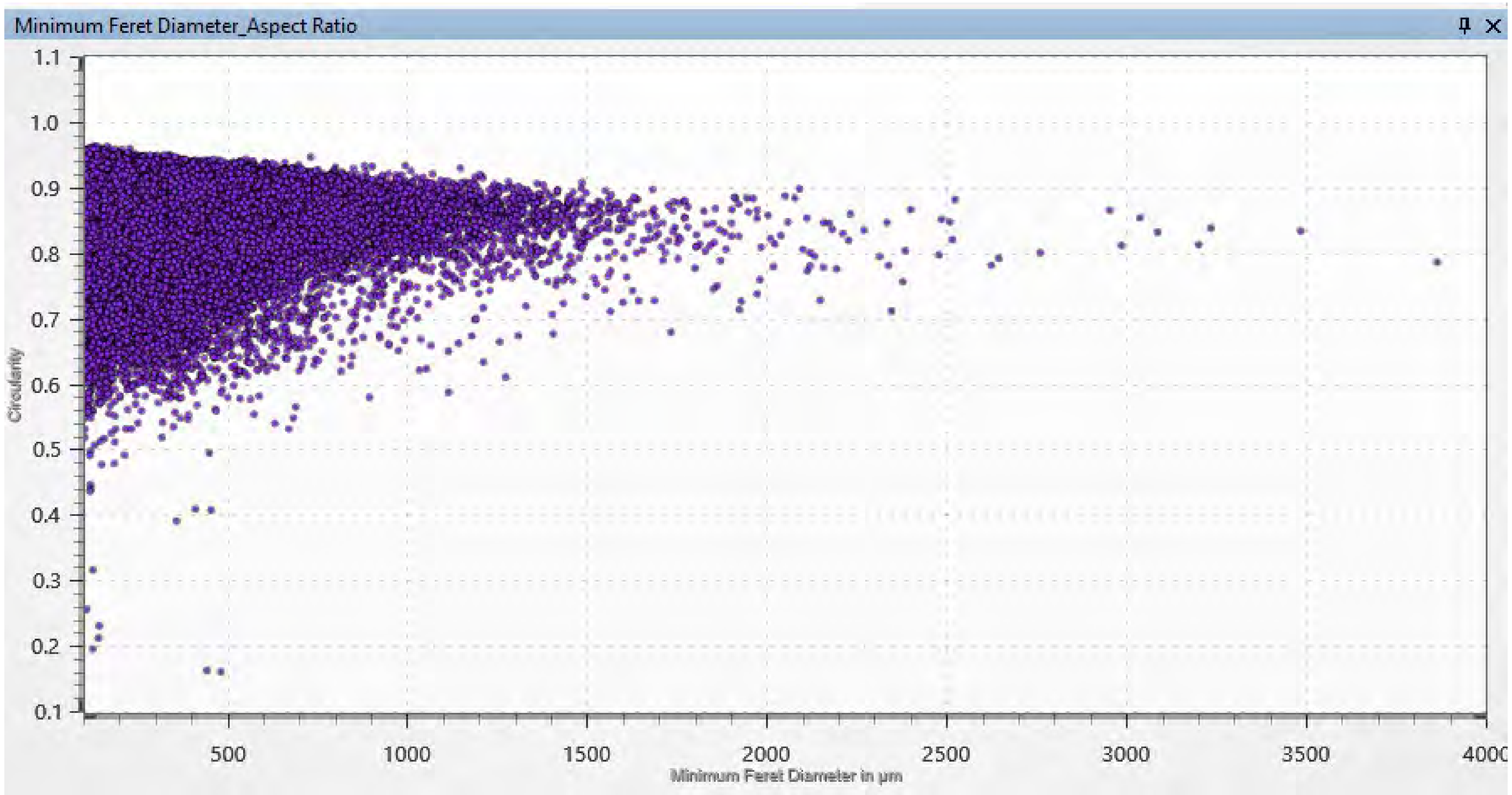
Aspect Ratio

| | N (valid) | d10,3 in | d50,3 in | d90,3 in |
|---|---|---|---|---|
| Sample 2 - River Sand 035 Lens... | 405581 (0) | 0.5939 | 0.7568 | 0.8744 |

# 2-D point cloud…

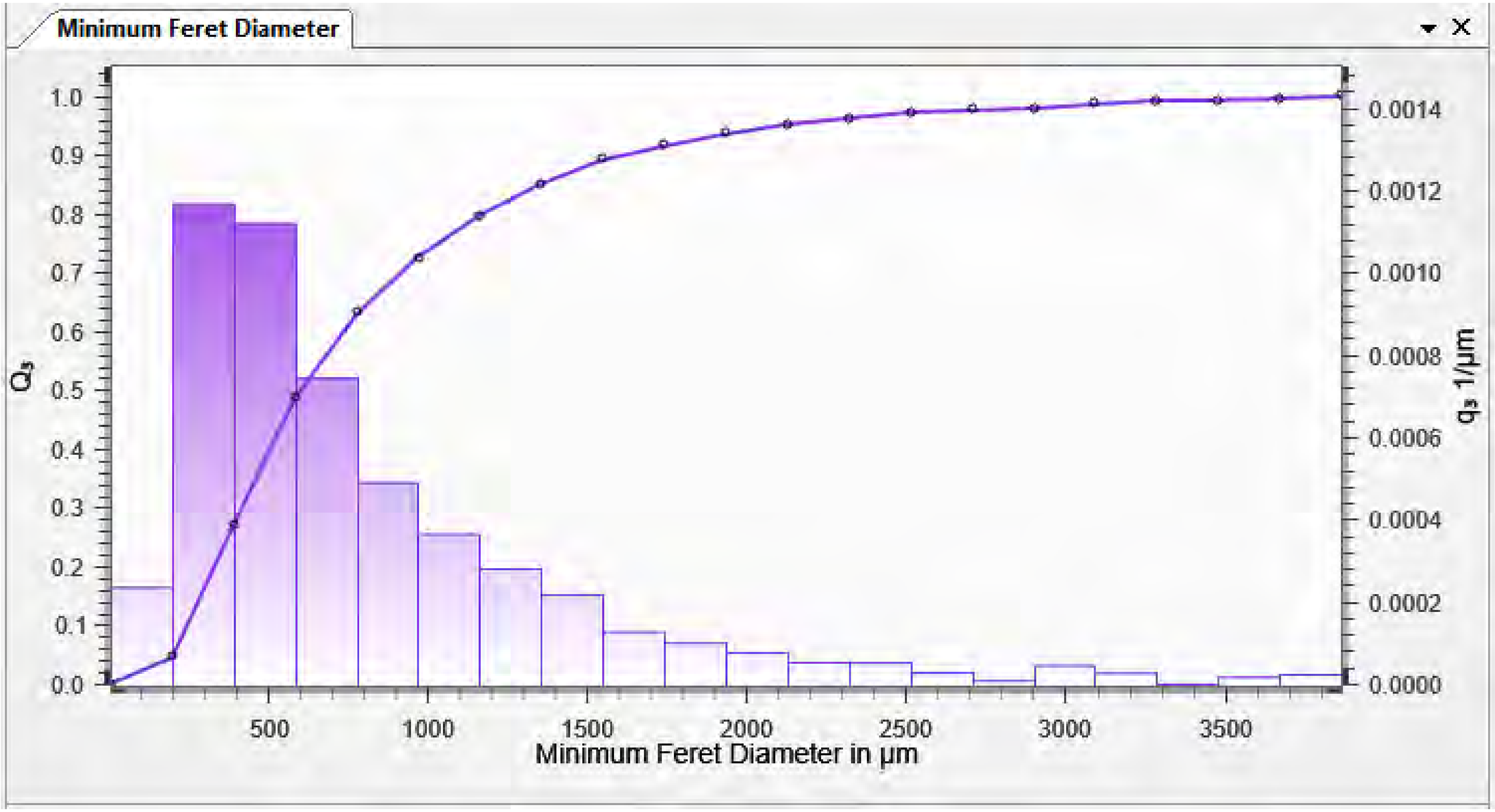
Each point is a measured particle. You can see the small number of larger particles leading to the relatively noisier data for shape in larger size classes.

This is two parameters for each particle. In truth there are more…



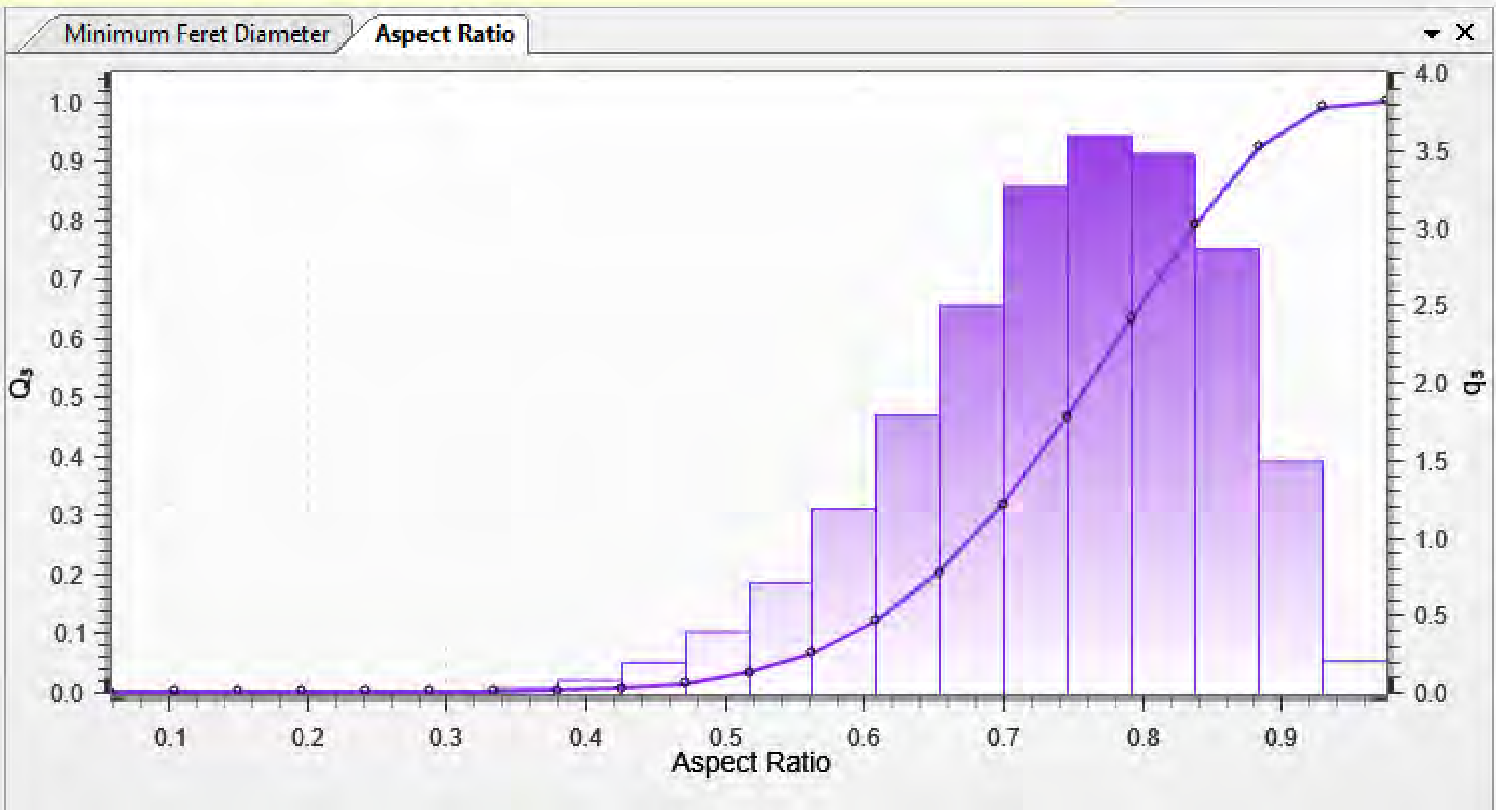
# Size distribution

Usual friend: quantity as a function of size. Use this to extract parameters like D10, D50, D90, mean size.

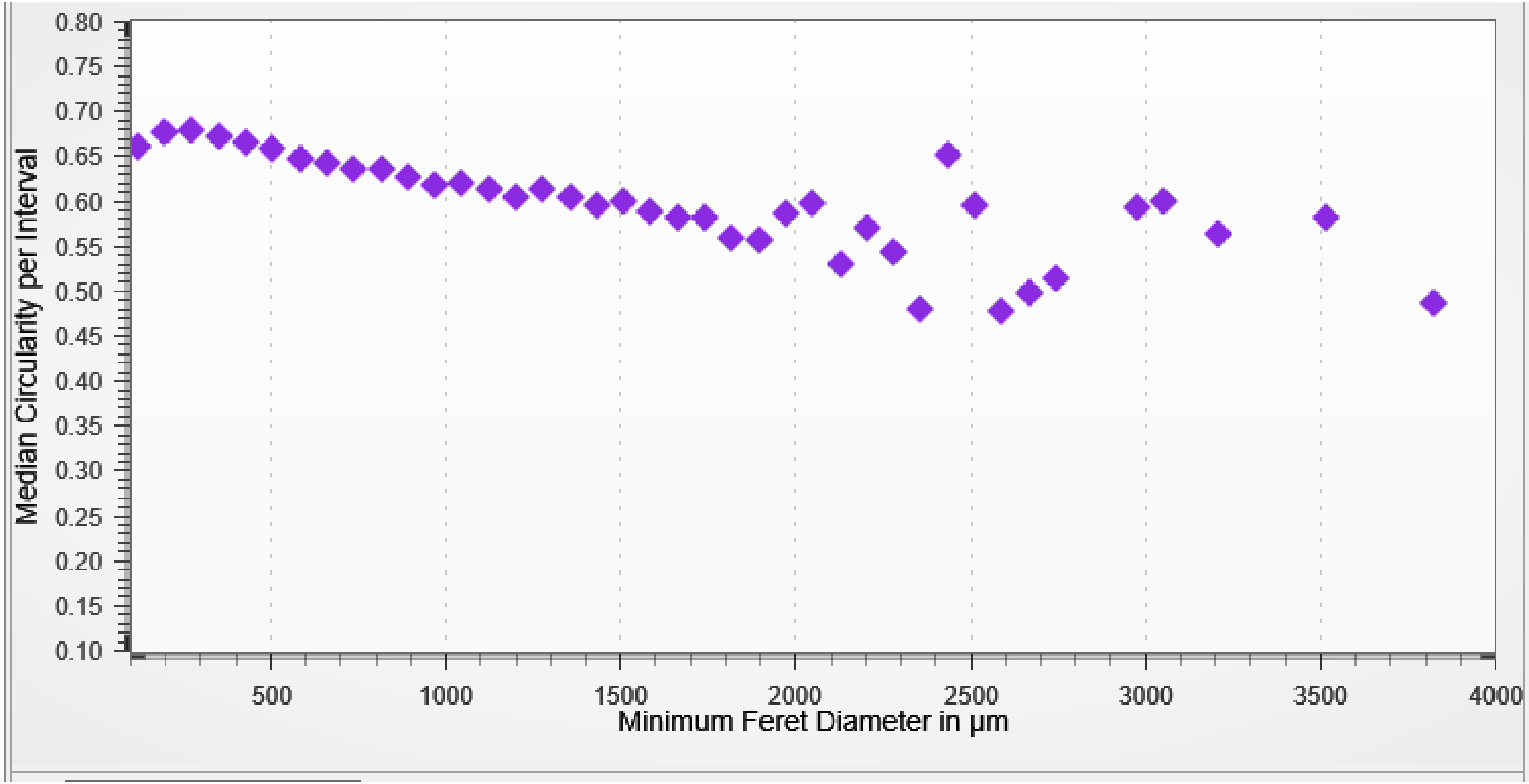



| | N (valid) | d10,3 in µm | d50,3 in µm | d90,3 in µm |
|---|---|---|---|---|
| Sample 2 - River Sand 035... | 405581 (0) | 258 | 601 | 1598 |

# Shape distribution



# Size vs shape

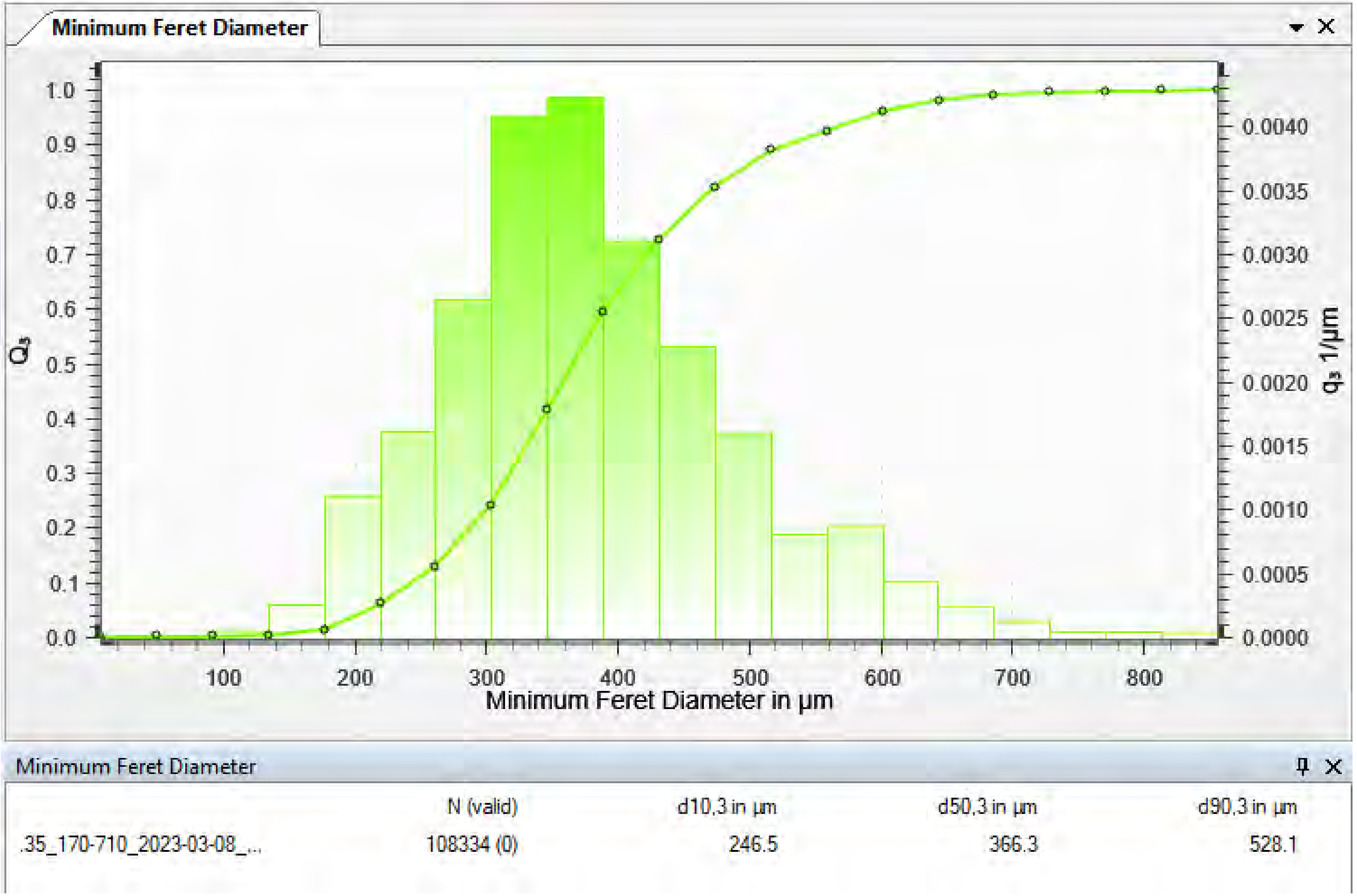



Plot is of circularity as a function of size

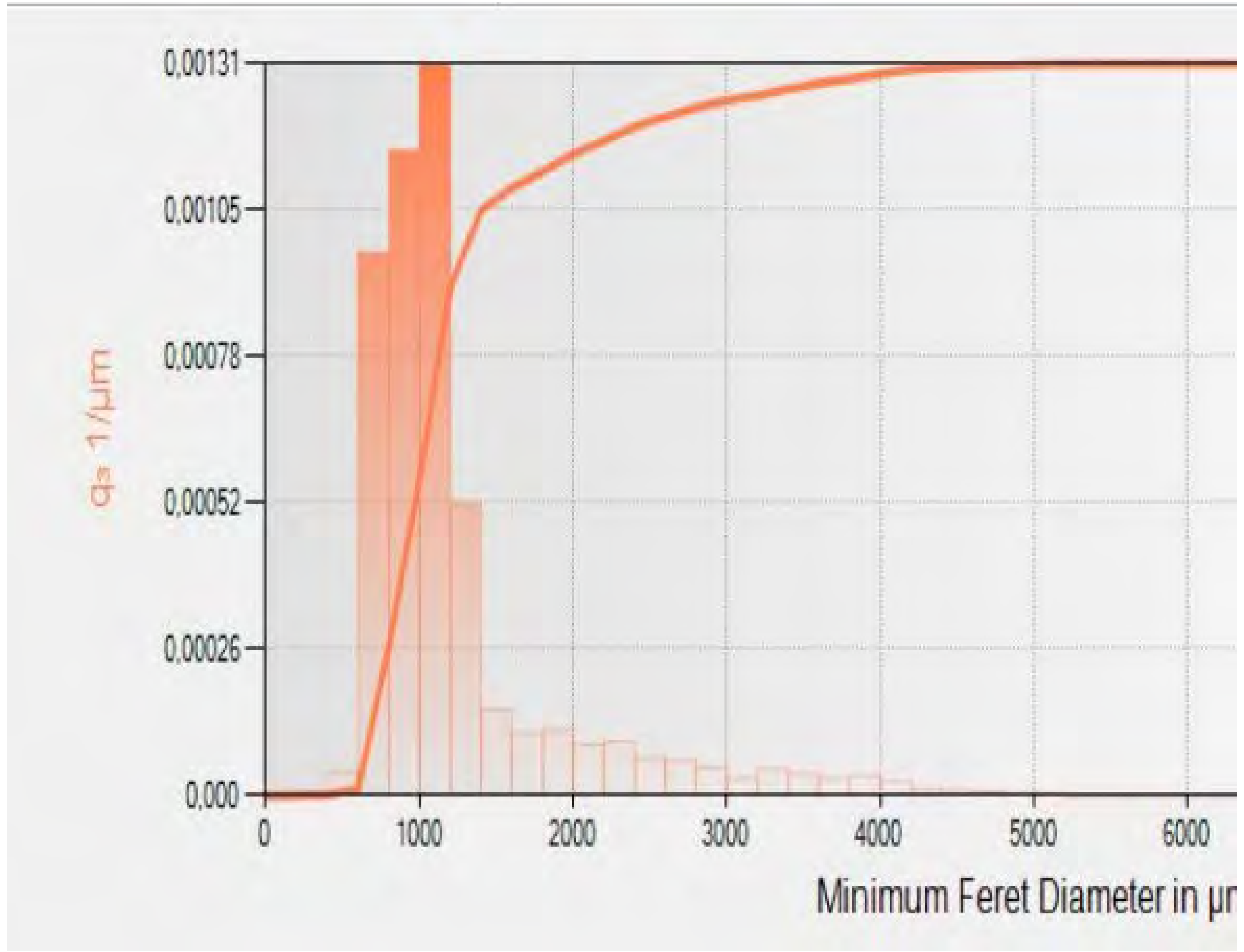
# Accuracy with standard

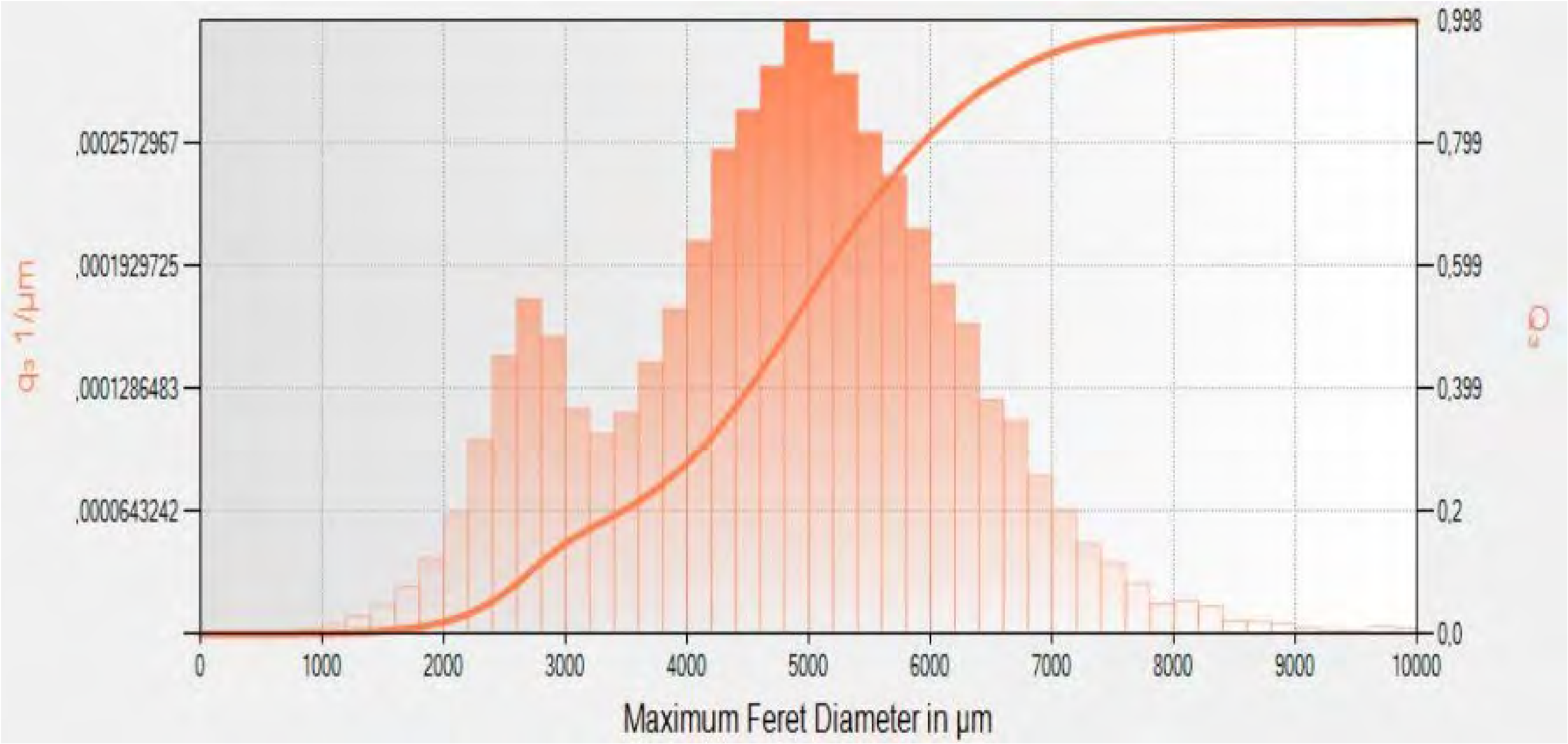
## Whitehouse IA025

| | D10 microns | D50 microns | D90 microns |
|---|---|---|---|
| Spec. | 254 | 364 | 520 |
| Uncert | 10 | 10 | 14 |
| Result | 246.5 | 366.3 | 528.1 |



# Seed mixture



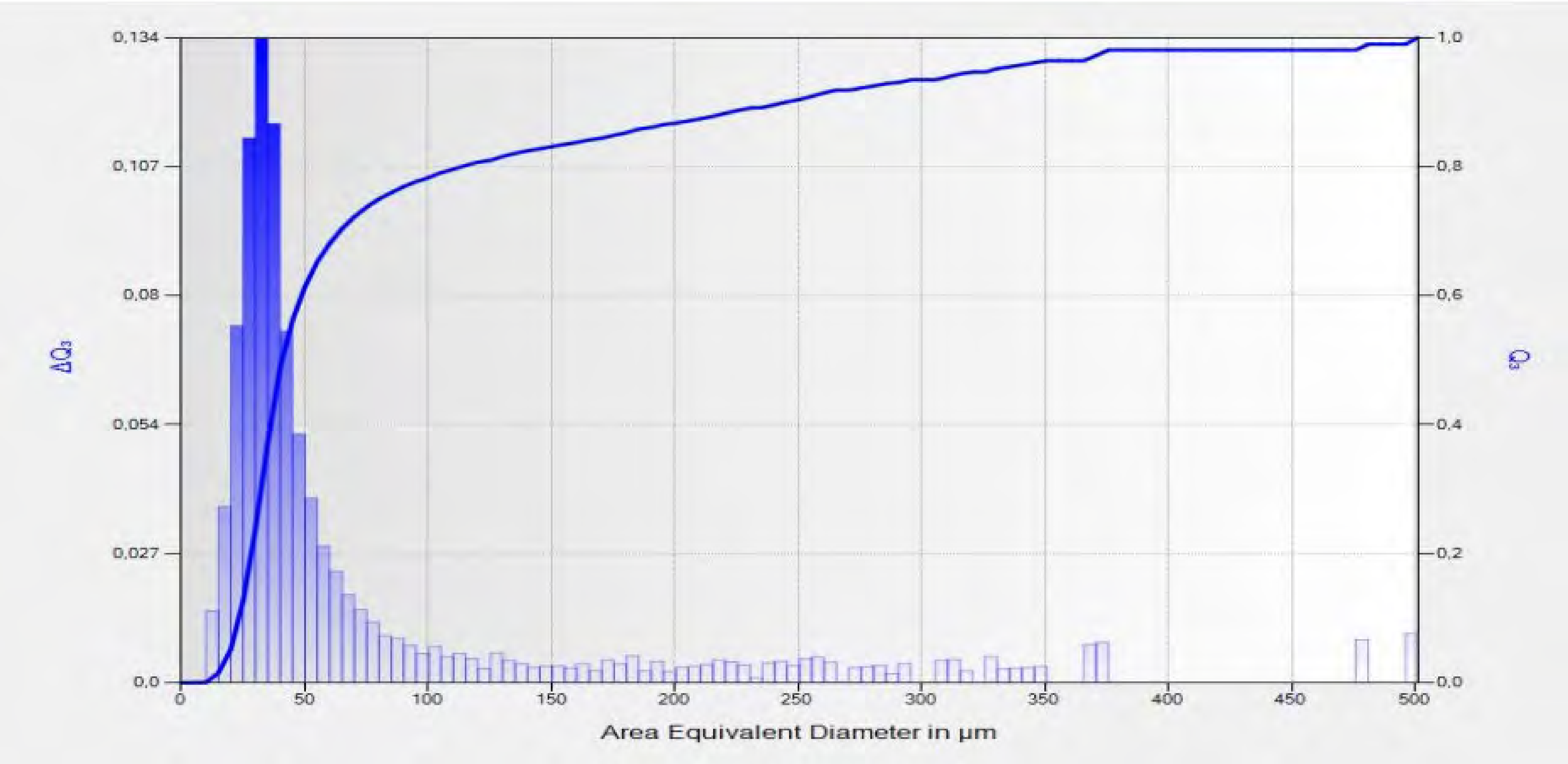




Note that looking at maximum Feret is a better way to differentiate between see types (or see mixture ratio).

Good repeatability either way.

| Particle size in µm for given Q3-Values (D-Values) | | D-Values | | |
|---|---|---|---|---|
| | | Q3 = 10 % | Q3 = 50 % | Q3 = 90 % |
| Maximum Feret Diameter | Seed_01 | 2,708 µm | 4,832 µm | 6,57 µm |
| | Seed_02 | 2,673 µm | 4,787 µm | 6,455 µm |
| Minimum Feret Diameter | Seed_01 | 0,717 µm | 1,076 µm | 2,868 µm |
| | Seed_02 | 0,71 µm | 1,049 µm | 2,498 µm |

# Ti64 metal powder

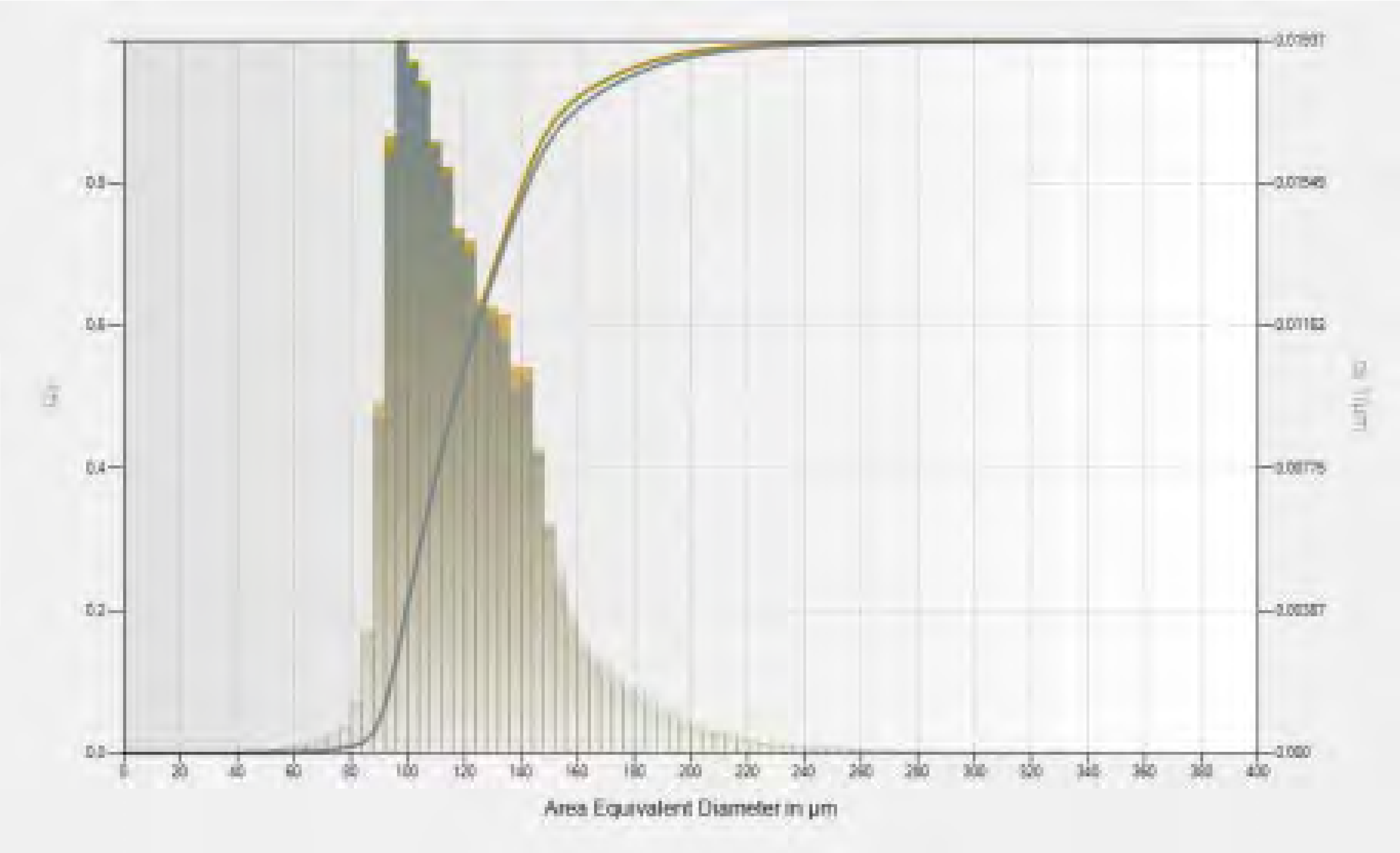



This is a metal powder used in additive manufacturing.

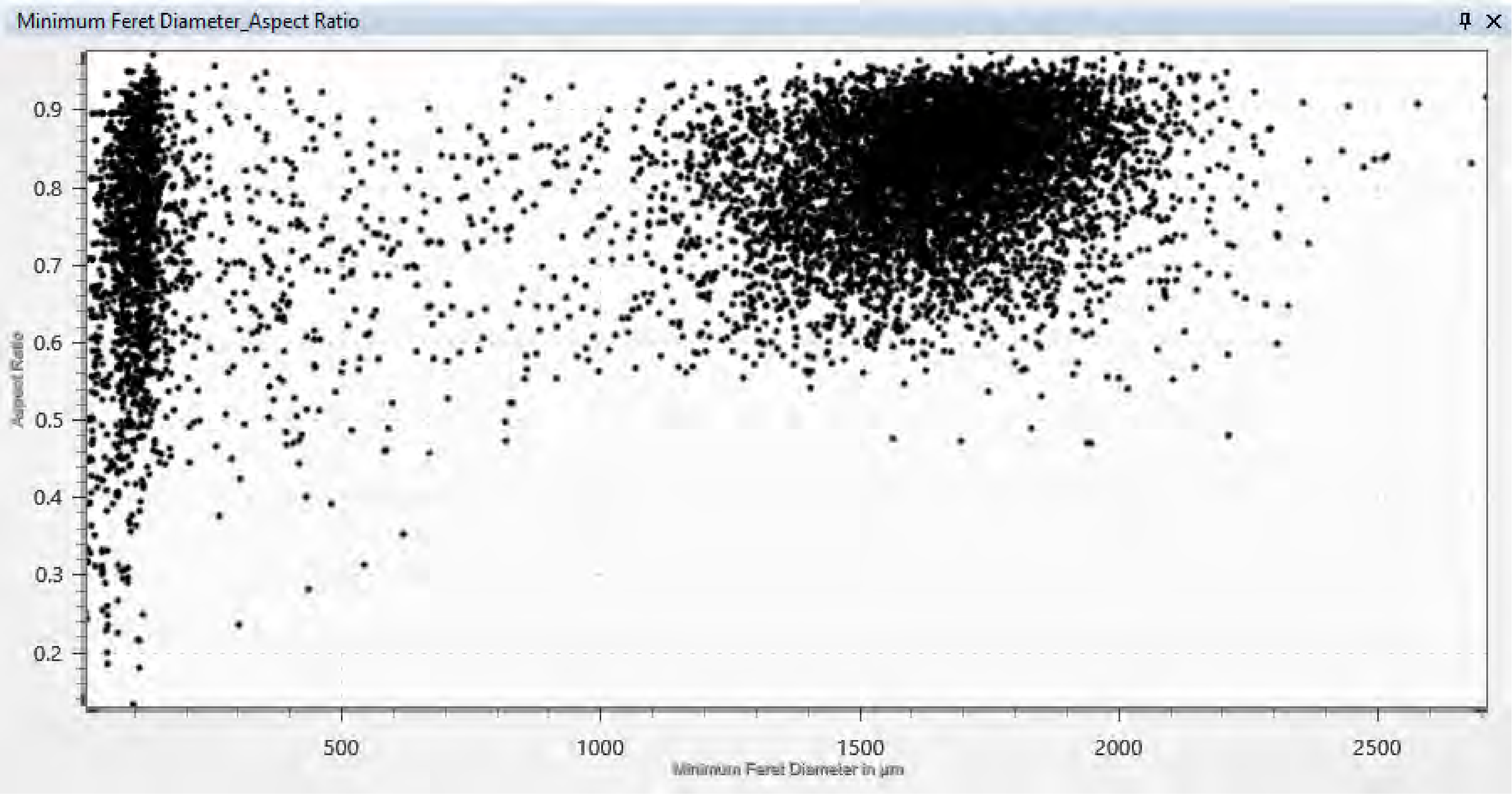
# Metal powder

Two repeats, note higher size and broader size distribution.

| D-Values | | |
|---|---|---|
| Q3 = 10 % | Q3 = 50 % | Q3 = 90 % |
| 93,9 µm | 116,8 µm | 154,7 µm |
| 93,7 µm | 117,4 µm | 159,2 µm |

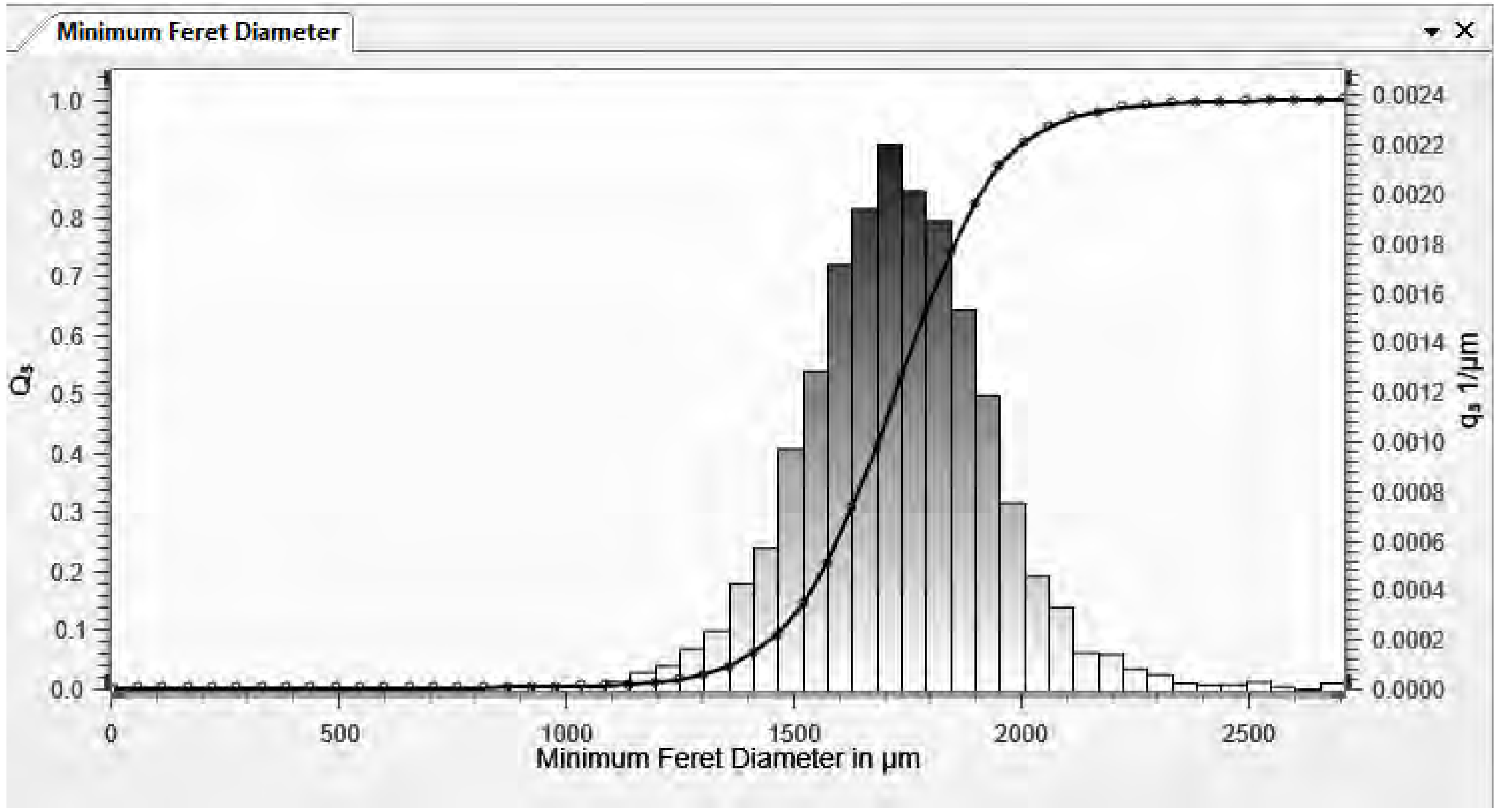


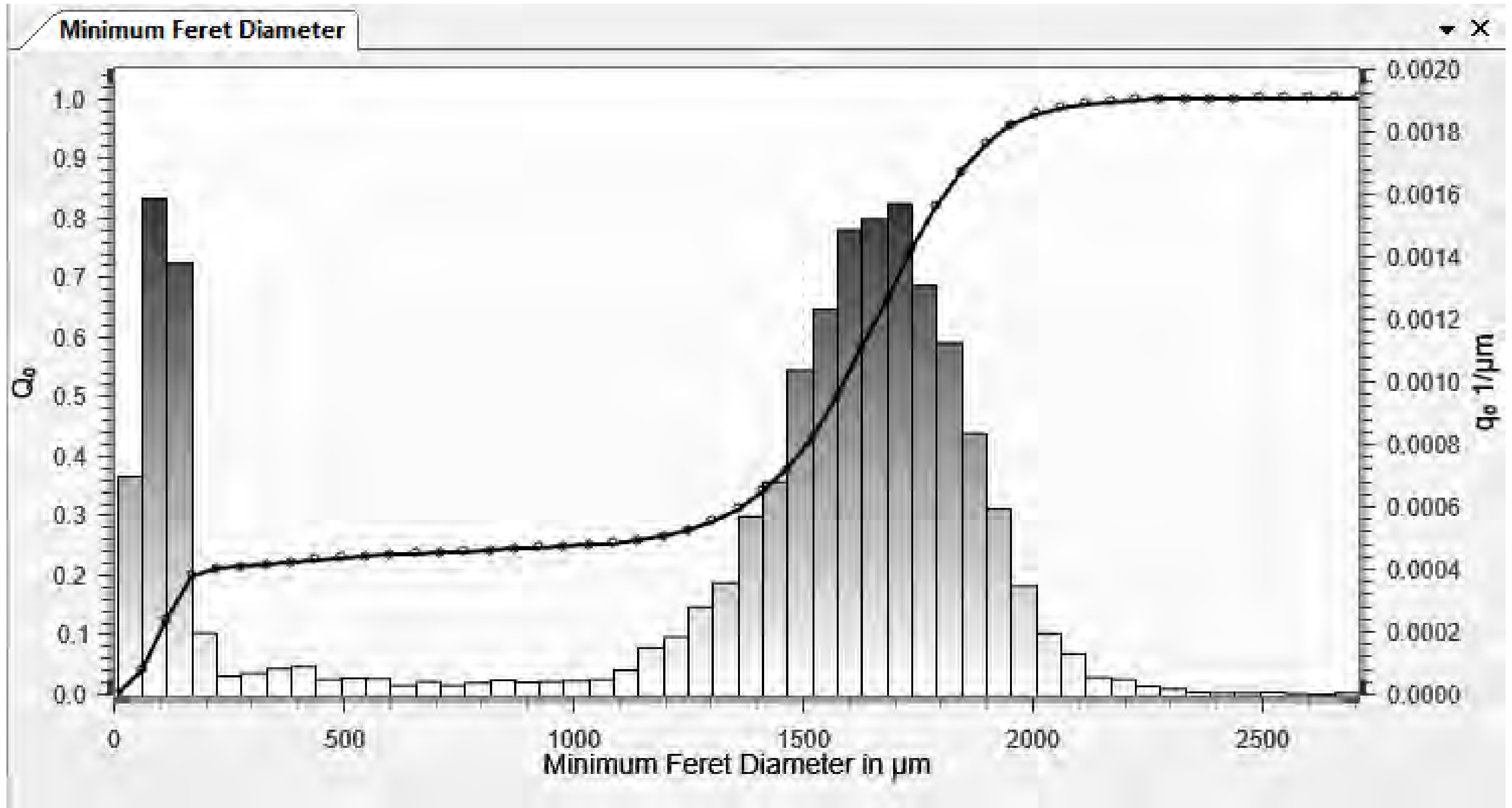
# Fertilizer




Kind of an interesting point cloud. There are two distinct populations (and each with a different shape distribution).

# Size distribution

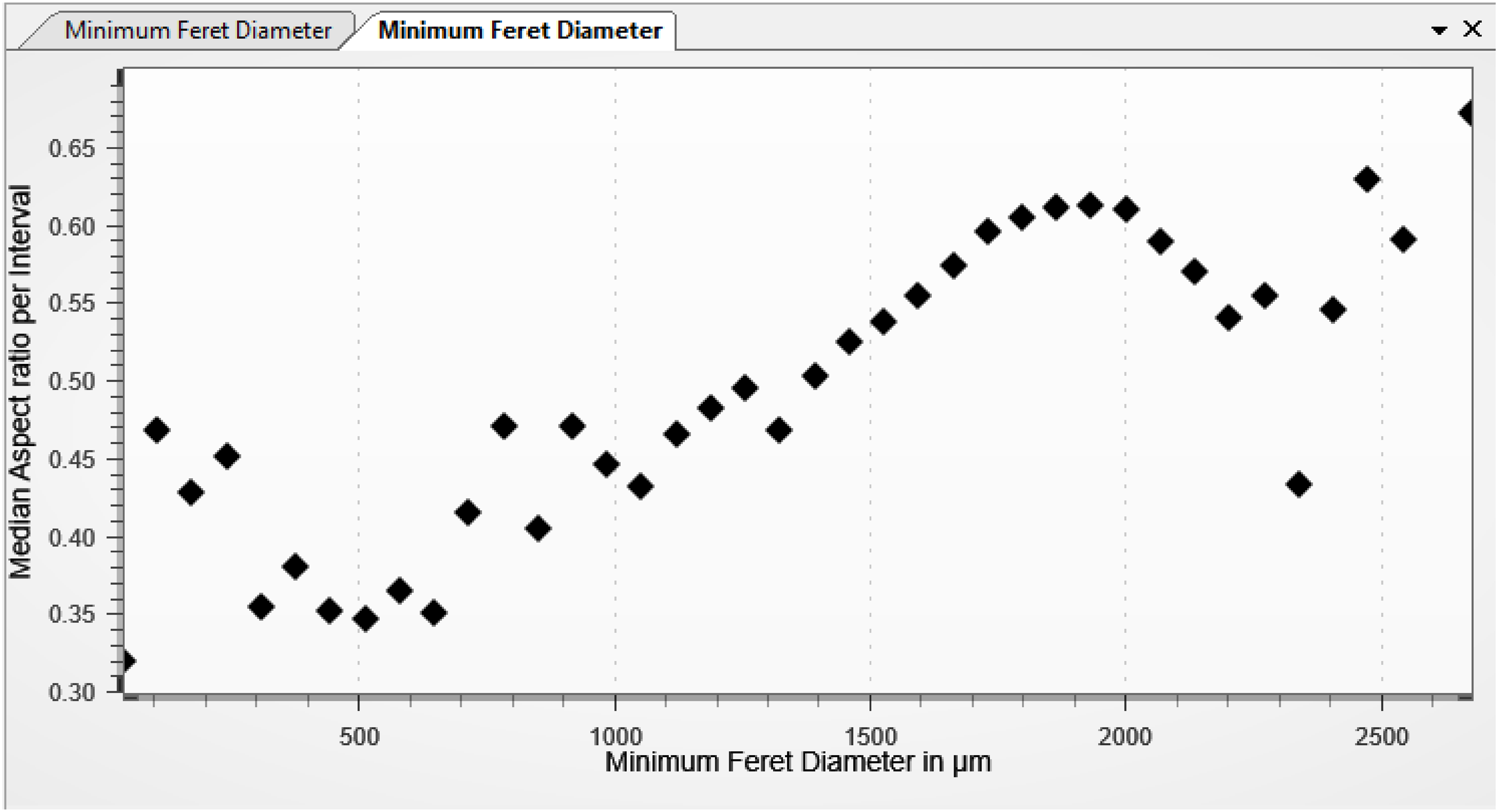



Same underlying data as a volume based and number-based distribution. Since small particles have a tiny tiny volume, they are not important in volume distributions.

This is the same underlying data shown two different ways. Use number based if fines are important.

# Shape vs size




Note that aspect ratio is a strong function of size.

# Common Questions

- How many particles?
- Minimum Particle Size
- Maximum Particle Size
- Accuracy

# <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 $\pm$ 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

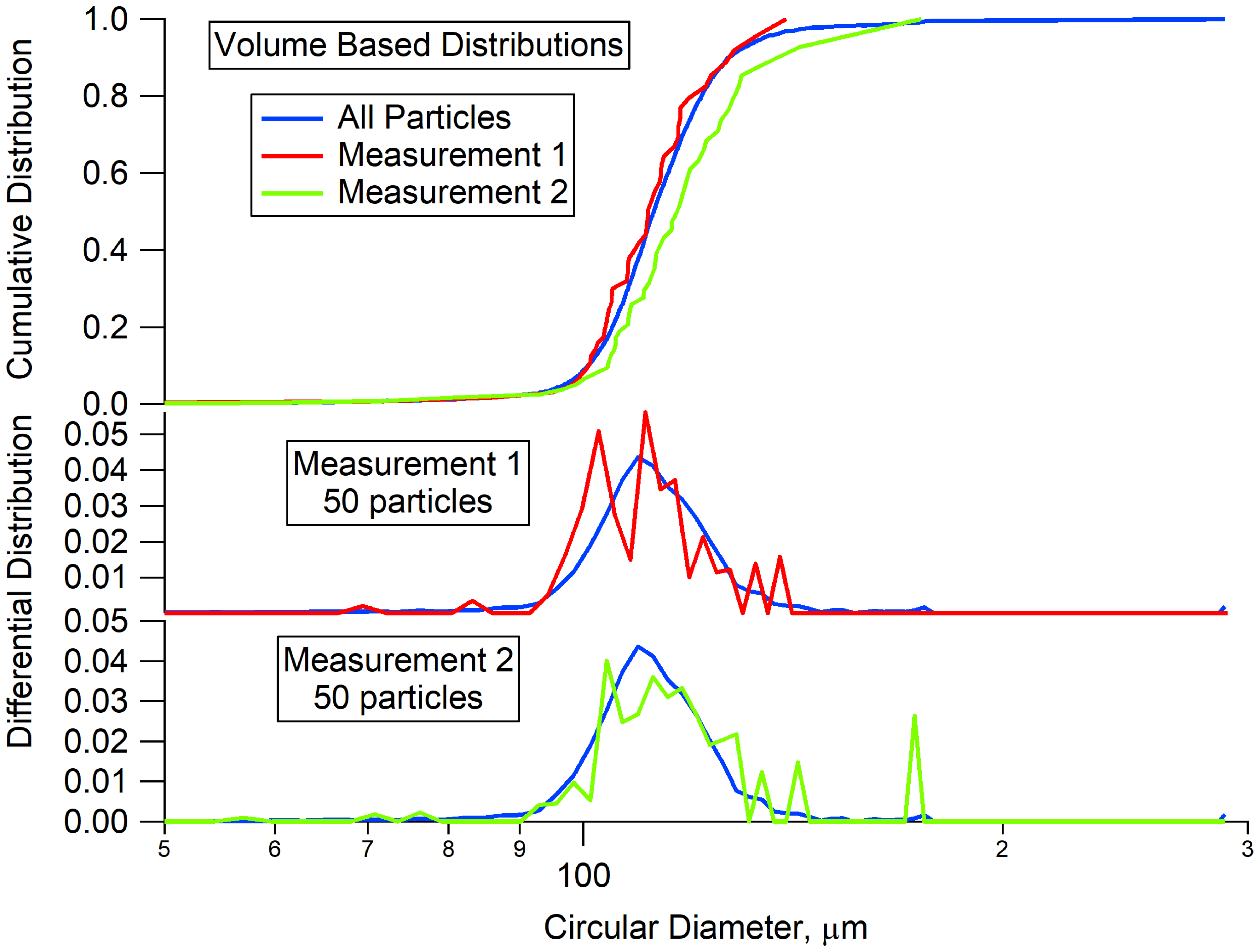
$$\sqrt{s^2\left(\frac{n}{\chi_a^2}\right)} < \sigma < \sqrt{s^2\left(\frac{n}{\chi_b^2}\right)}$$

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

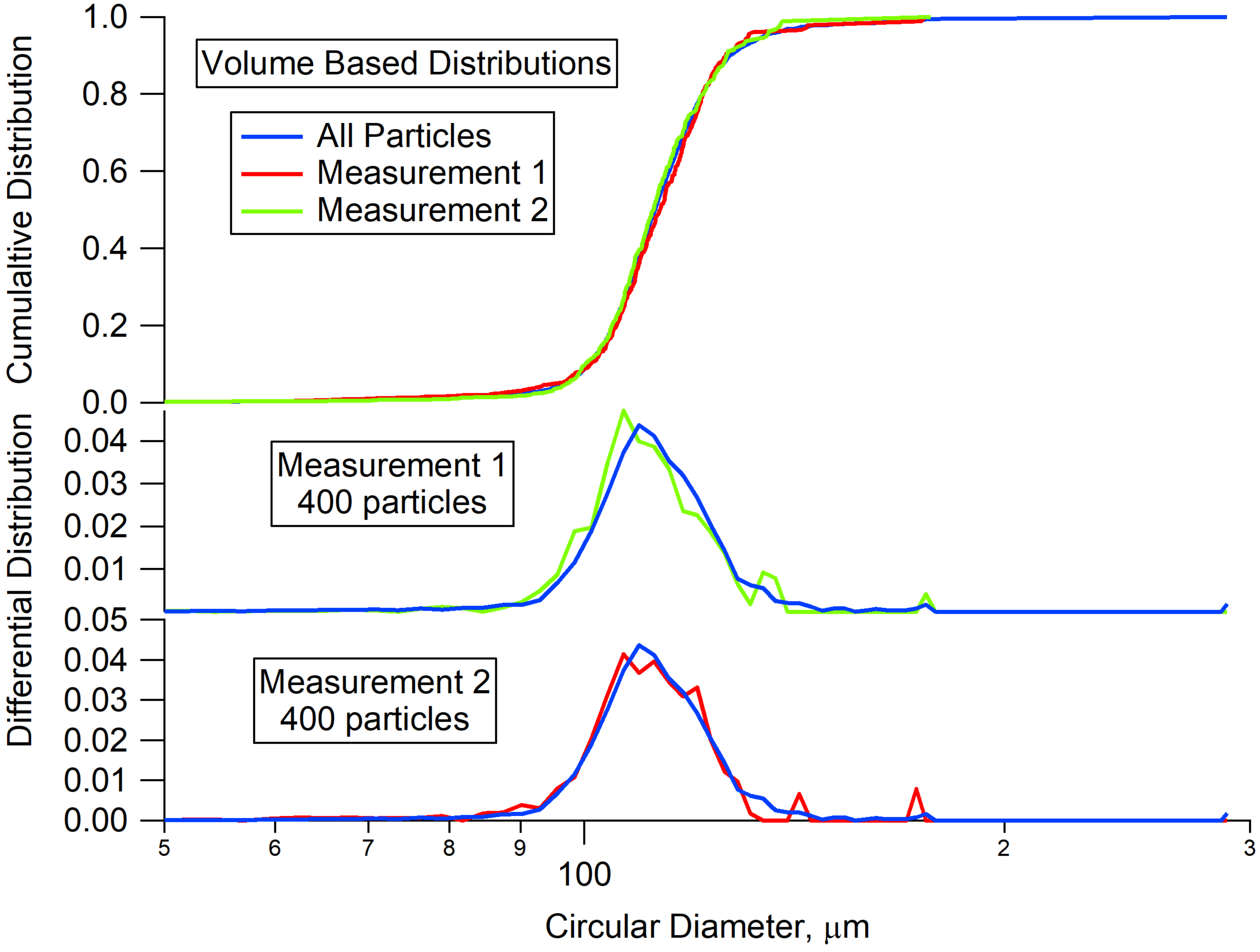
These limits are asymmetric around the standard deviation.

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

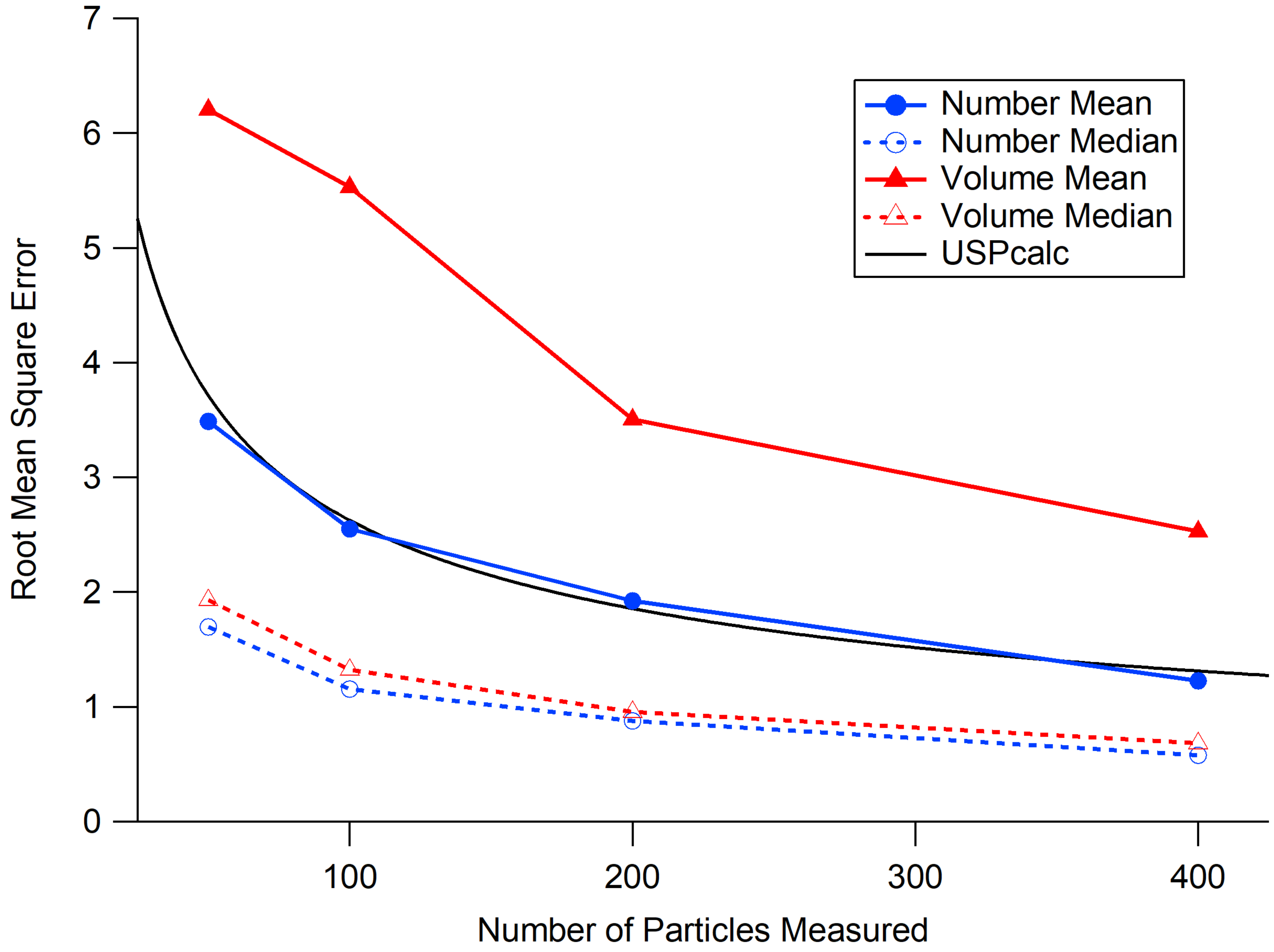
# 50 particles



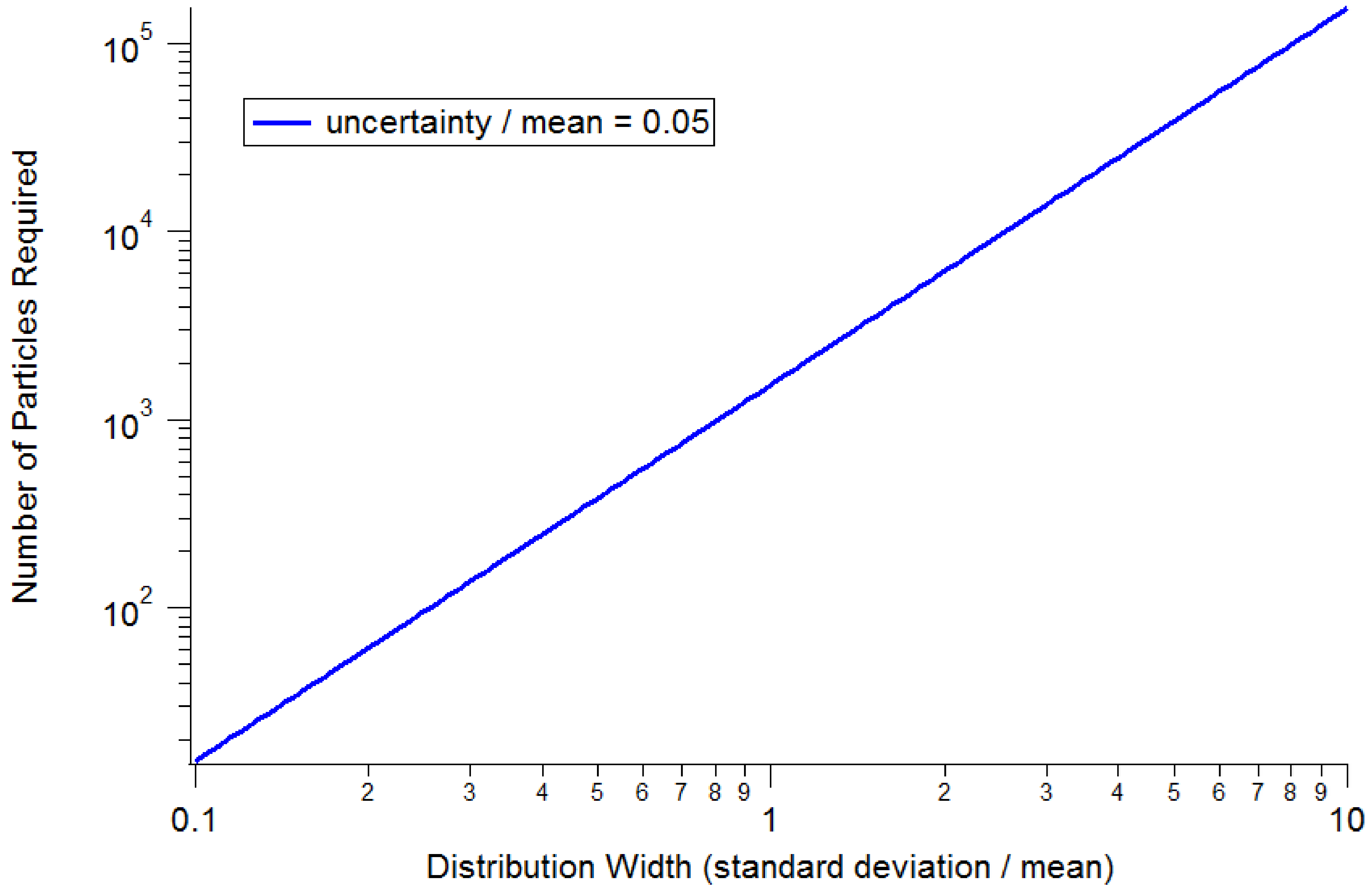
# 400 particles



# 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.

# Concluding Comment

- Image Analysis is good for…
  - Replacing Sieves
  - Size
  - Shape
  - Supplementing other techniques

- Watch out for…
  - Sample preparation
  - Sampling technique (representative aliquote)
  - Material behavior
  - Measure enough particles


# Thank you!

Send us a chat or email us: **Labinfo@horiba.com**

# Q&A