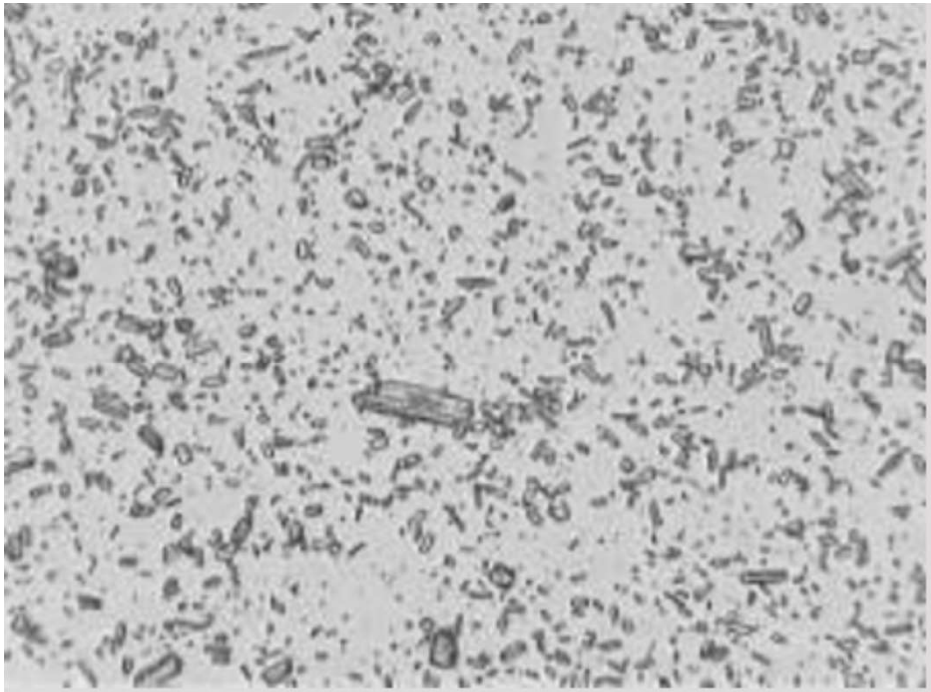
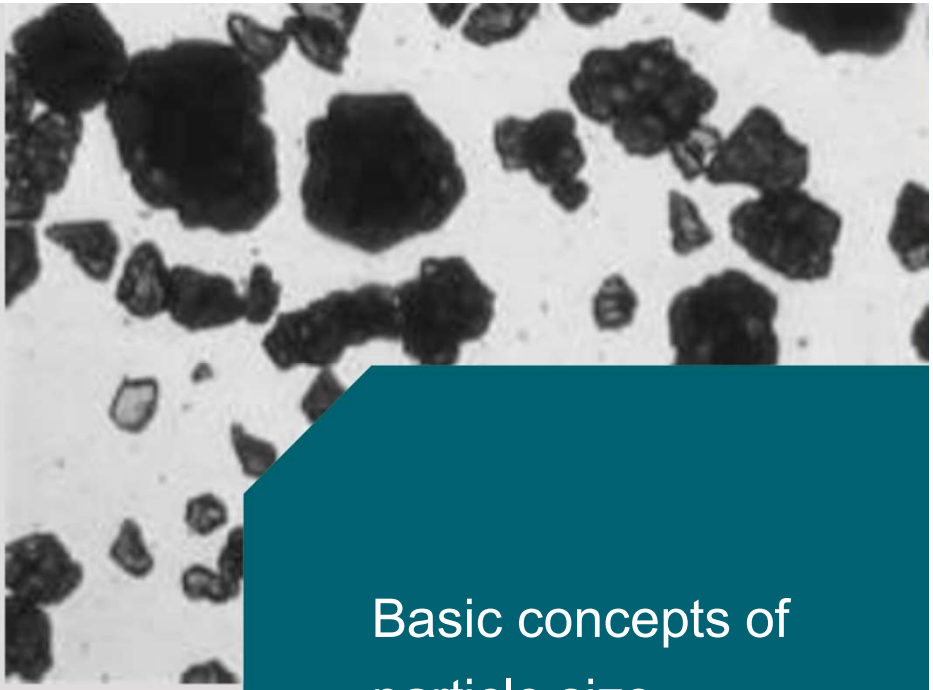
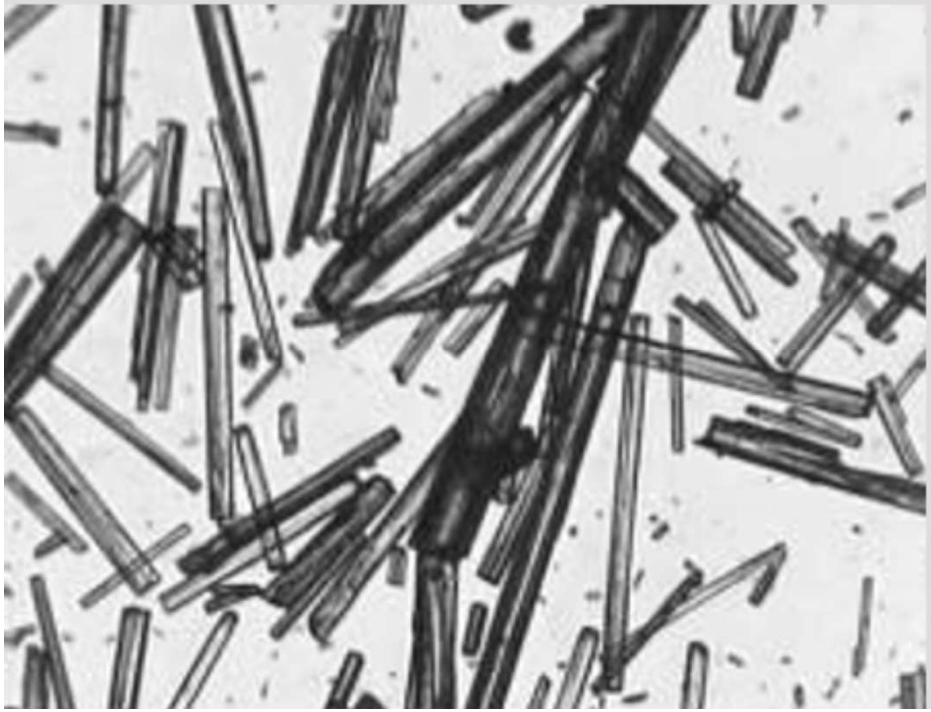
Malvern Panalytical

Mastersizer 3000 Customer Training Course

Part 1: Basic Principles and Data Quality







Basic concepts of particle size

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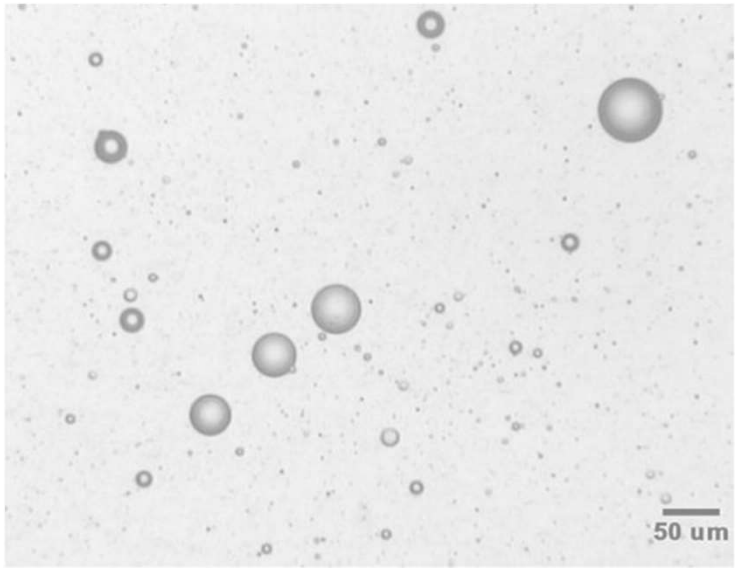
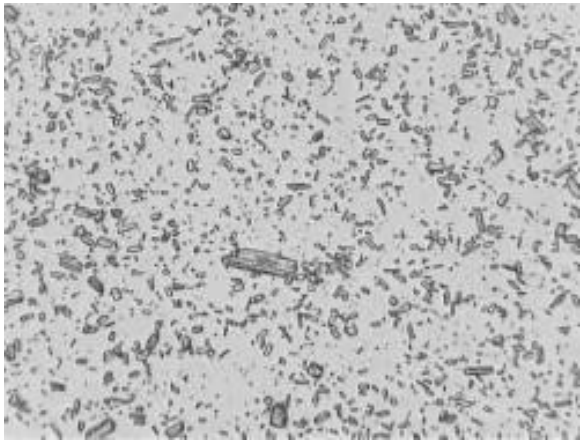
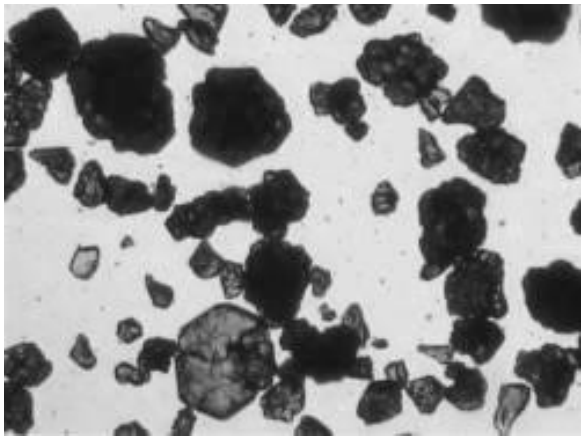
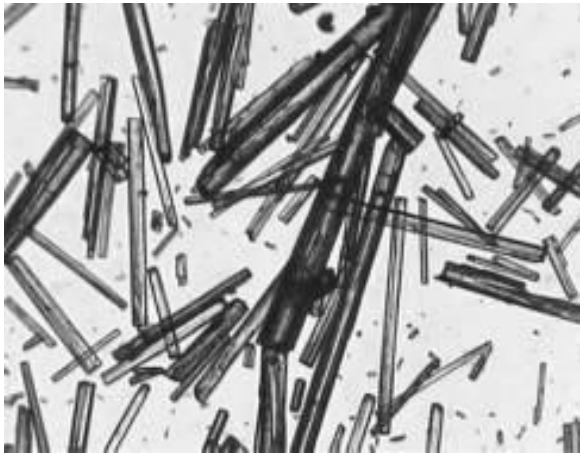
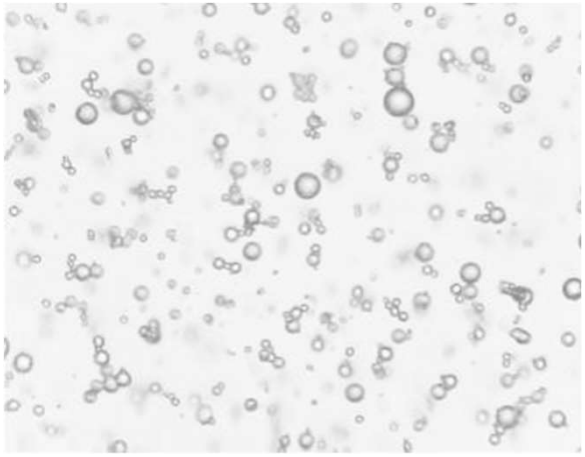
What do we mean by a particle?

- A particle can be described as a discrete sub-portion of a substance, e.g.
  - solid particles
  - gas bubbles
  - or liquid droplets



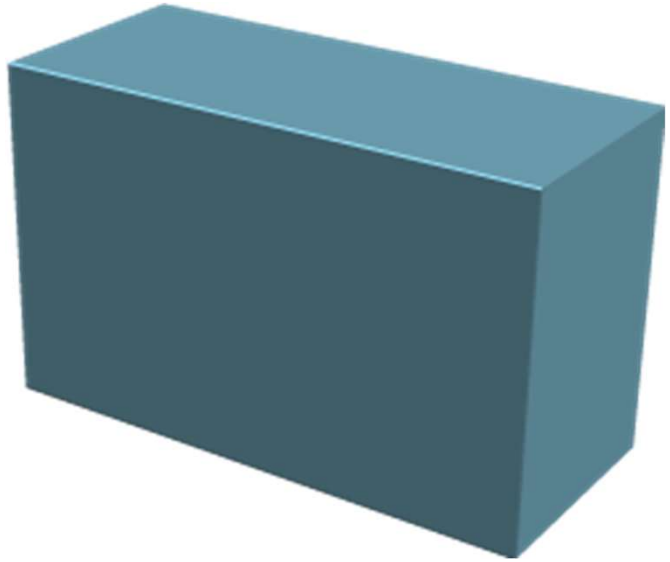
- Laser diffraction measures particles in the size range from nanometres to millimetres

Particles come in many different shapes (as well as sizes)

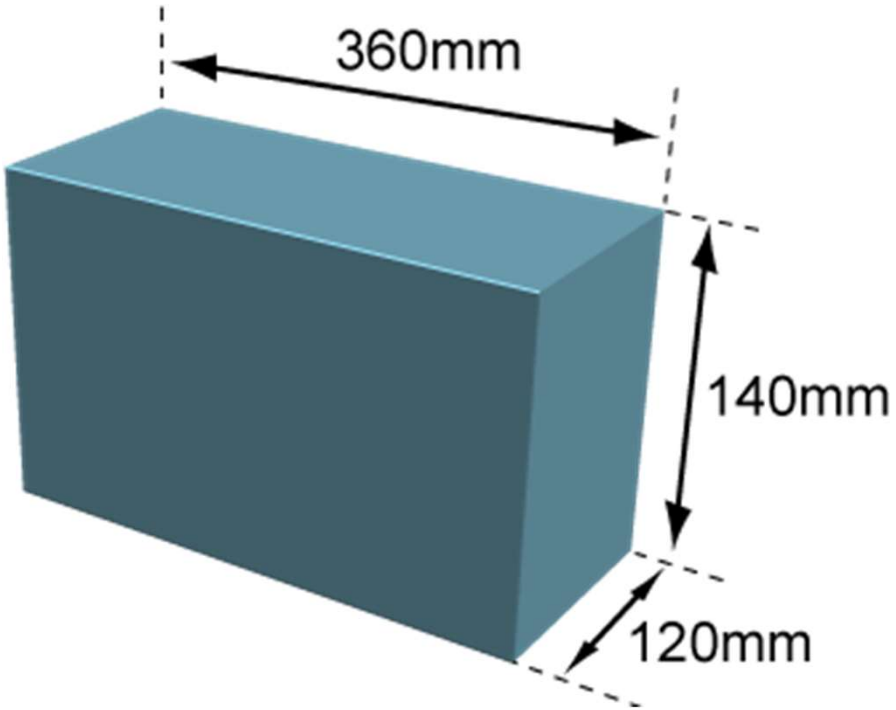
- How do we describe the size of these particles?

Basic concepts of particle sizing



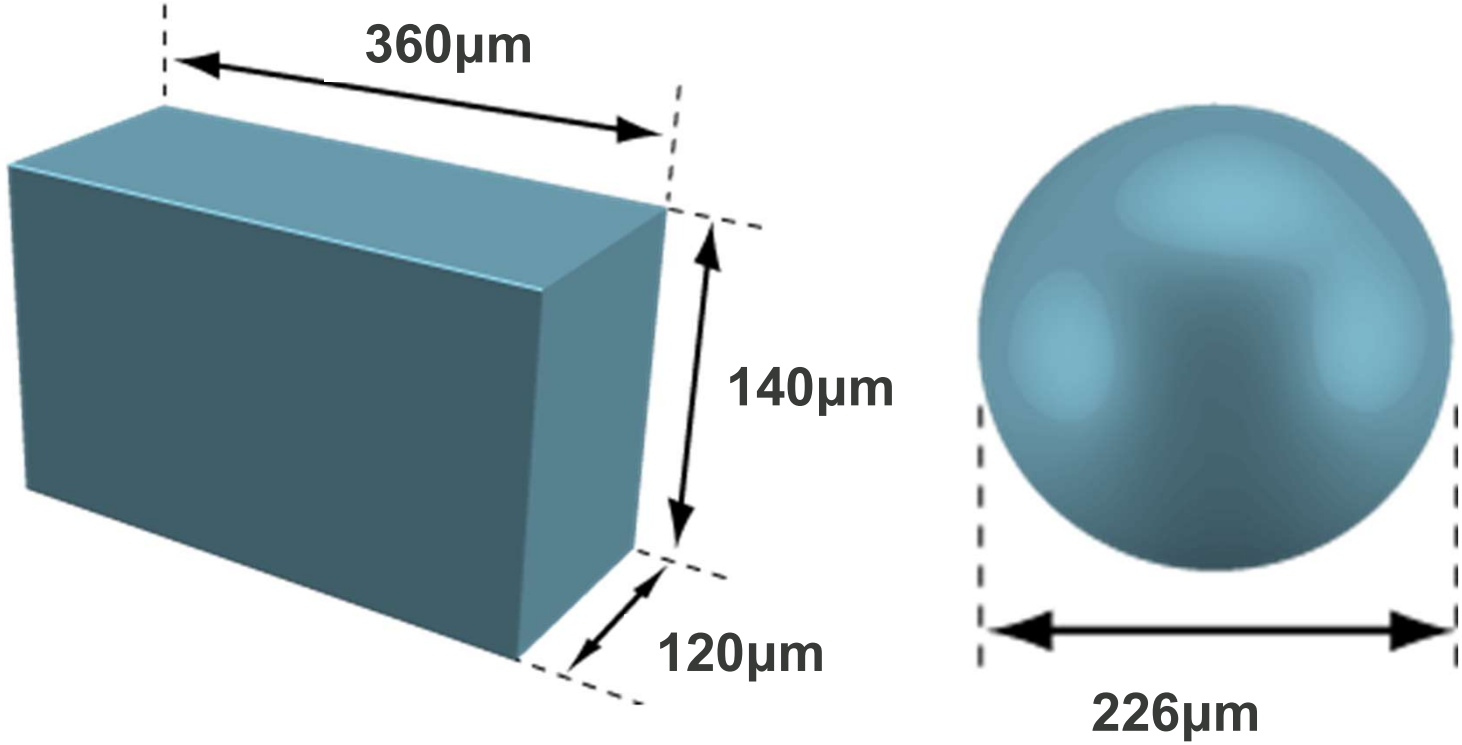
- You are given a regular-shaped object and a ruler and asked to give a one-number indication of its size
- What would your reply be ?

Basic concepts of particle sizing



- You may reply: “360x140x120mm”
  - Which might be correct but it is not **one** number.
  - It is not possible to describe the size of this 3-dimensional object with a single number

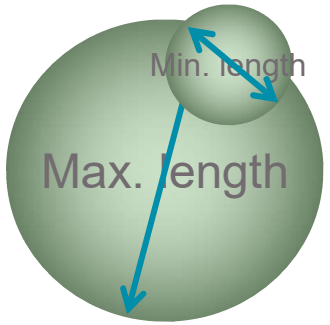
Concept of equivalent spherical diameters

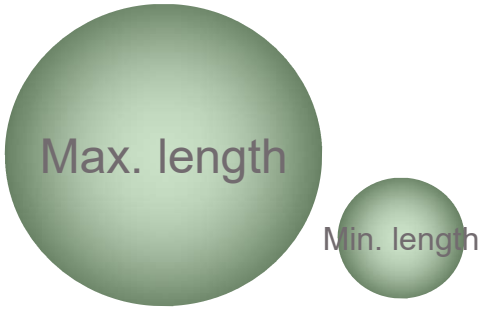


- The rectangular box has the same volume as a sphere of 226µm diameter.
  - The volume equivalent spherical diameter is 226µm

How do we describe the size of these particles

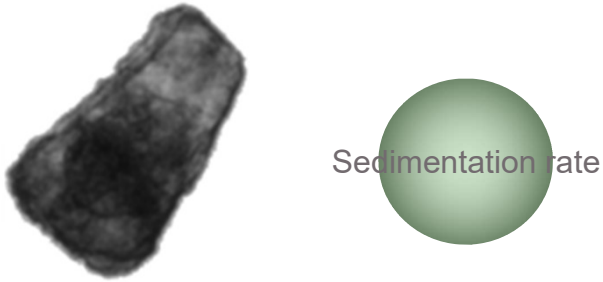
- Equivalent spheres
  - Maximum length
  - Minimum length

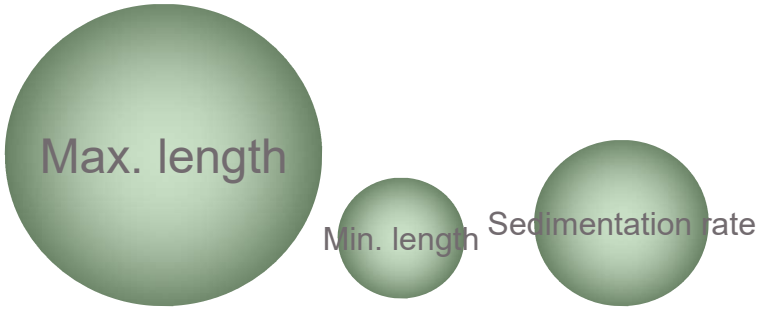




How do we describe the size of these particles

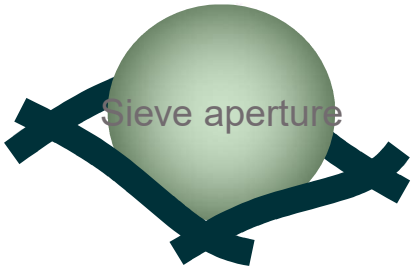
- Equivalent spheres
  - Maximum length
  - Minimum length
  - Sedimentation rate

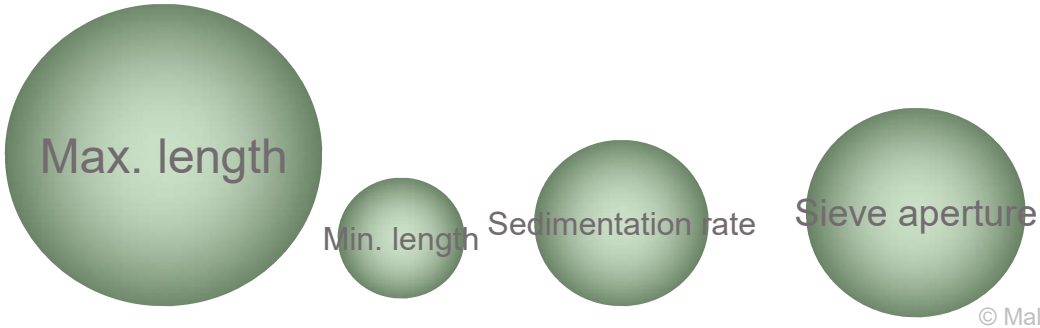




How do we describe the size of these particles

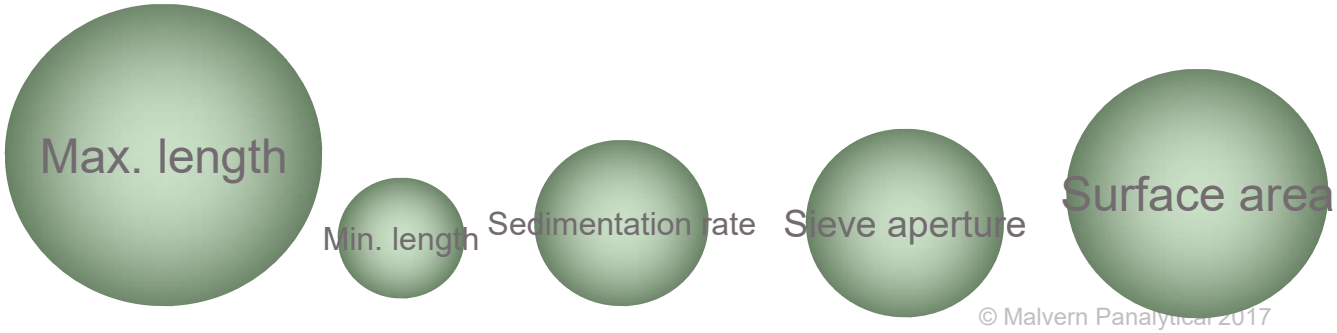
- Equivalent spheres
  - Maximum length
  - Minimum length
  - Sedimentation rate
  - Sieve aperture





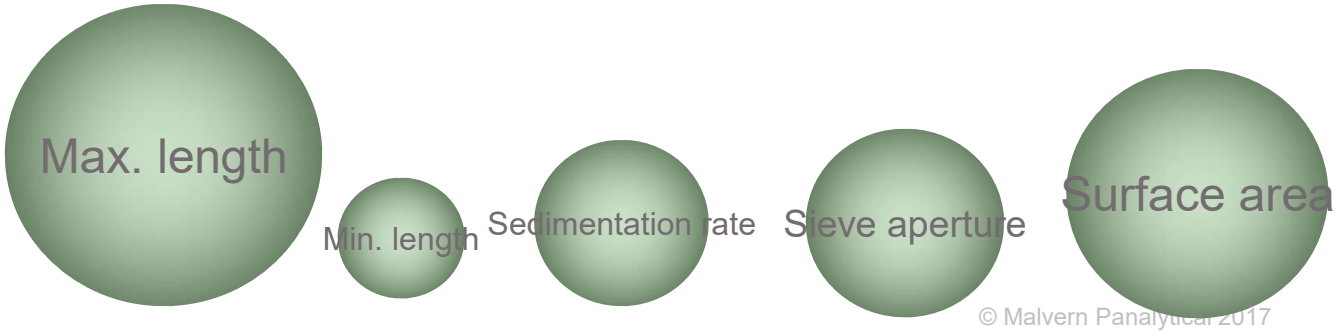
How do we describe the size of these particles

- Equivalent spheres
  - Maximum length
  - Minimum length
  - Sedimentation rate
  - Sieve aperture
  - Surface area



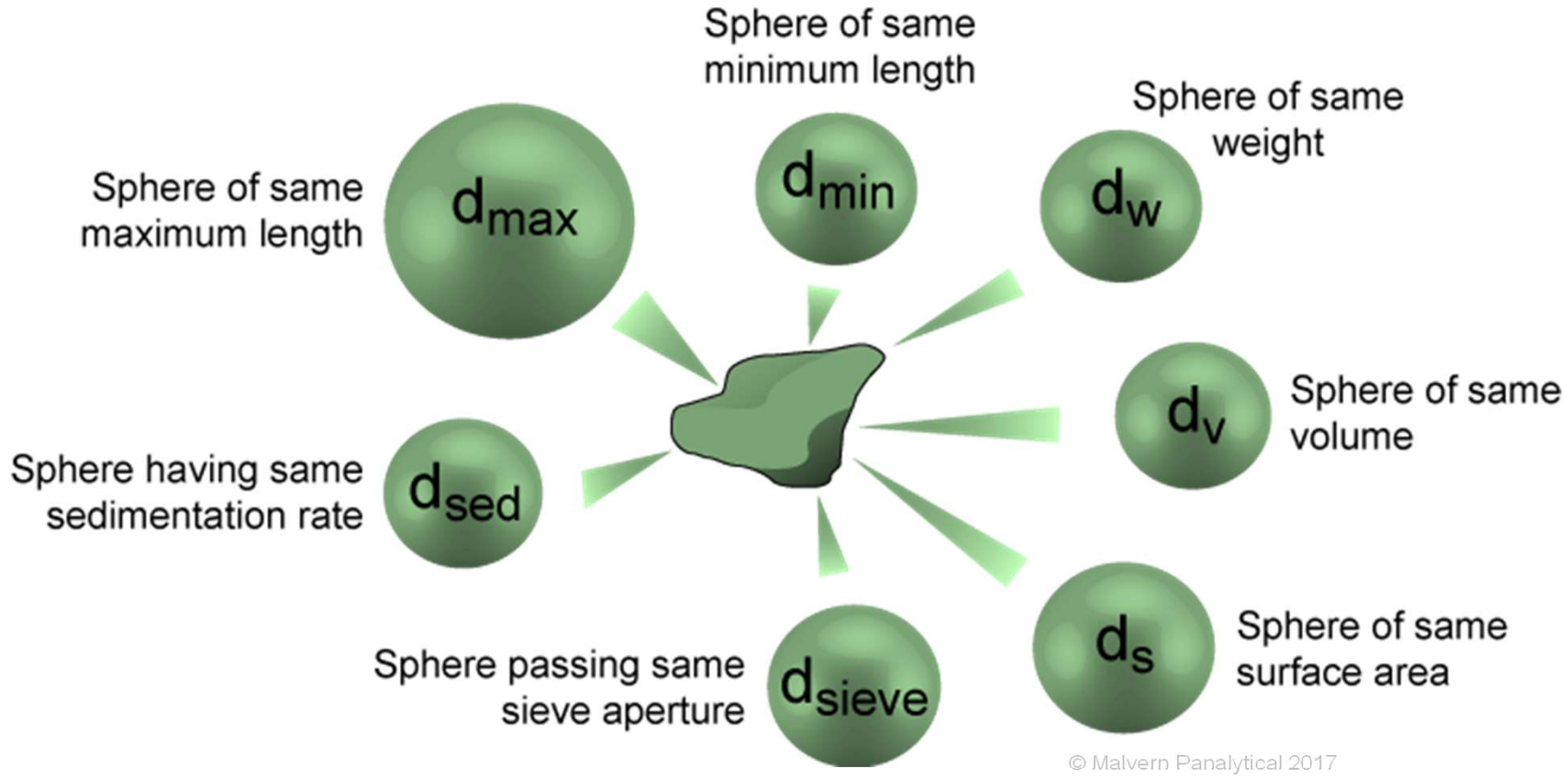
How do we describe the size of these particles

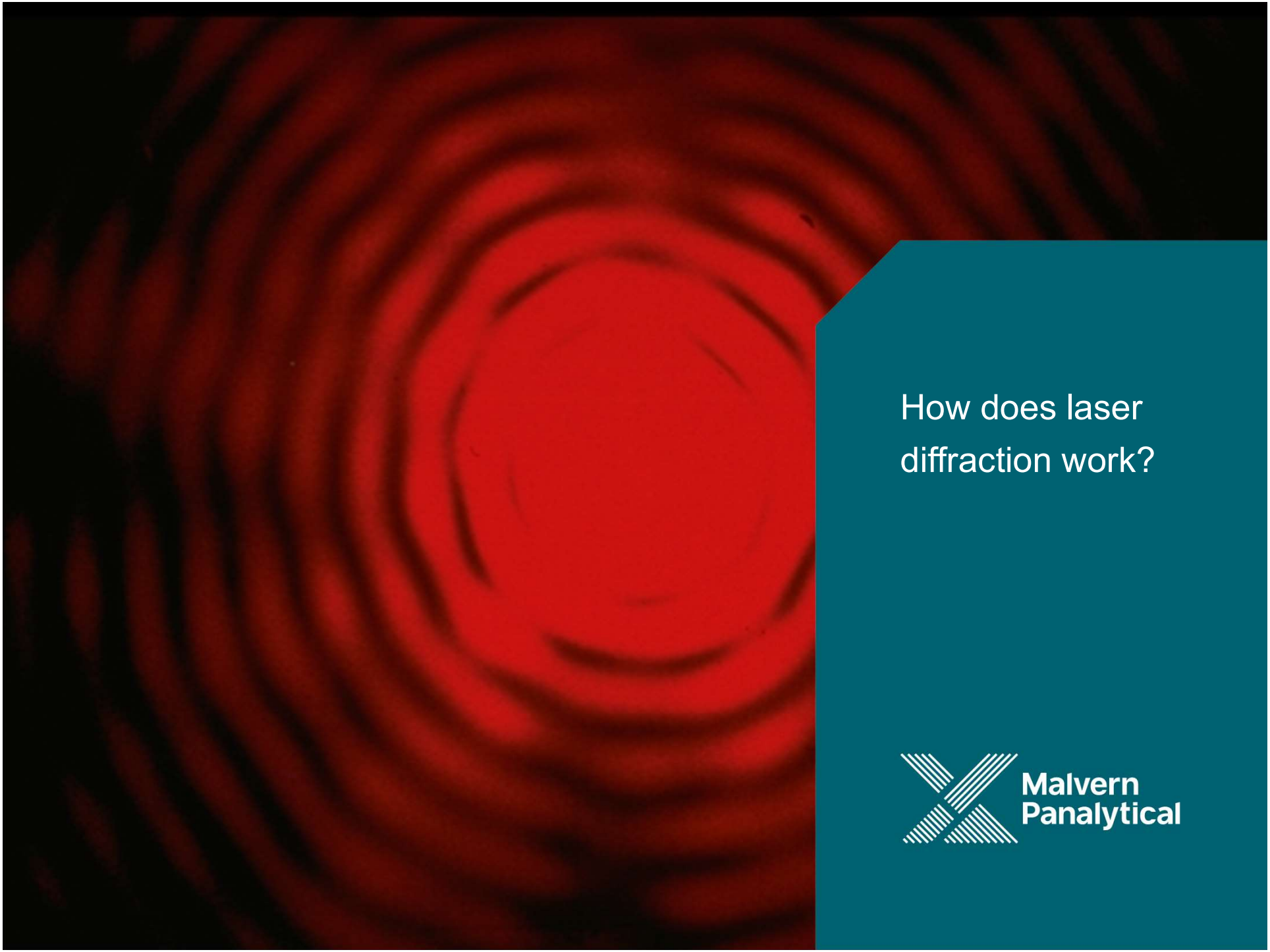
- Equivalent spheres
  - Maximum length
  - Minimum length
  - Sedimentation rate
  - Sieve aperture
  - Surface area
  - Volume



Concept of equivalent spherical diameters

- Different particle sizing techniques report different equivalent spherical diameters
  - Dependent on the physical property that is measured





How does laser diffraction work?

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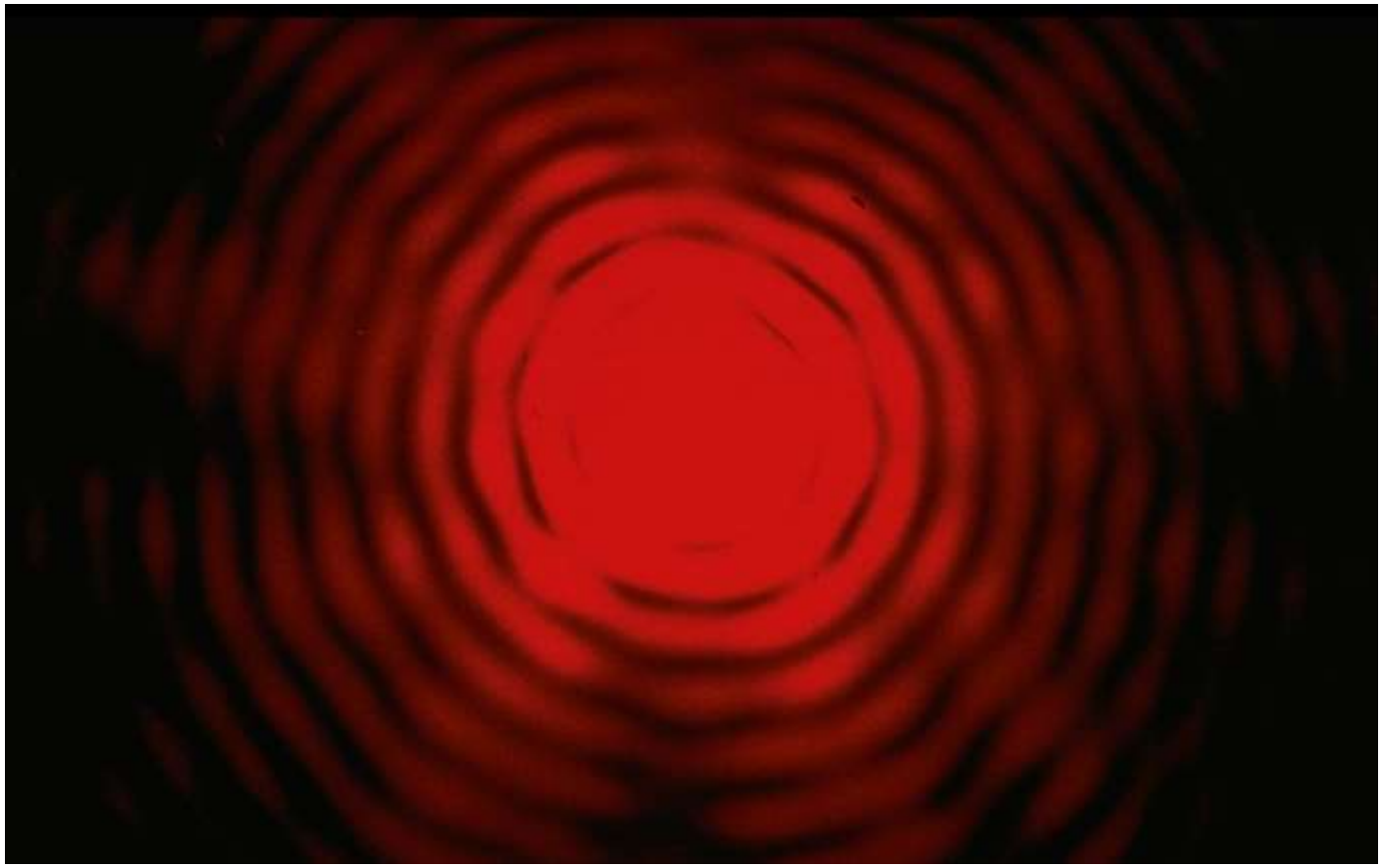
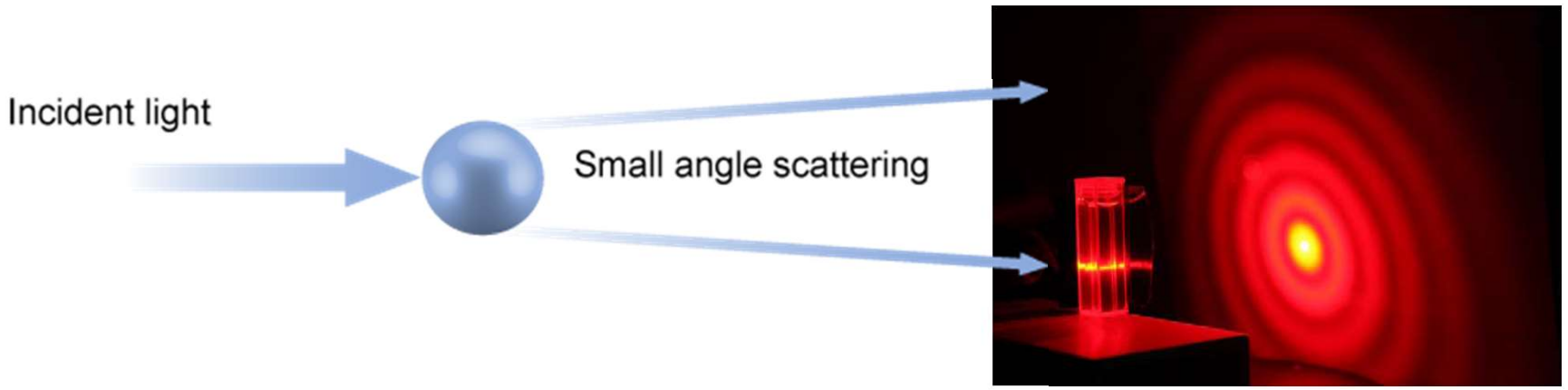
Laser Diffraction – The light scattering pattern from a group of particles



Laser diffraction: light scattering

Large particles



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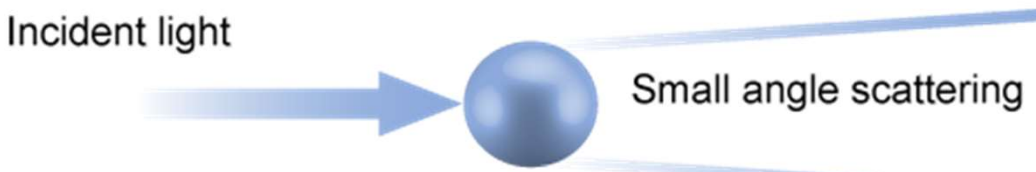
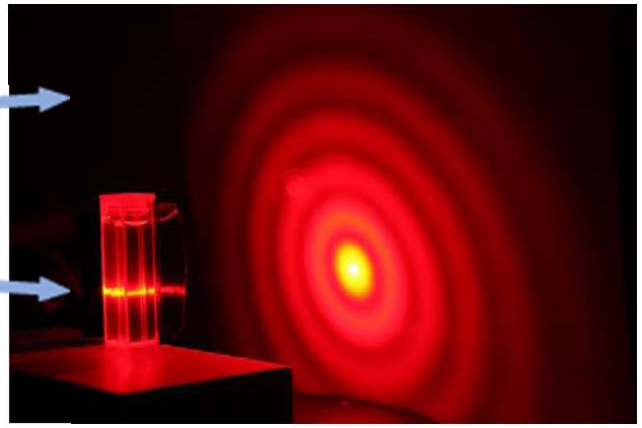
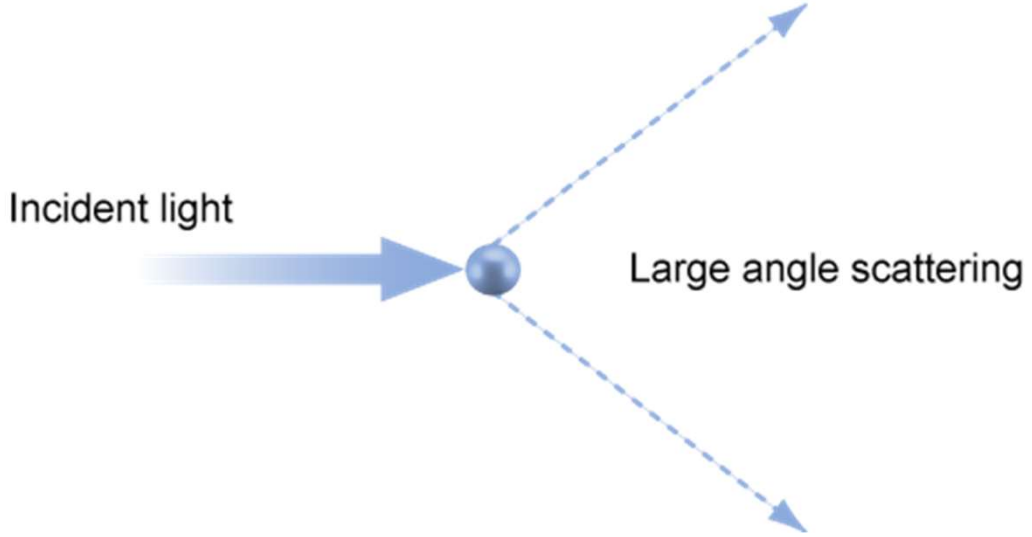
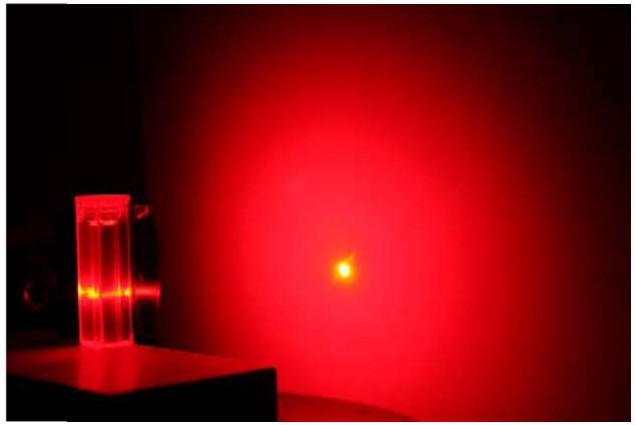
Laser diffraction: light scattering



Large particles







Submicron particles

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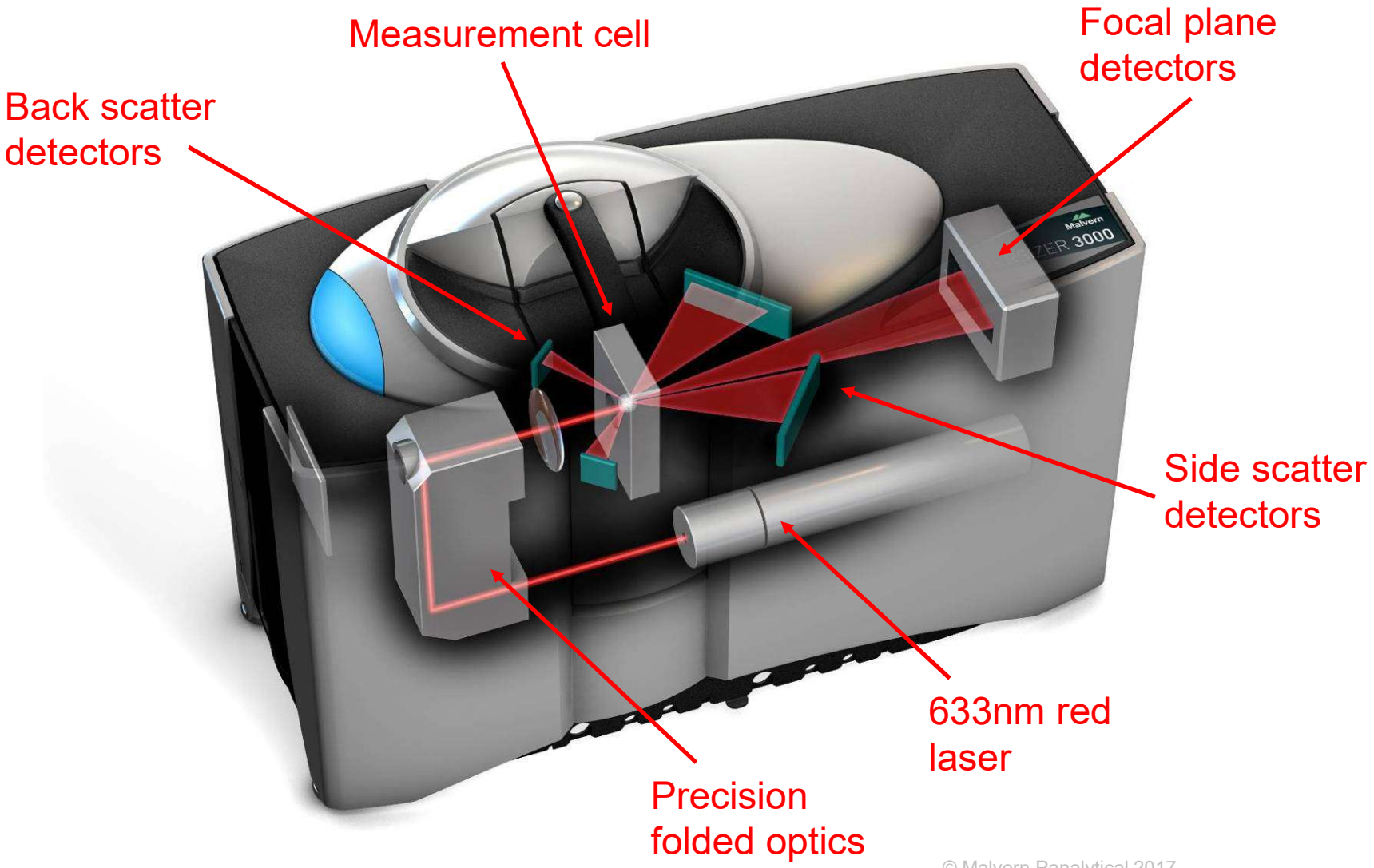
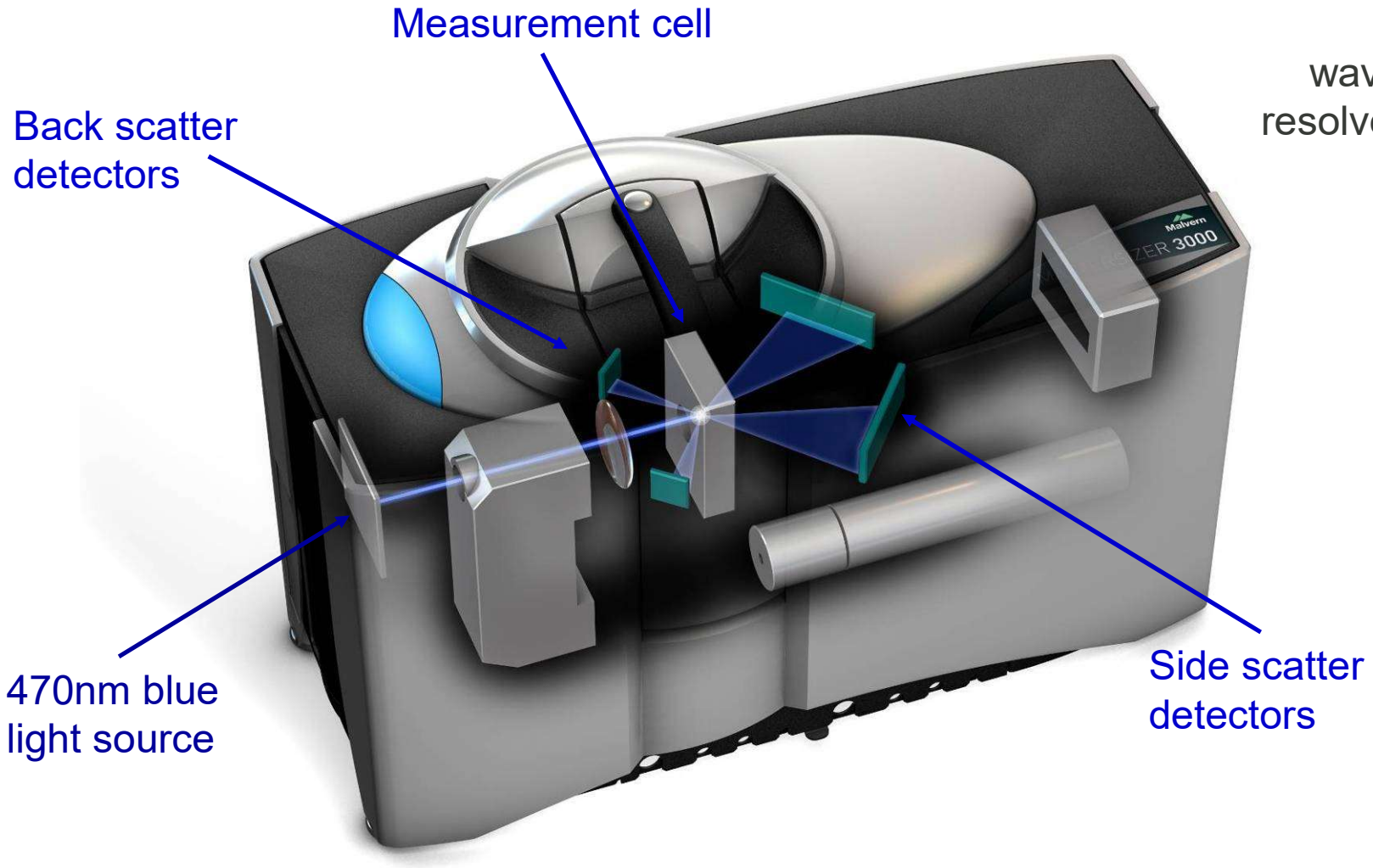
Mastersizer 3000 optics: red light measurements



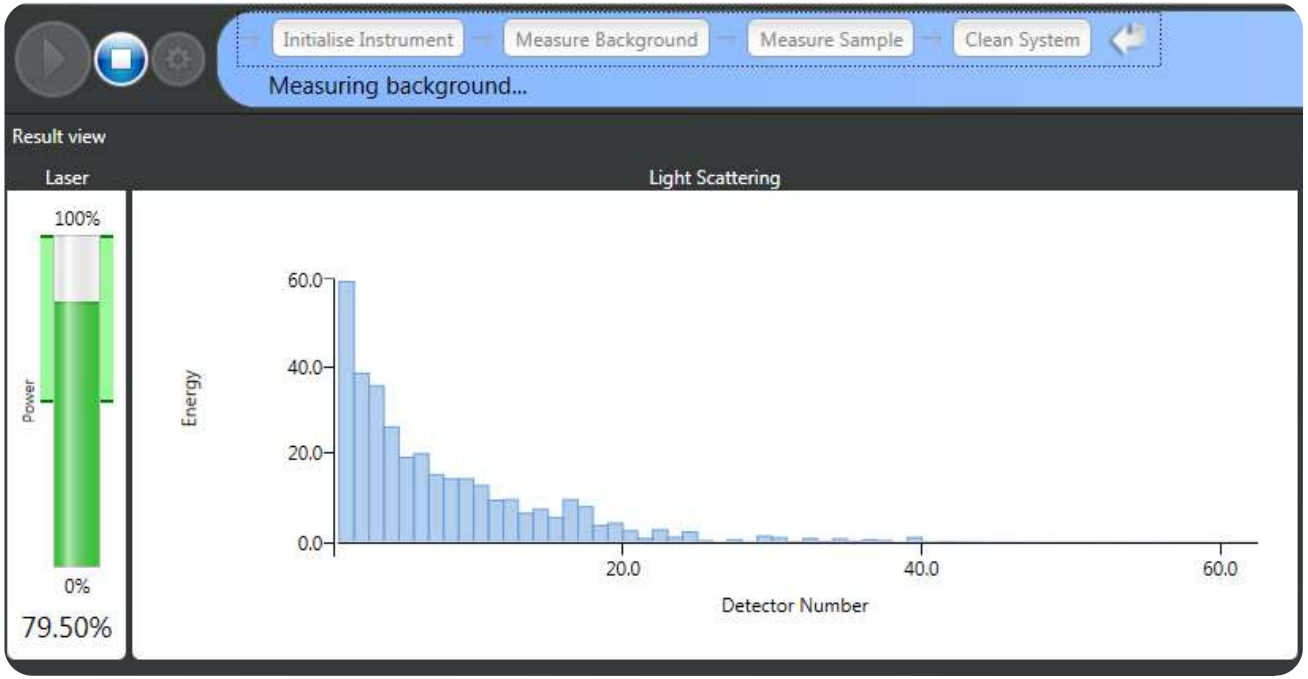
Mastersizer 3000 optics: blue light measurements



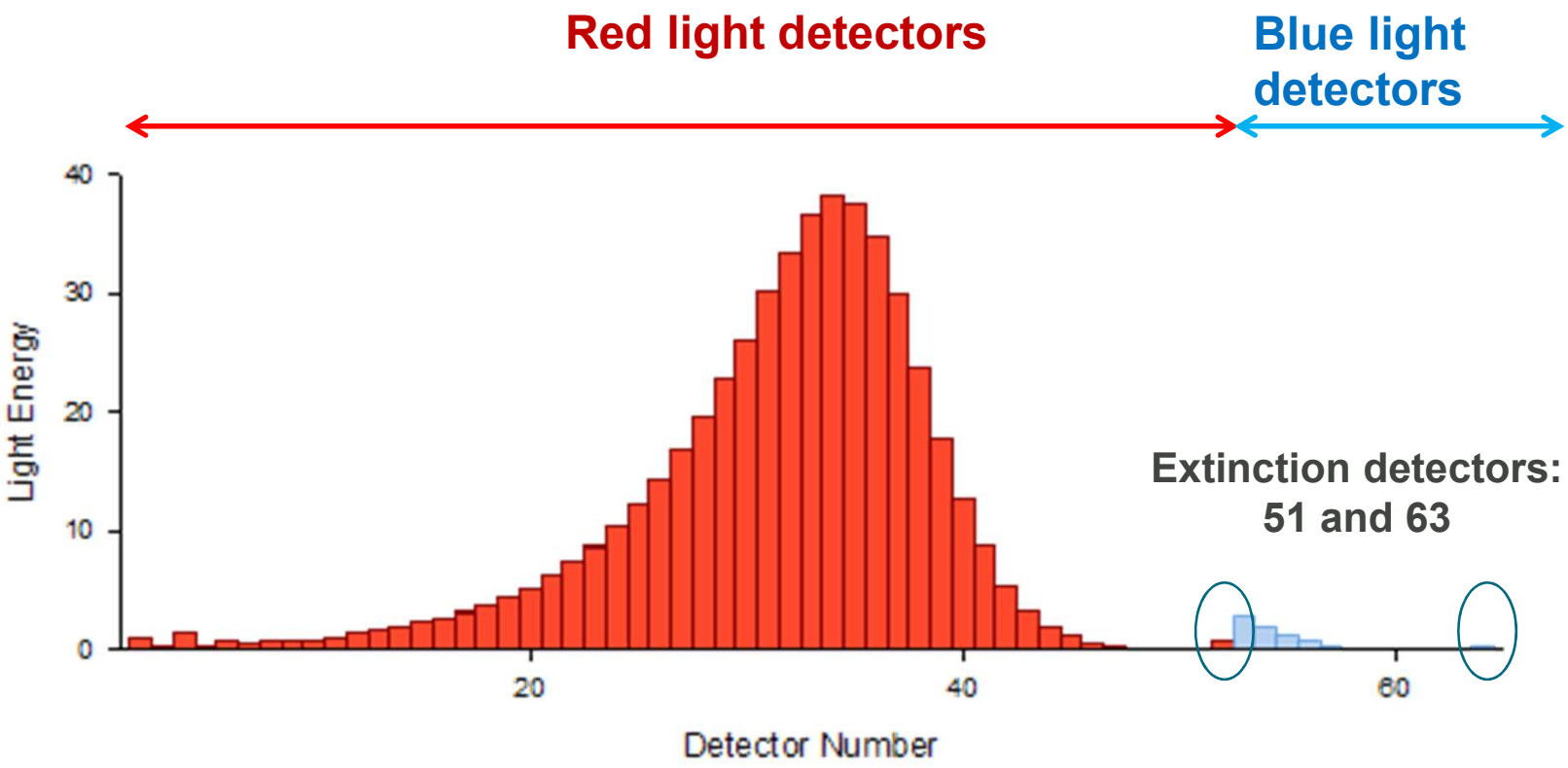
Shorter wavelengths resolve smaller particles.

Measuring the scattering data

- Angular scattering data is presented in real-time in the measurement manager
- Increasing detector numbers represent increasing angle



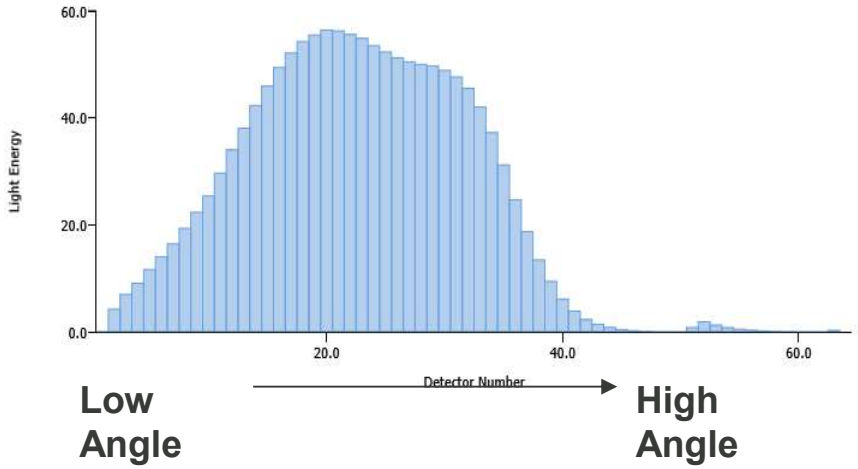
The measured scattering data



Increasing angle / Decreasing particle size

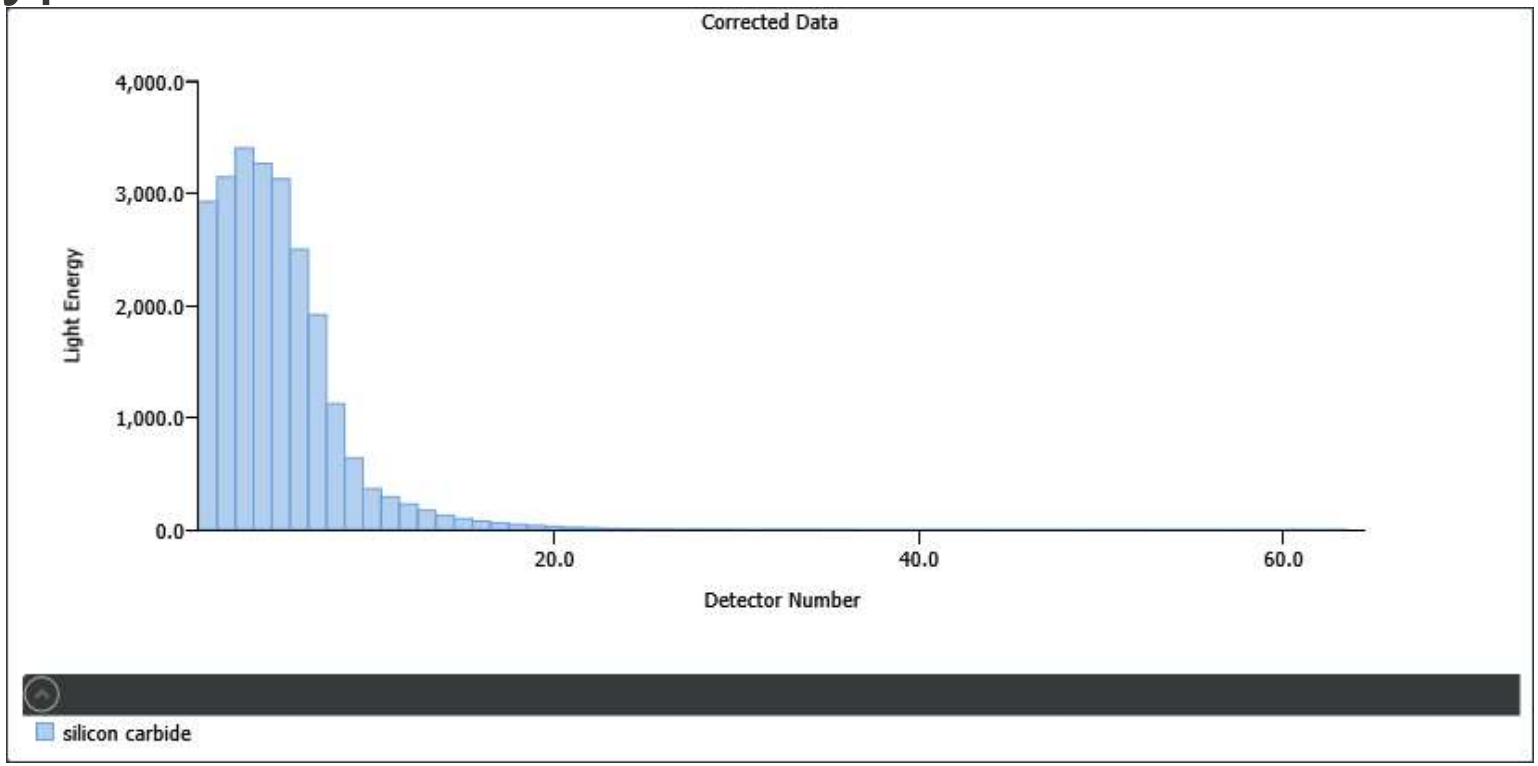
The Mastersizer 3000 detector arrangement

Detector number range		Laser	Detectors
1 to	39	Red	Focal plane
40 to	47	Red	Large angle
48 to	50	Red	Backscatter
51		Red	Red extinction
52 to	59	Blue	Large angle
60 to	62	Blue	Backscatter
63		Blue	Blue extinction



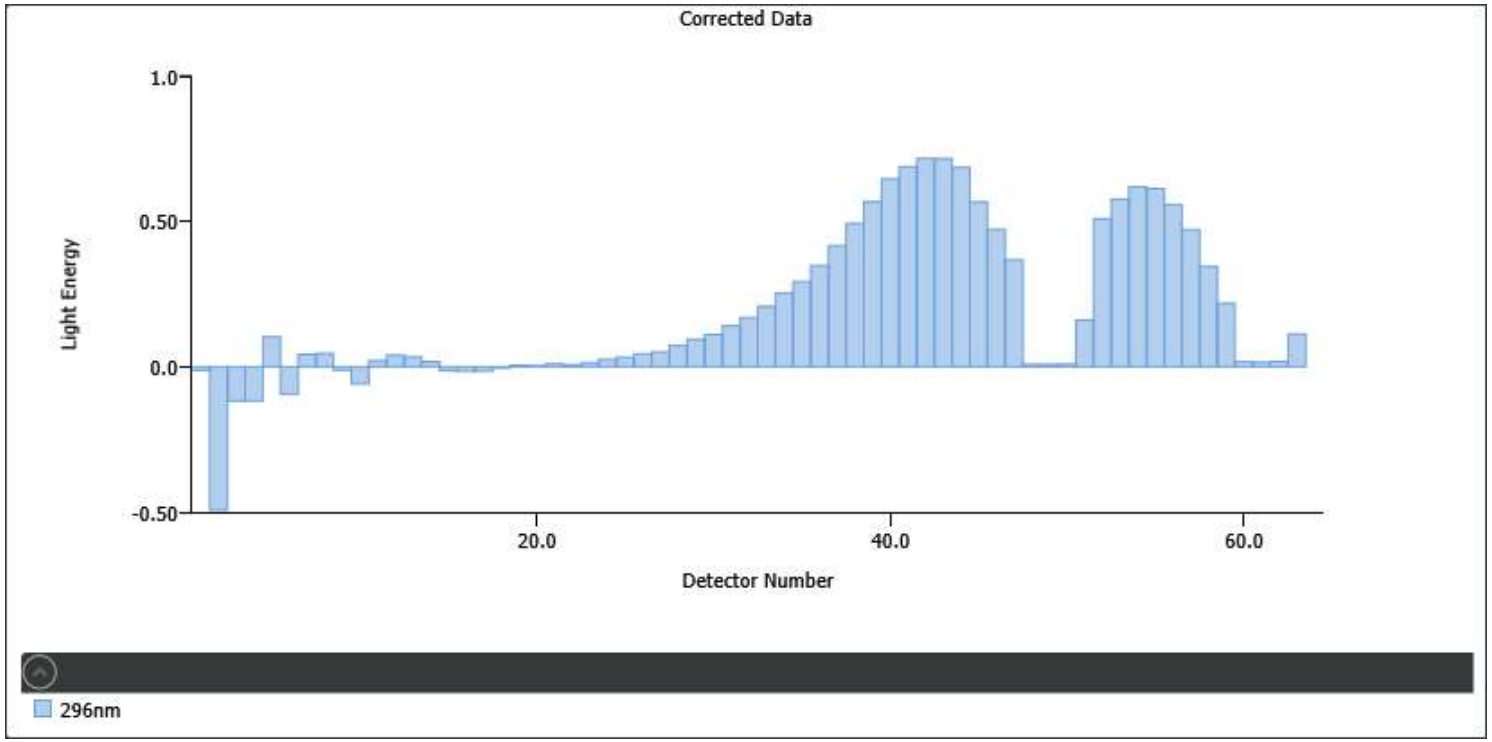
For the curious

The Mastersizer 3000
Typical Data Set – Coarse Particles

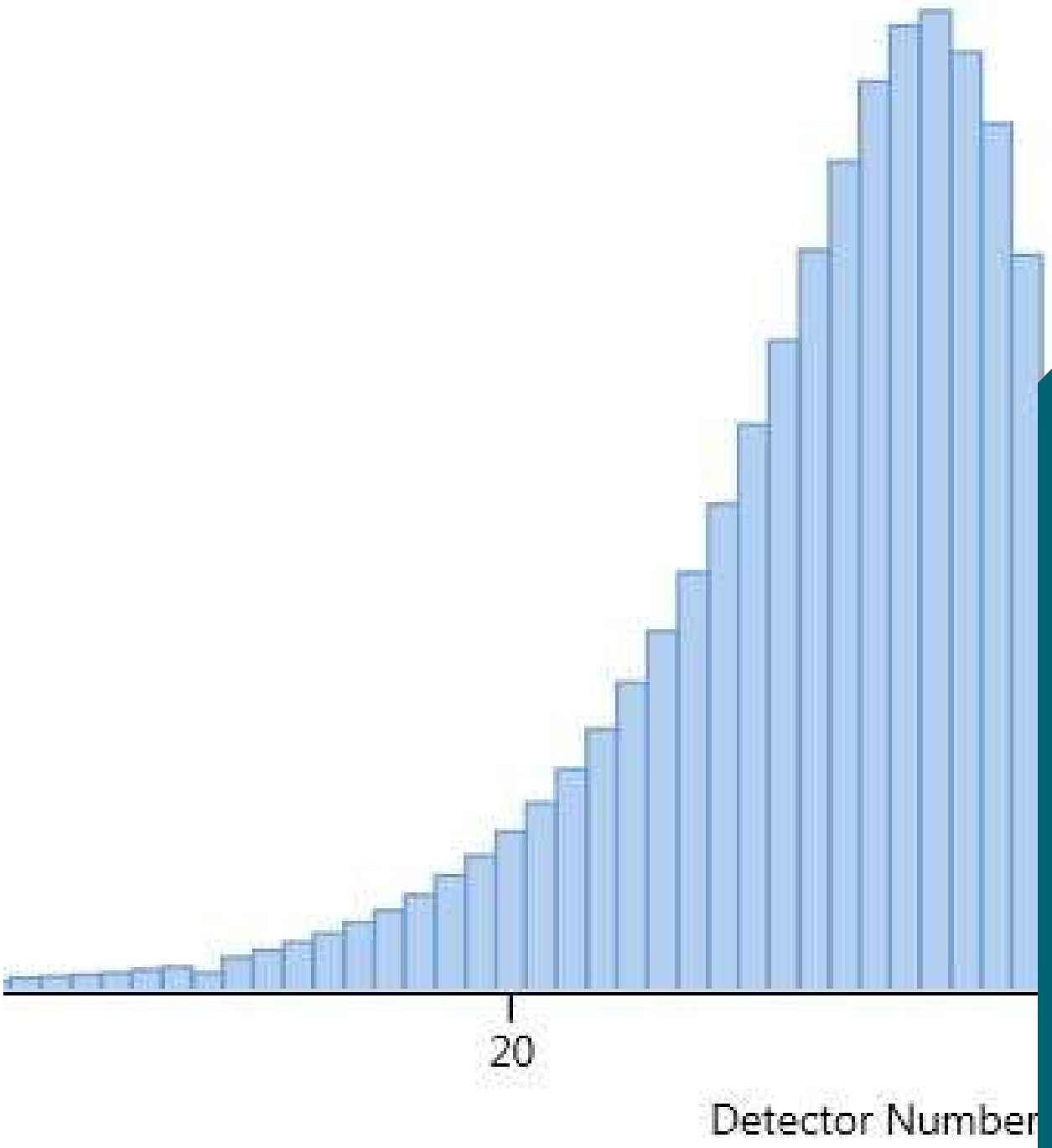


Large particle scattering is concentrated in the low angle region which corresponds to low detector numbers.

The Mastersizer 3000
Typical Data Set – Sub-Micron Particles



Small particles scatter light at high angles which produces data in the high detector number region.



Data quality

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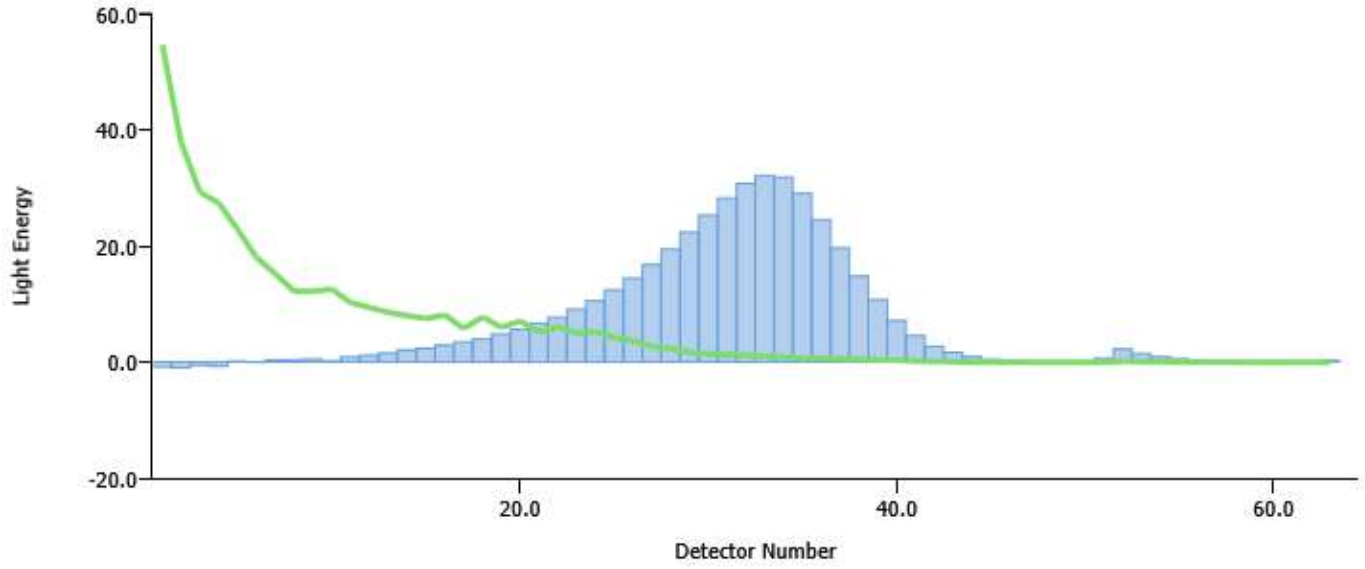
Data quality - introduction

- Data is the fundamental light scattering caused by the sample

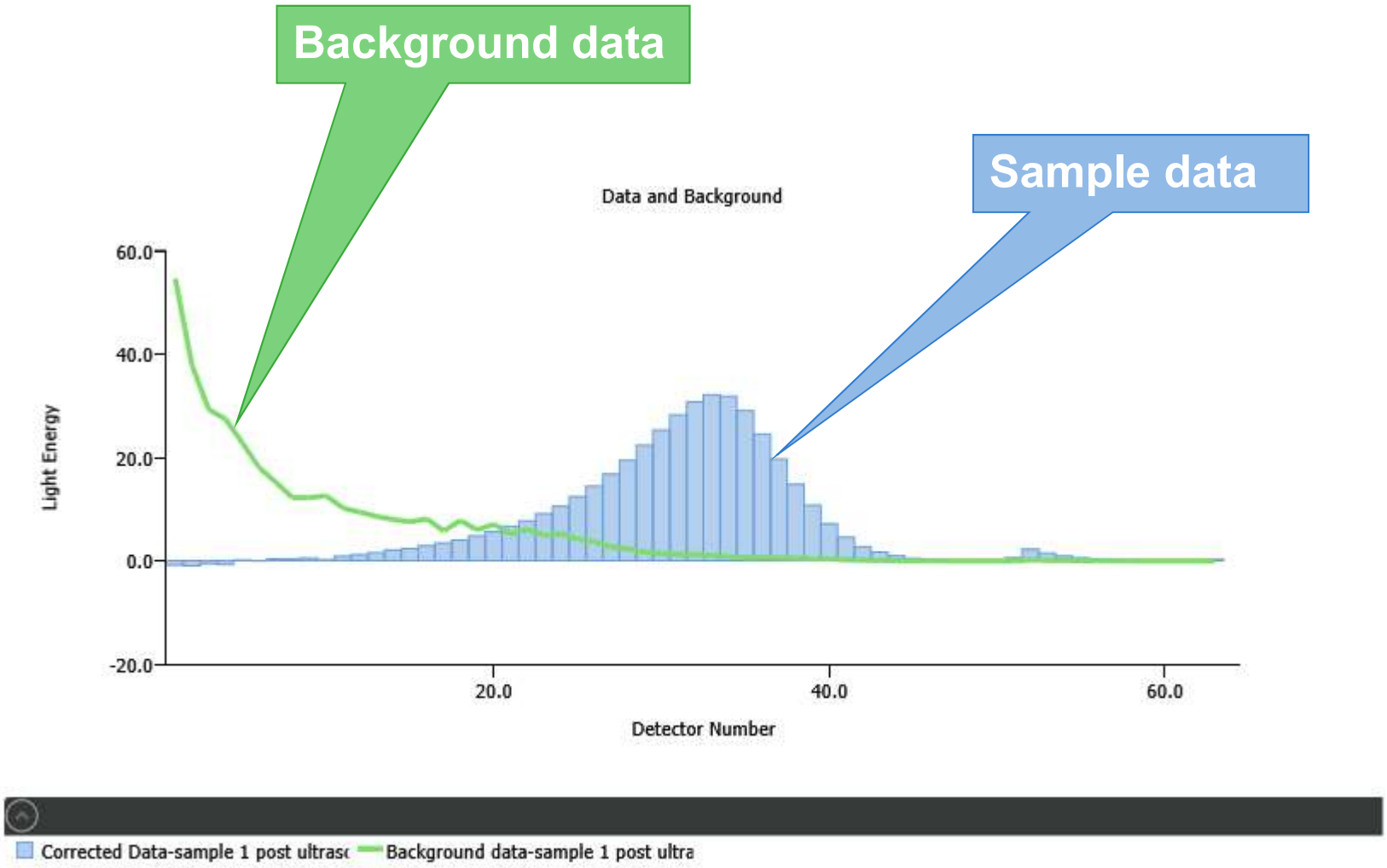


- Data is not the particle size result (.pdf)
- Data is independent of the optical model
- A stable result requires stable data

What is good quality data?

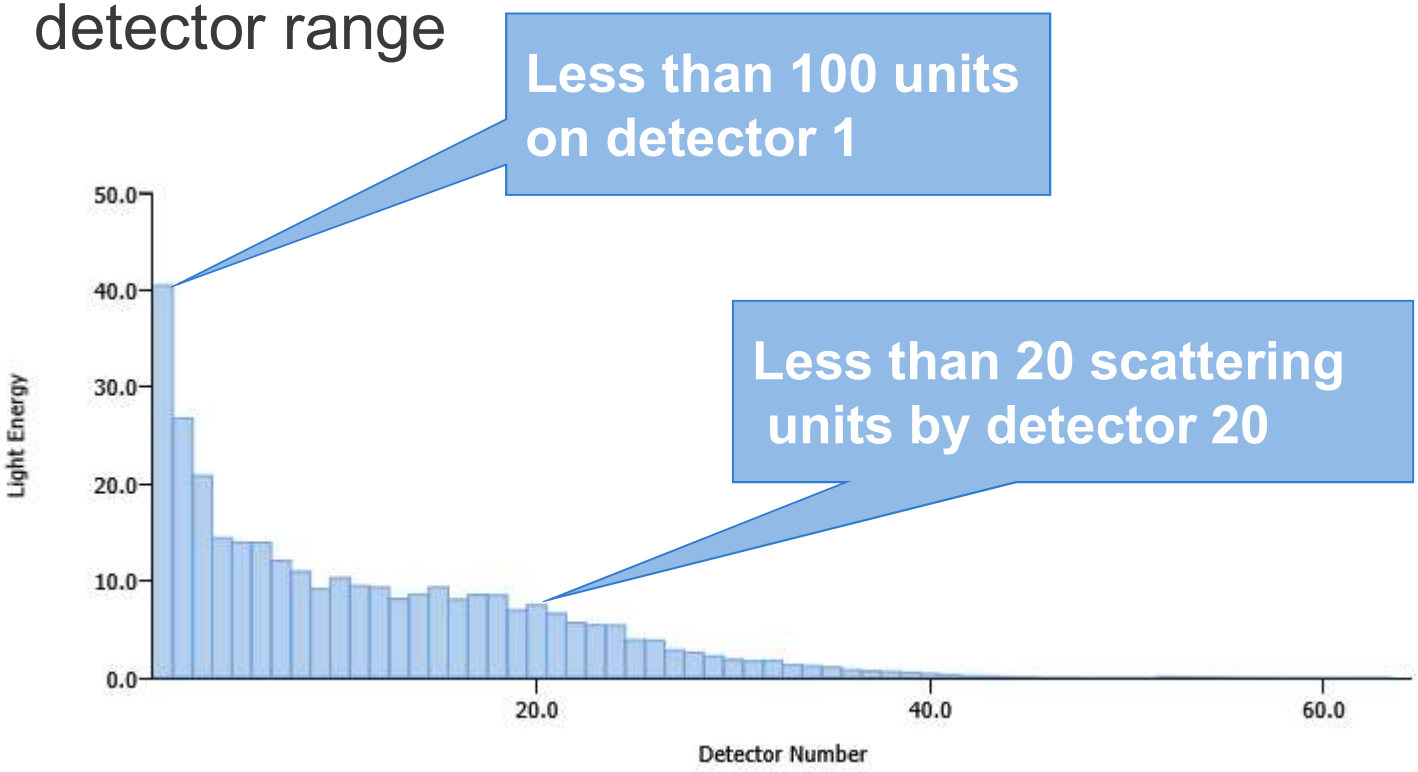
- A good background measurement shows:
  - Clean cell windows and dispersant
  - Good alignment of the system
  - Stability of the dispersant
- Good sample measurement should have:
  - Sufficient signal to noise ratio
  - Limited negative data
  - No multiple scattering
  - No beam steering

Data components - types of data

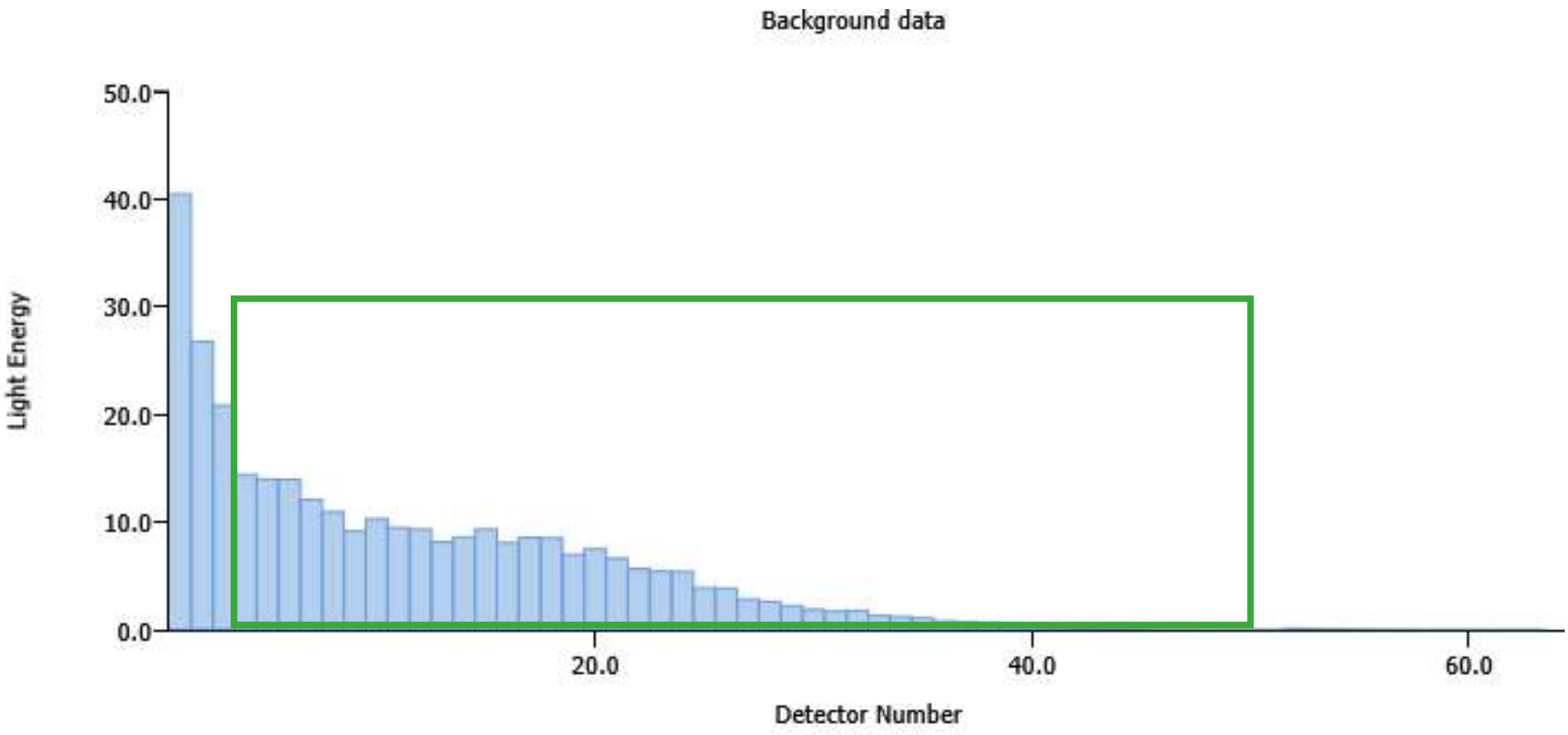


Background data and system cleanliness

- A good measurement requires a clean, stable background
- This should show progressive decrease across the detector range

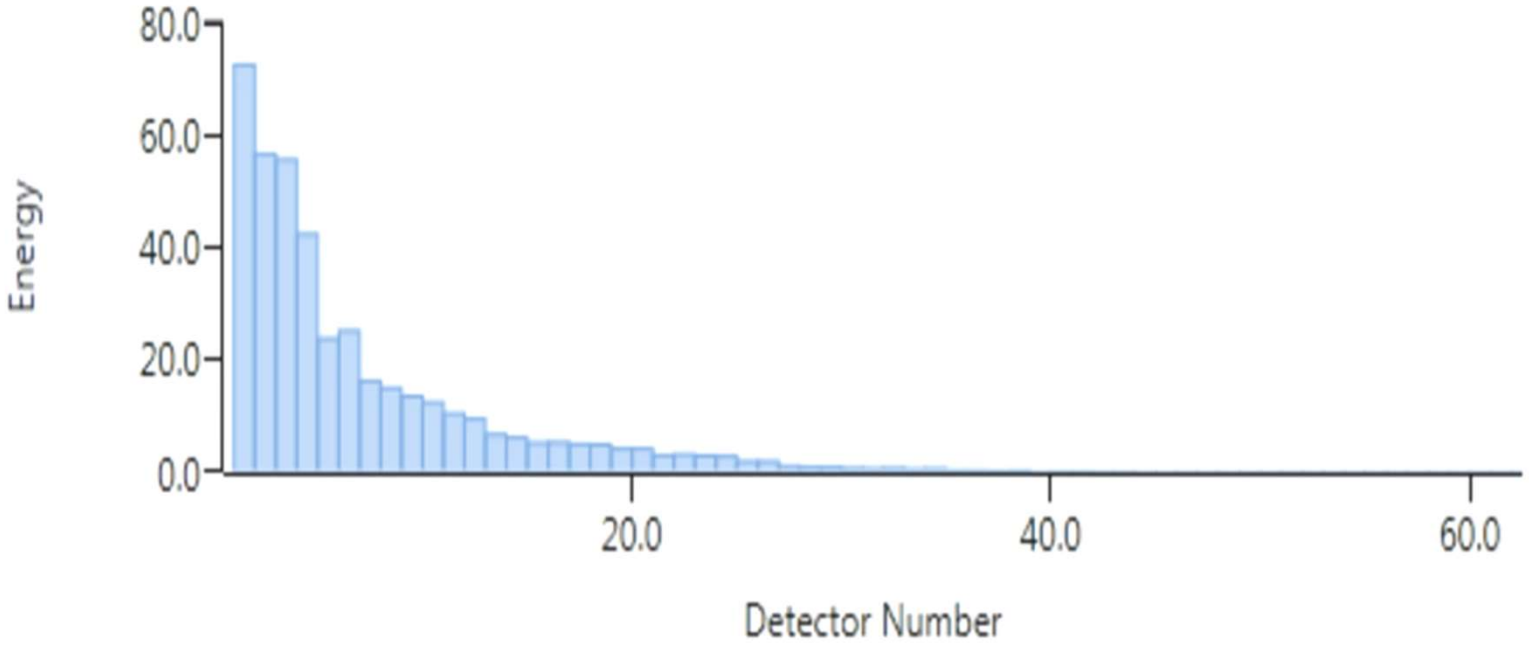


MS3000E Background



A clean background – wet system

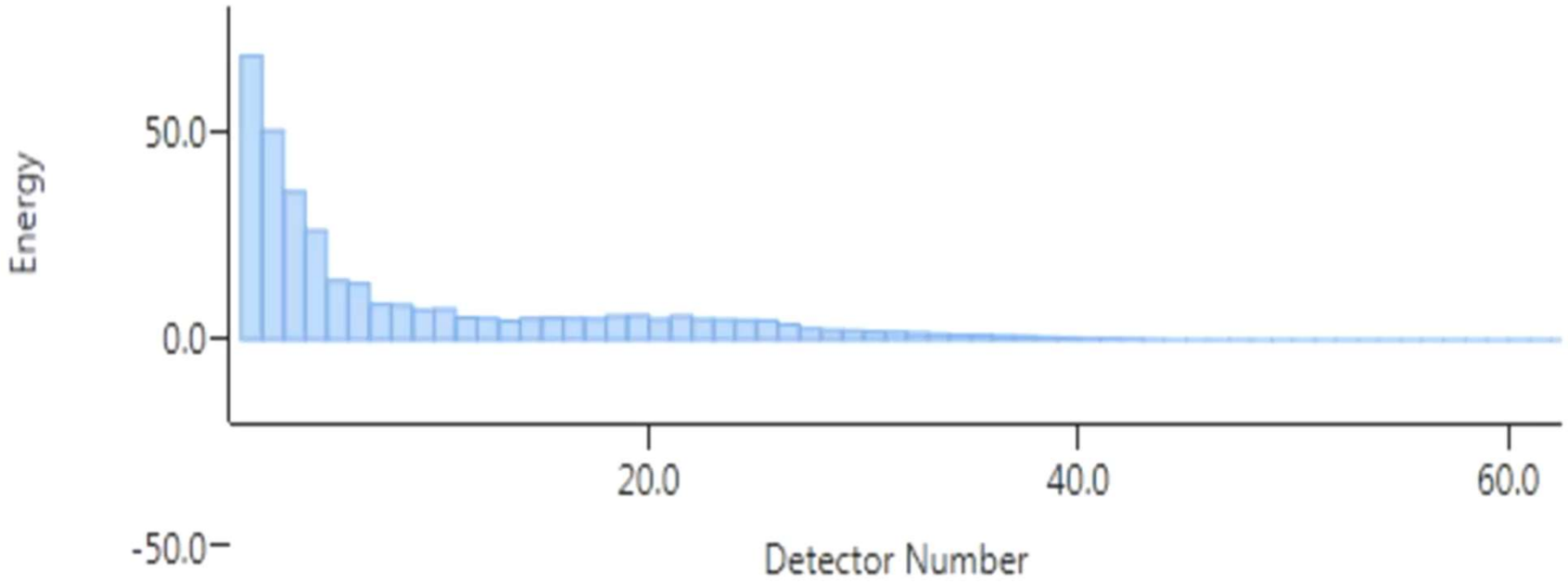
- A good clean background on a wet system should look very similar to this...



- Less than 100 on detector 1
- Less than 20 on detector 20
- Decreasing curve
- Limited fluctuations

A clean background – dry system

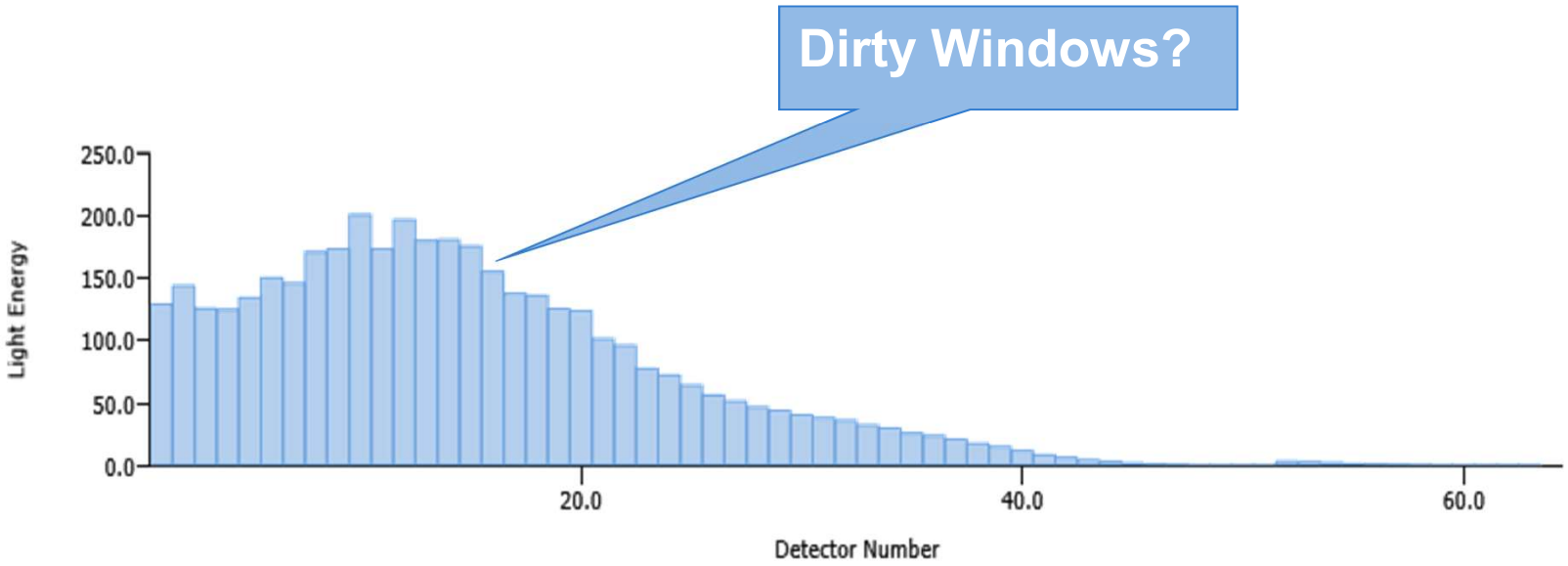
- The air flow causes larger fluctuations in the background than in a wet measurement.
- The same rules apply!



- Less than 100 on detector 1
- Less than 20 on detector 20
- Decreasing curve
- Limited fluctuations

Poor background - material stuck to the windows

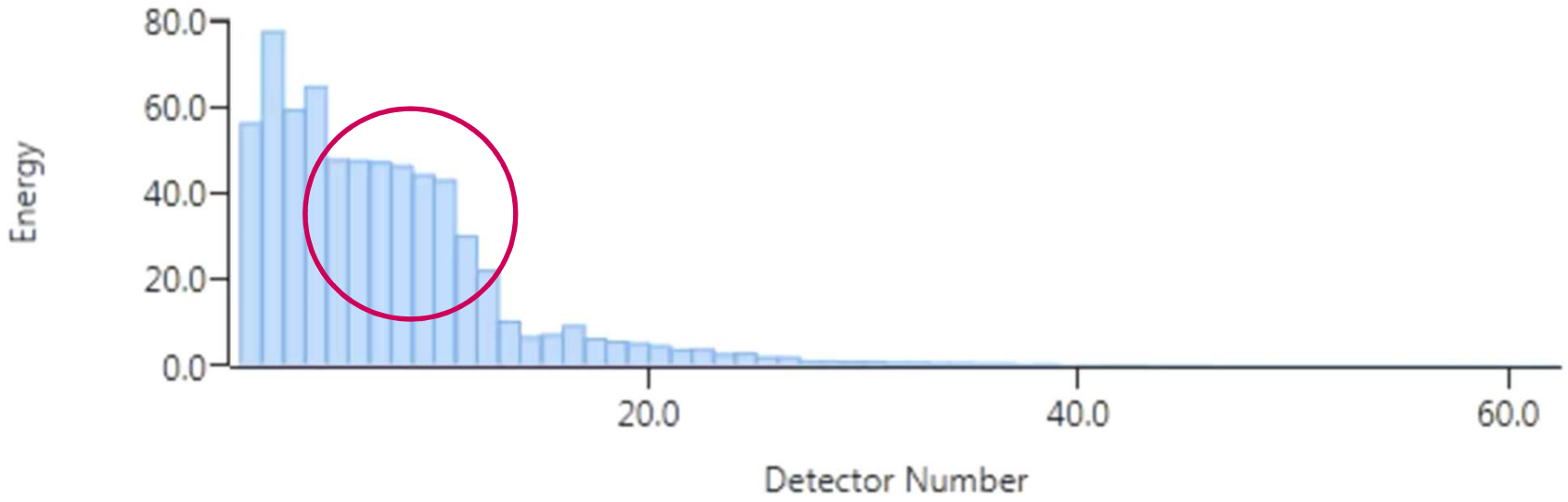
- A ‘hump’ in the data is often an indication of material stuck to the cell windows



- All scattered light is included in the measurement

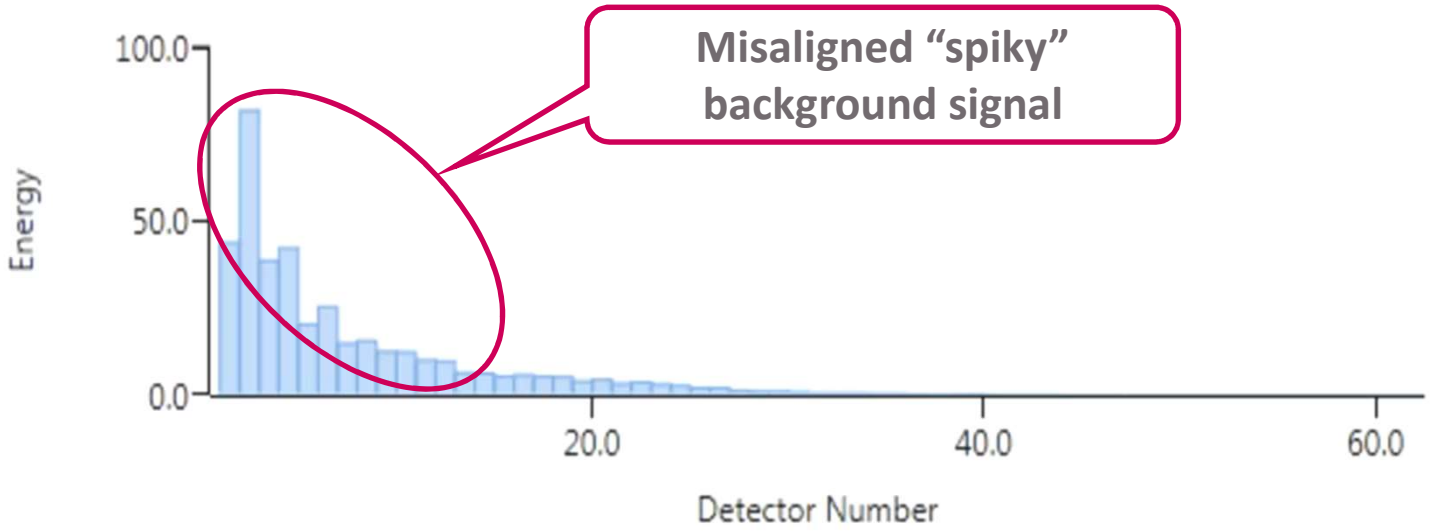
Poor background – contaminated dispersant

- Intermittent peaks in the background may be caused by contaminants in the dispersant
  - Particulates (rinse the dispersion unit)
  - Bubbles (degas dispersant, stop-start pump)



Poor background – misaligned system

- A spiky background signal indicates misalignment
  - Detectors are arranged on opposite sides of the pinhole
- Misalignment can be caused by
  - Contamination on the cell windows
  - A change in the refractive index of the dispersant

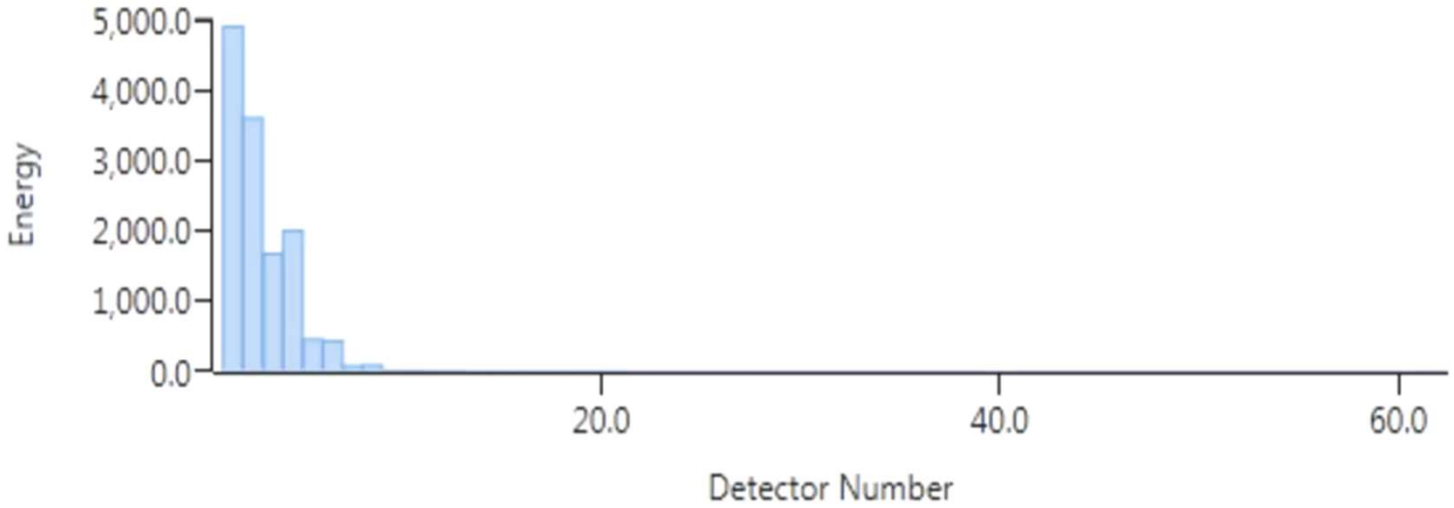


Poor background: instability due to thermal gradients

- The dispersion unit may be warmer than the dispersant
- This temperature difference causes thermal gradients in the dispersant
  - And high backgrounds and possible alignment problems

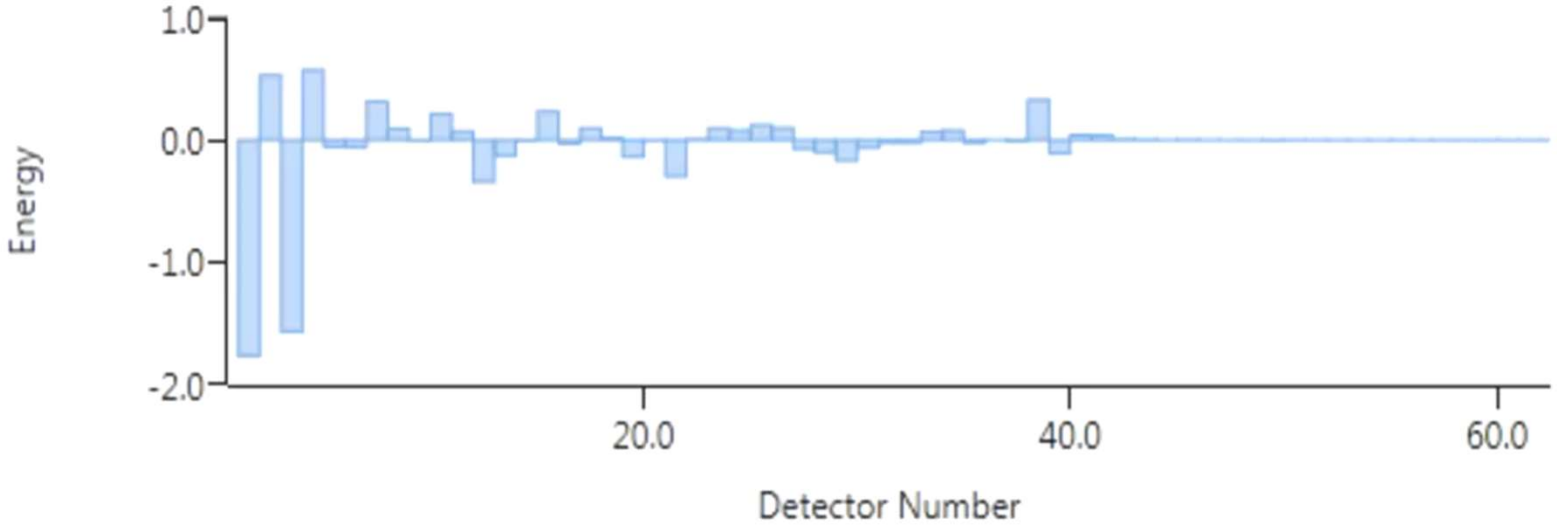
How will I recognise thermal gradients?

- Thermal gradients cause high background signals, and large fluctuations
  - The background signal decreases as the temperature stabilises and refractive index gradients disappear
  - This will take longer for more volatile dispersants



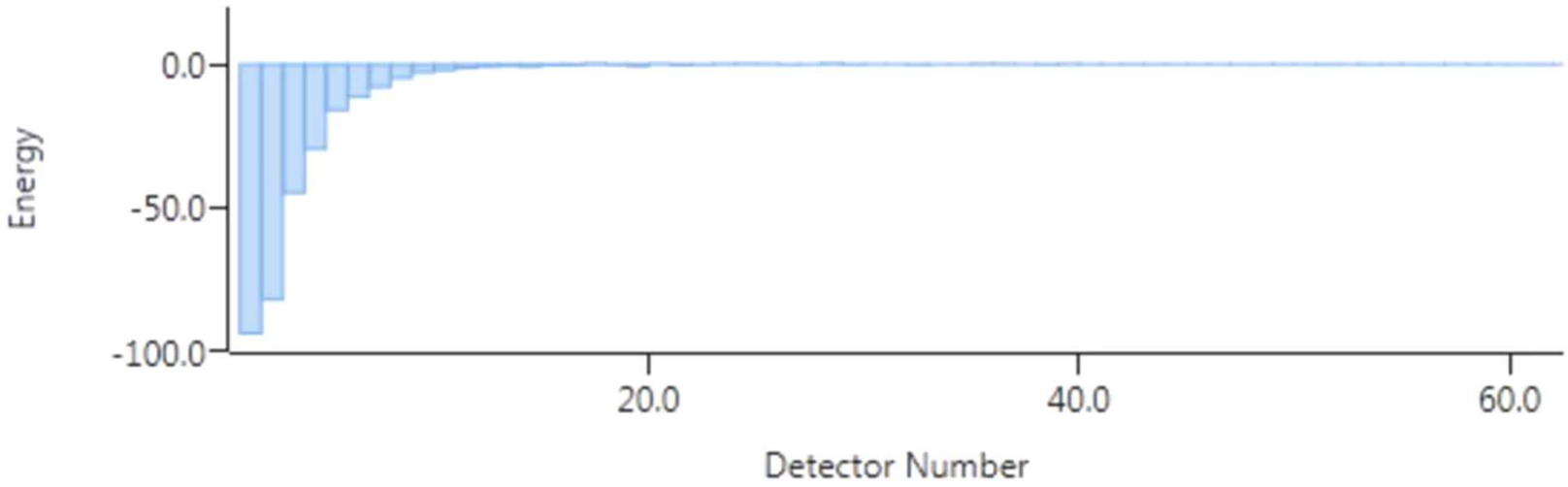
The Add Sample stage

- The background signal as been subtracted
- The live data should then show random fluctuations around zero
- Any ‘blocks’ of scattering will indicate dispersant contamination



The Add Sample stage – negative data

- The background signal has been subtracted
- The live data should then show random fluctuations around zero
  - Significant negative data suggest that the background had not stabilised before it was measured
  - If you see this signal, re-measure the background



Sample addition

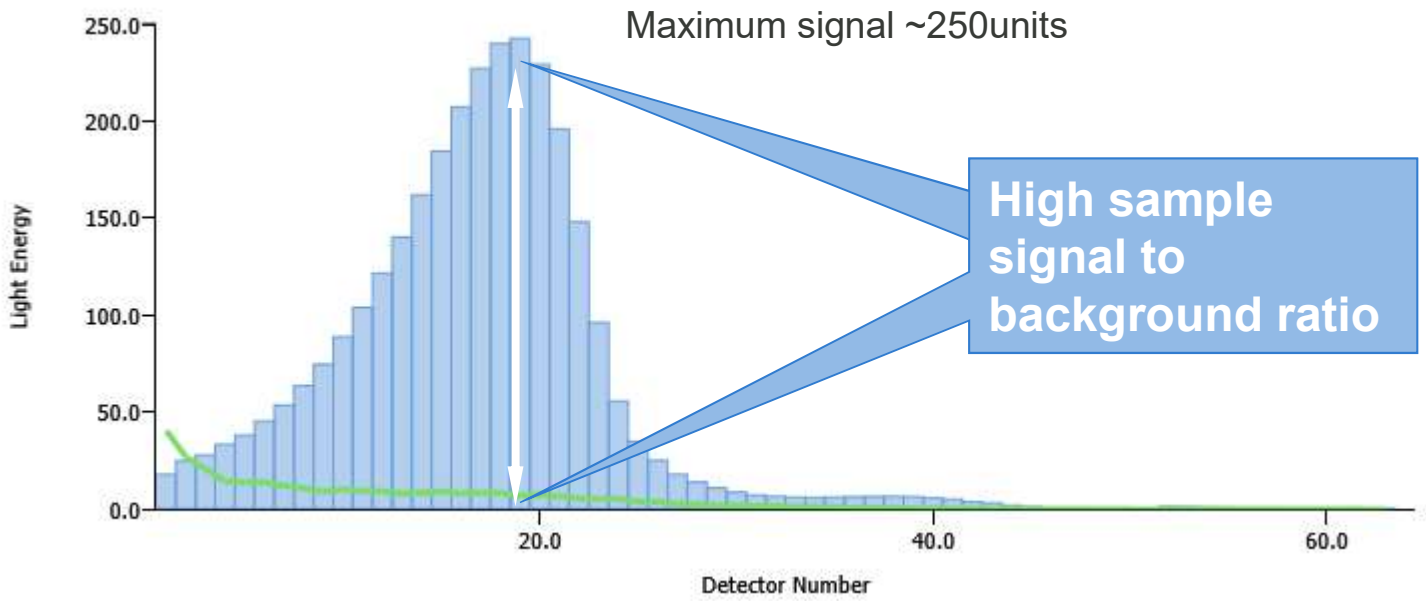
- How much sample should be added to the dispersion unit?
  - **Too little**:
    - Signal to noise ratio may be poor, or
    - Not enough sample may have been added to be representative of the bulk – particularly if the sample is very polydisperse
  - **Too much:**
    - Multiple scattering may affect the reported particle size distribution – particularly if the material is small (typically < 10 microns)

- What is the *correct* obscuration range?

Obscuration = amount of laser light blocked and/or scattered by the sample, a guide to concentration

Low obscuration limit: signal to noise ratio

(42.58 micron glass beads measured at an obscuration of **only** 7%)

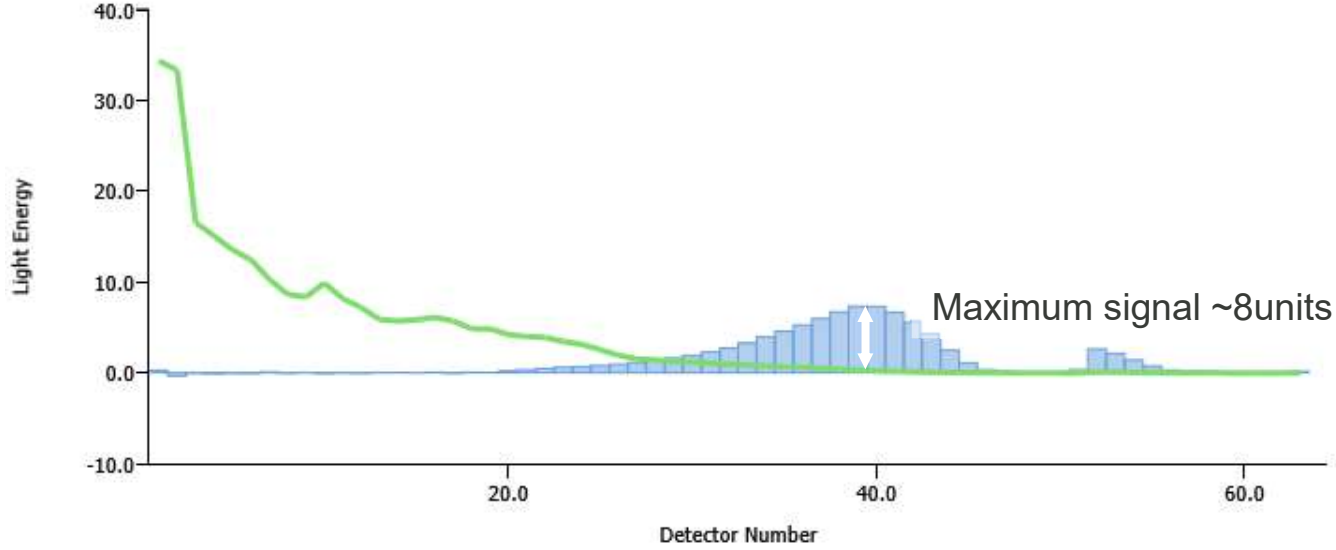


Note: the signal to noise ratio is usually high for large particles because these scatter light more strongly.

Consequently, signal-to-noise ratio is less of an issue for large particles.

Low obscuration limit: signal to noise ratio

(1 micron Latex measured at an obscuration of 5%)



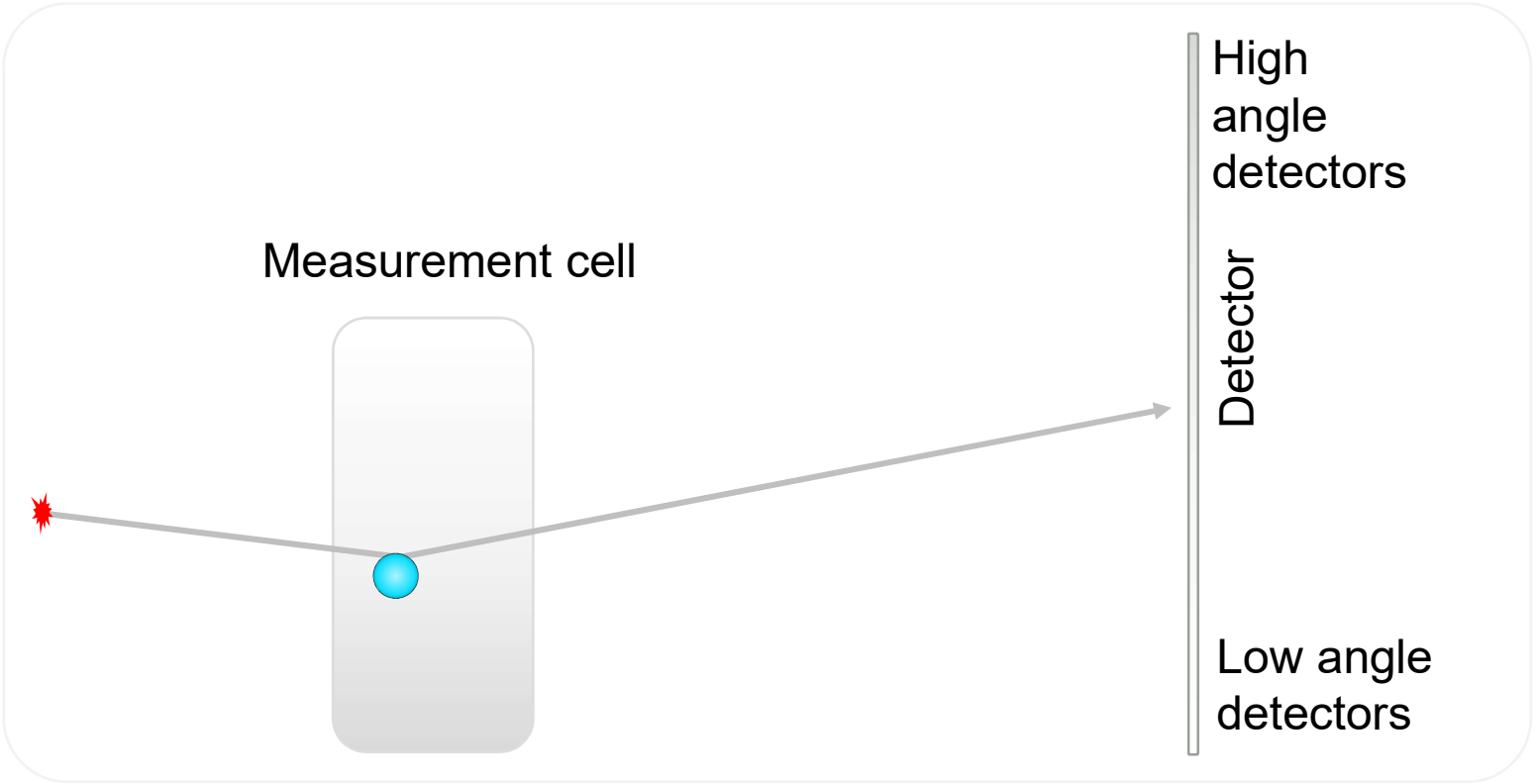
The signal-to-noise ratio is the amount of sample data relative to the background data.

Because small particles scatter light weakly, it is important that the background does not swamp the data signal.

However, in this graph, the data is good since it falls where there is little or no overlap between the sample data and the background data.

What defines the upper obscuration limit?

- If we add too much sample the results may be affected by multiple scattering
  - This generally affects samples smaller than 10µm



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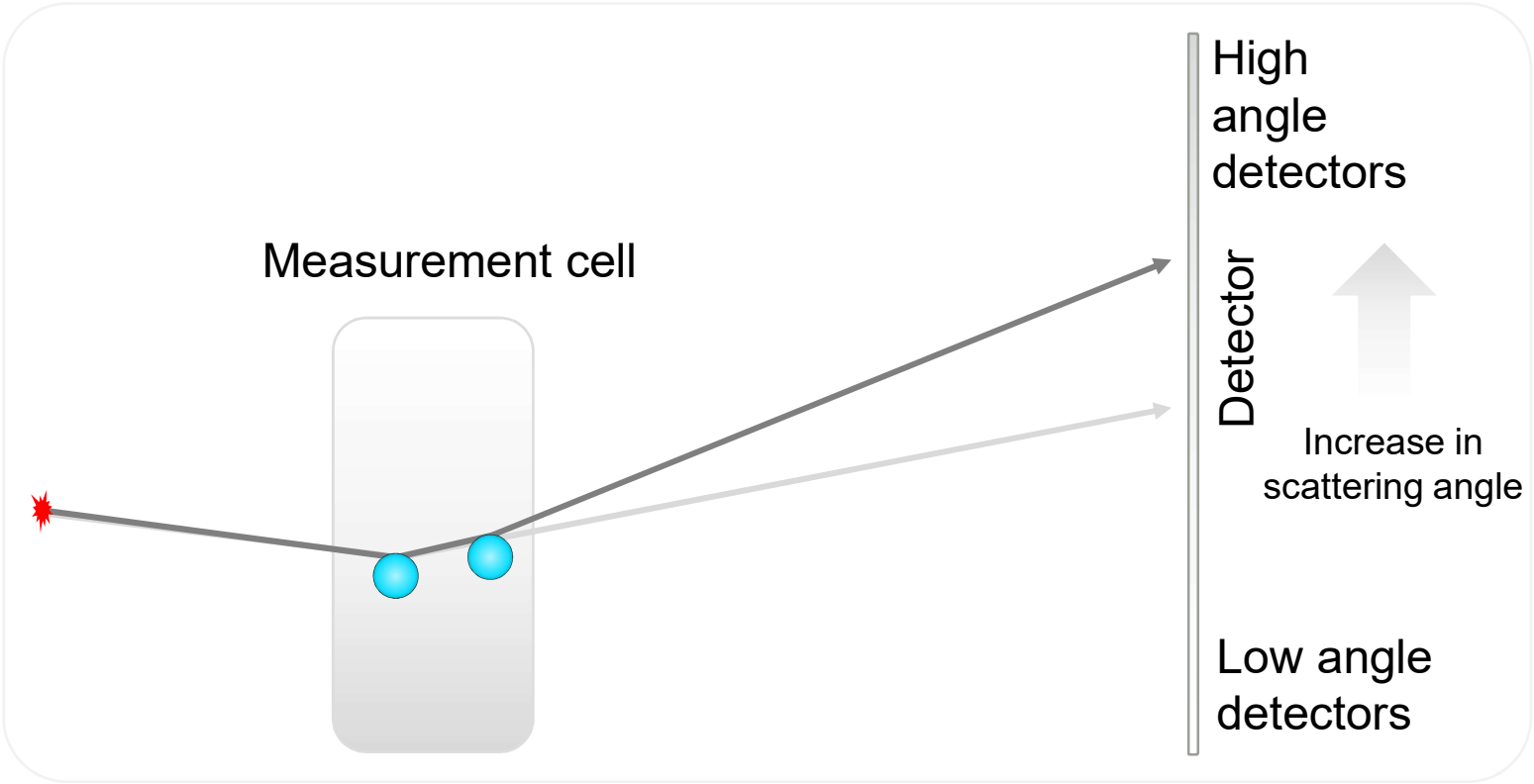
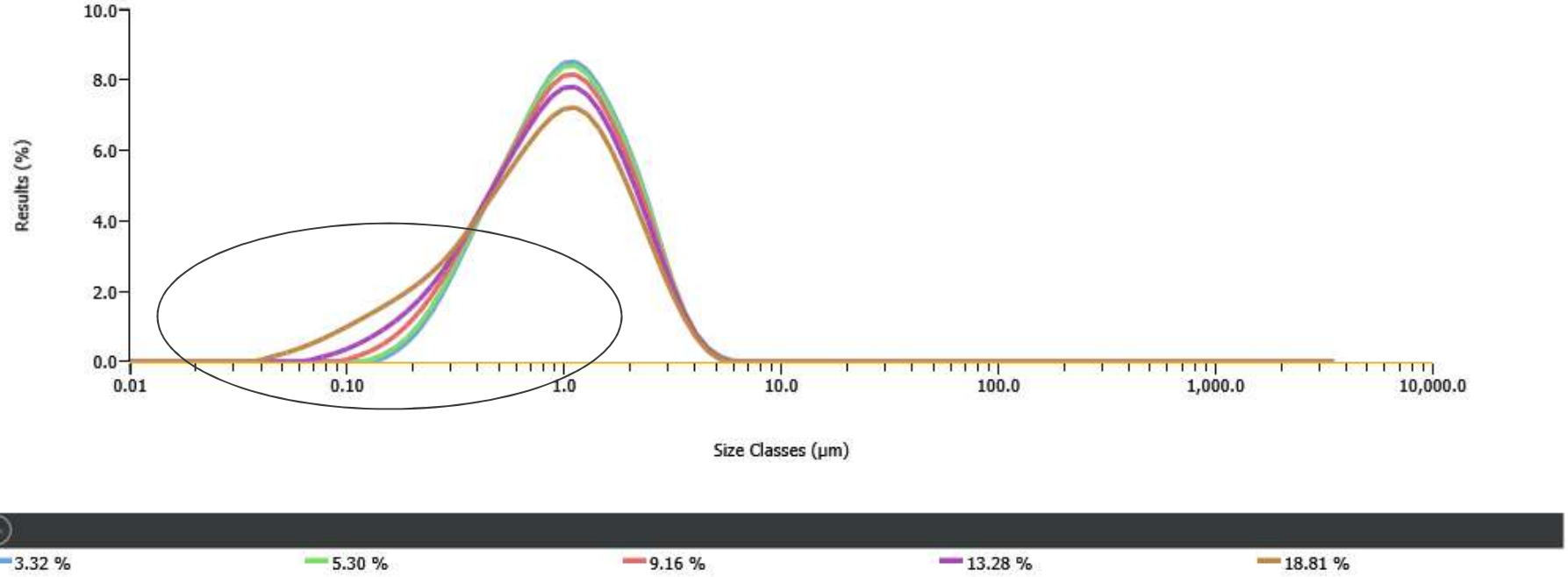


Wet analysis - multiple scattering

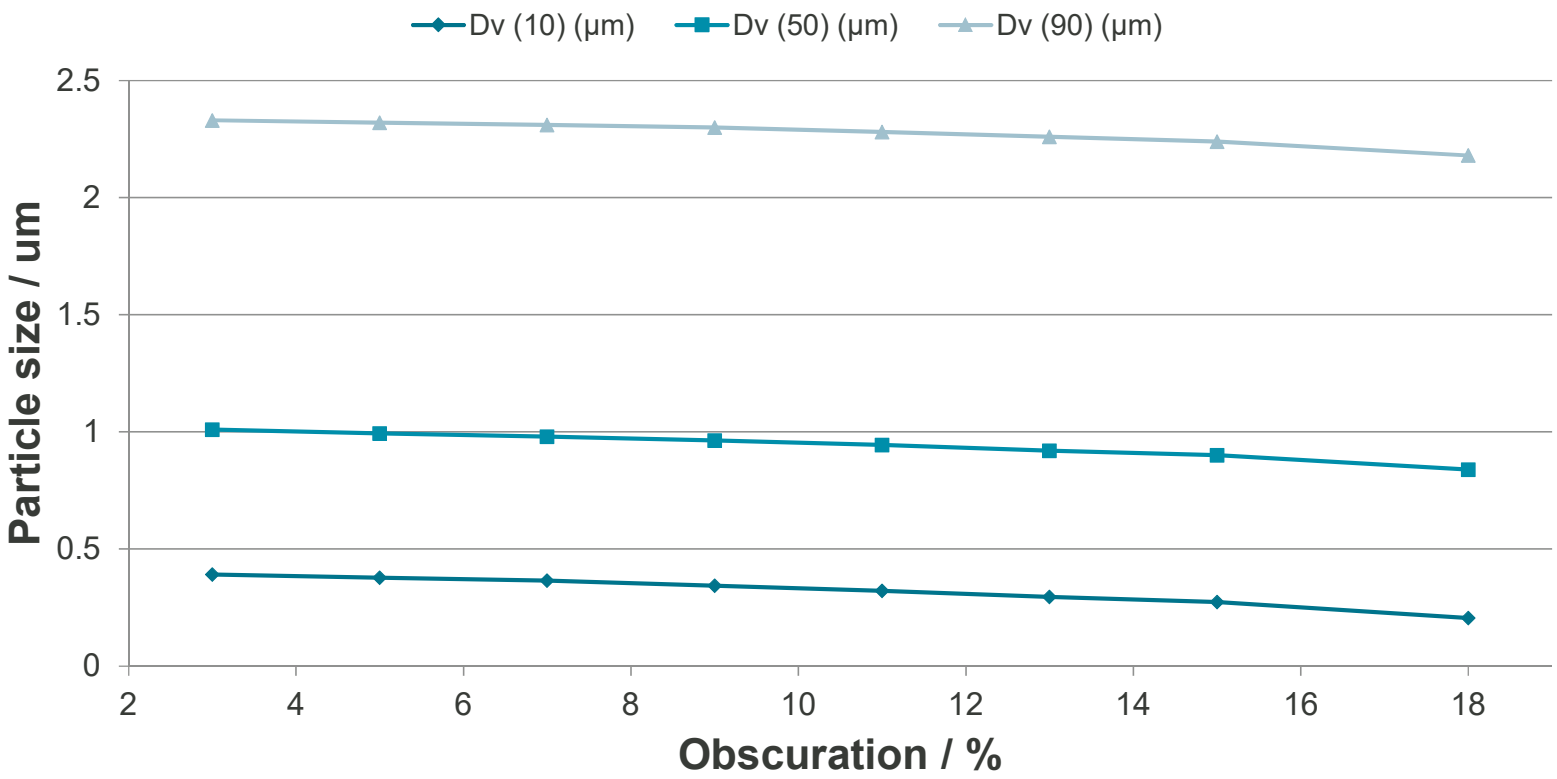
….leading to exaggerated fines being interpreted



- If in doubt, carry out an obscuration titration to determine the effect of measuring at increasing obscurations on the particle size distribution.

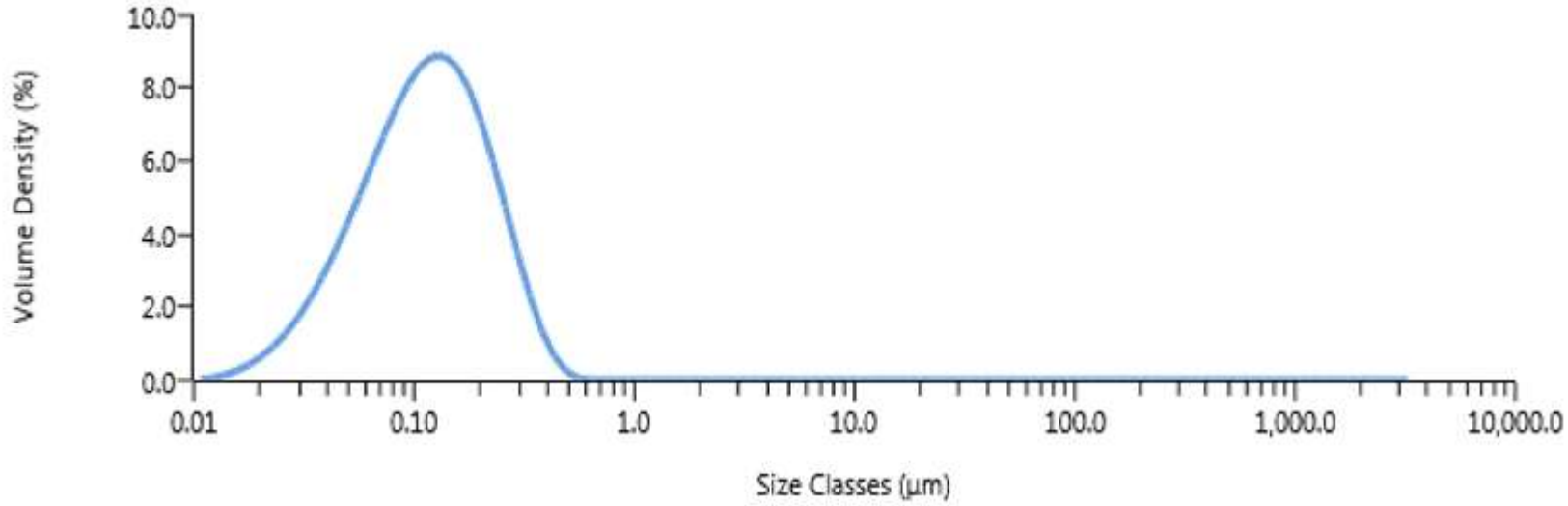
High obscuration limit: multiple scattering

- The upper limit of the obscuration range depends on multiple scattering:
  - Sample should be measured in the range where size is stable with obscuration.



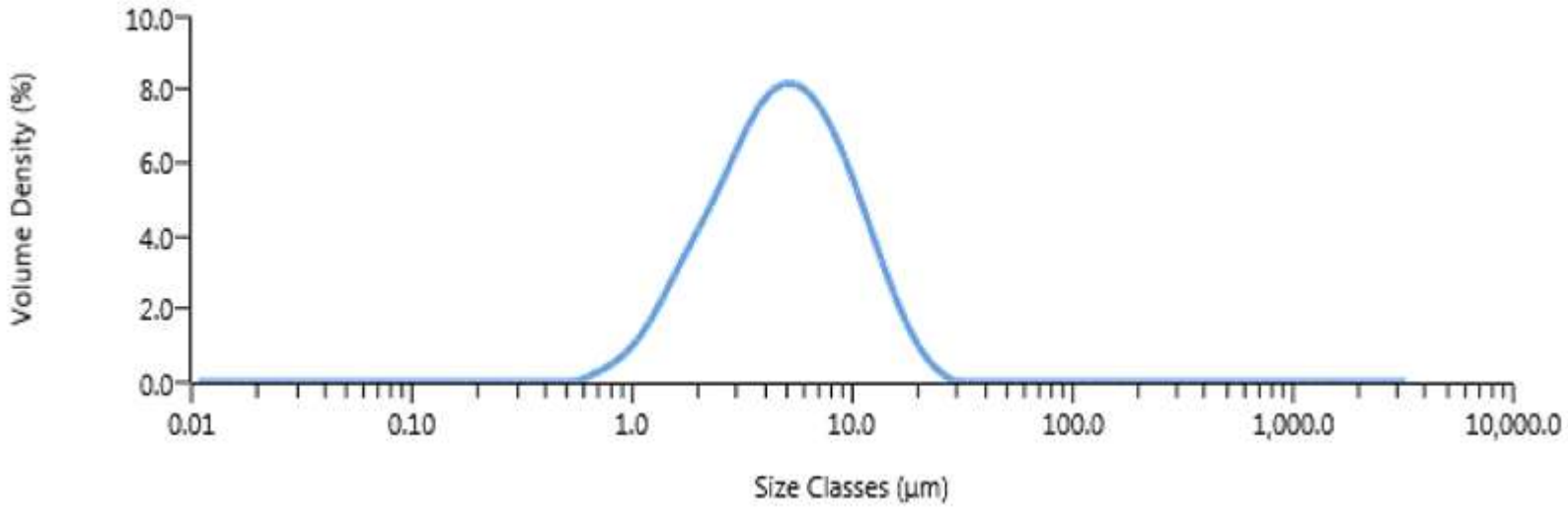
Target obscuration ranges: Wet measurements

- Very fine particles
- <1um
- <5% obscuration



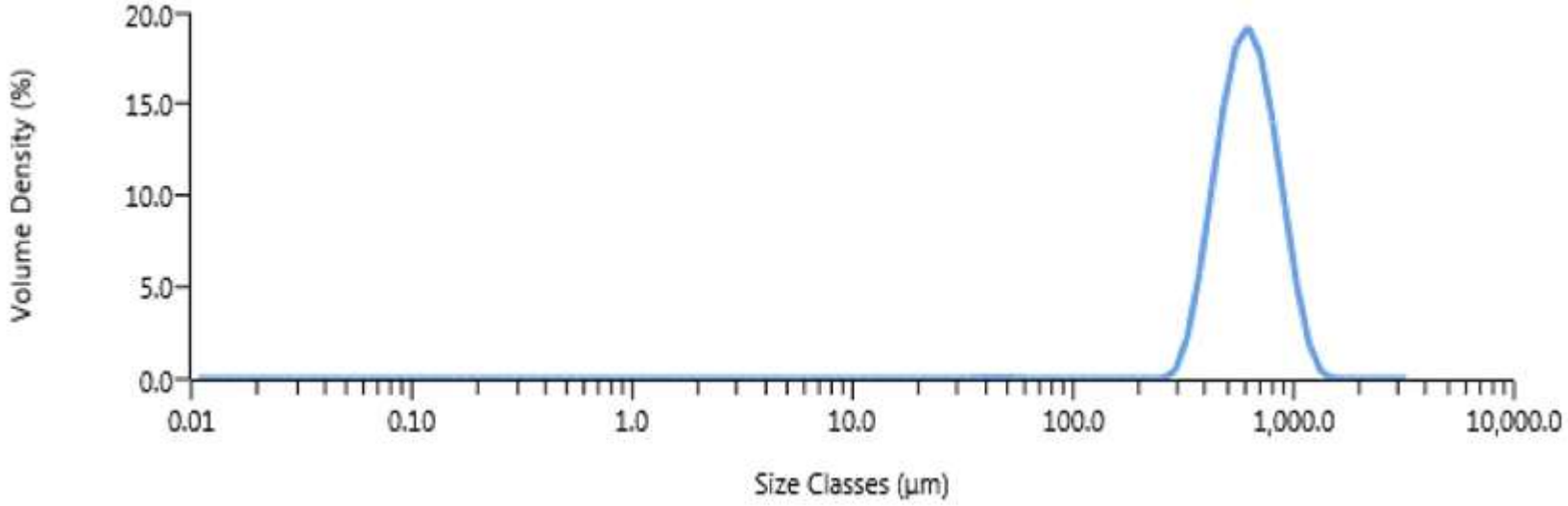
Target obscuration ranges: Wet measurements

- Fine particles
- 1-100um
- 5-10% obscuration



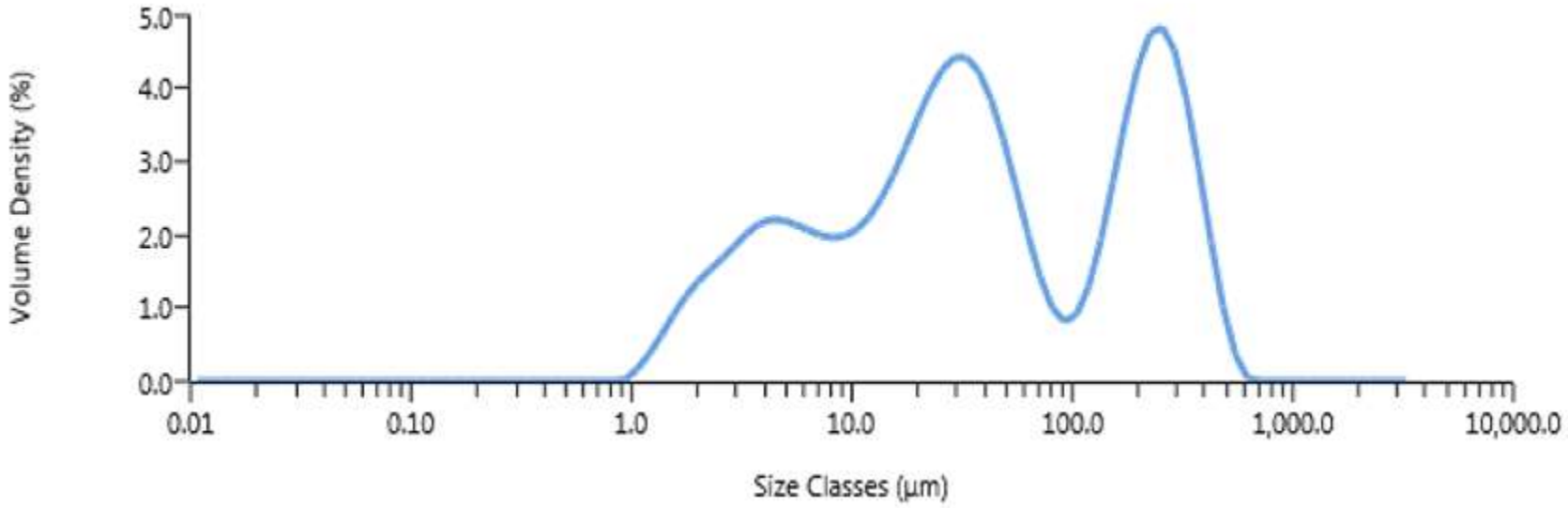
Target obscuration ranges: Wet measurements

- Coarse particles
- >100um
- 10-20% obscuration



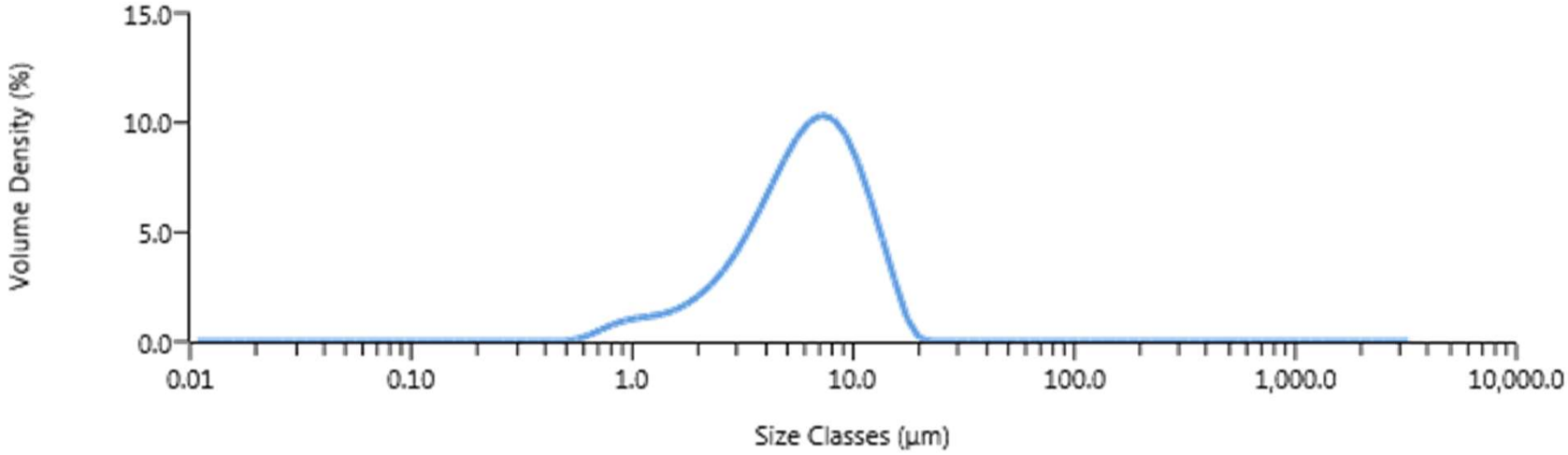
Target obscuration ranges: Wet measurements

- Polydisperse samples
- eg 1-500um in one sample
- 10-20% obscuration



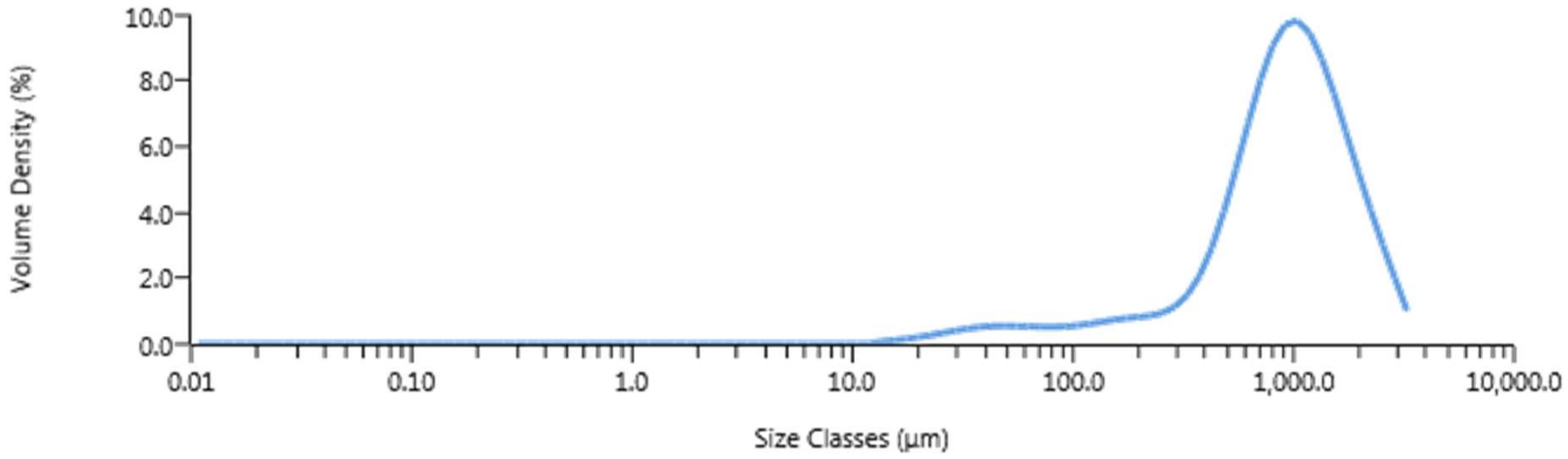
Target obscuration ranges: Dry measurements

- Fine and cohesive powders
- 0.5 to 3-5% obscuration
- Obscuration filtering ensures that only detector scans within the set obscuration range are included in the results



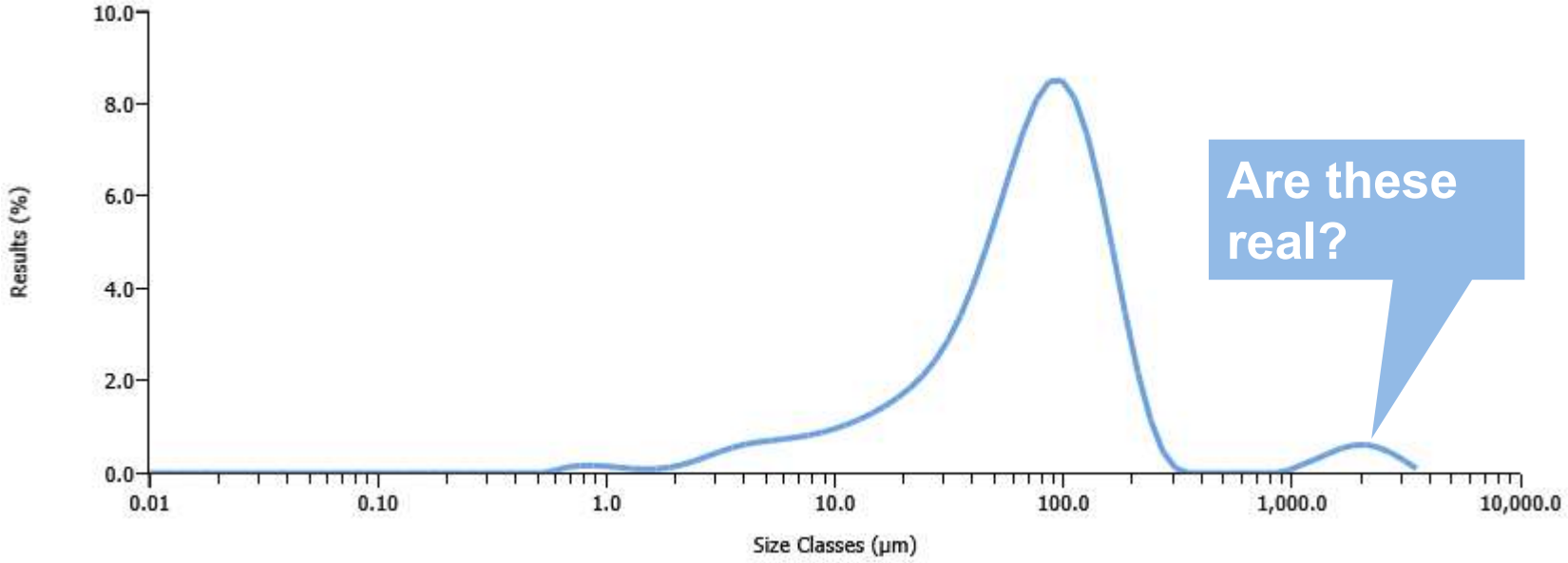
Target obscuration ranges: Dry measurements

- Coarse and free-flowing powders
- 0.1 to 6-8% obscuration
- Obscuration filtering ensures that only detector scans within the set obscuration range are included in the results



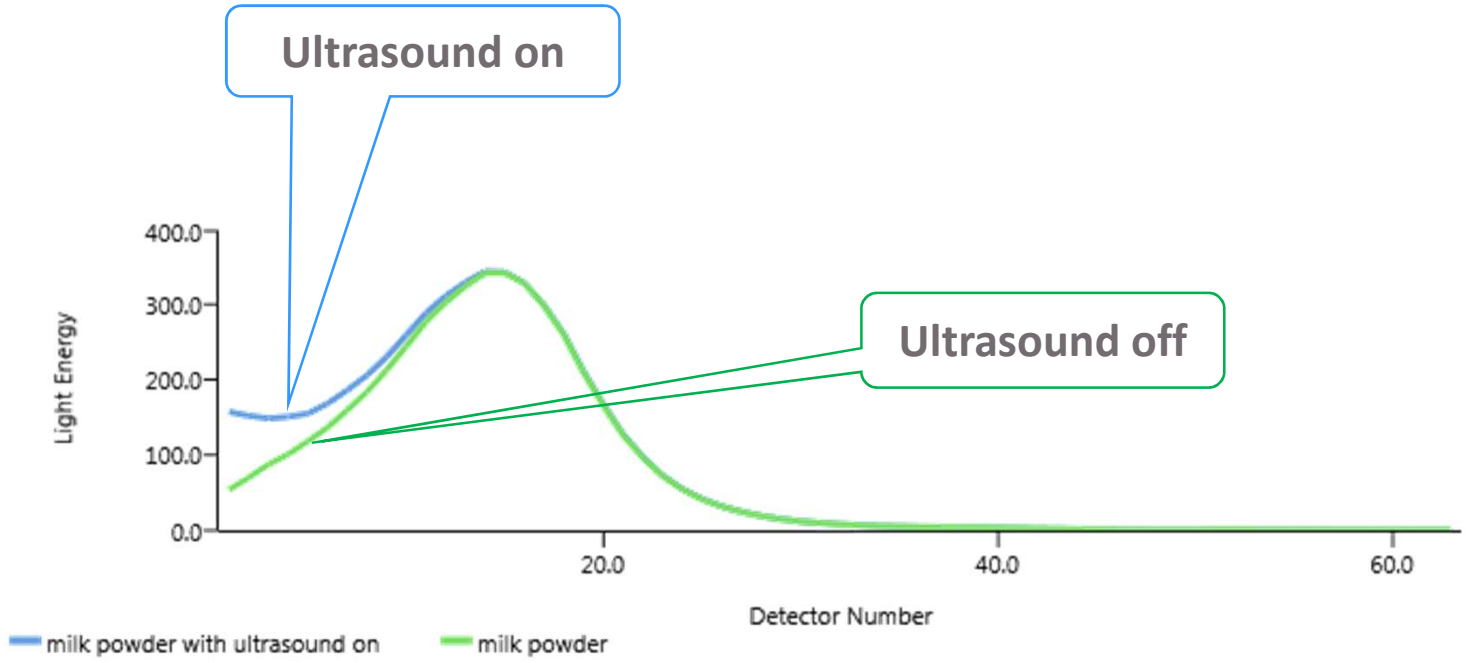
Beam Steering

- The peak on the right of this graph suggests the presence of large particles.
- However this could be caused by Beam Steering, resulting from:
  - Thermal instability following the use of ultrasound in solvents
  - Partial dissolution of the sample in the dispersant - changing its refractive index
- Or real large particles?



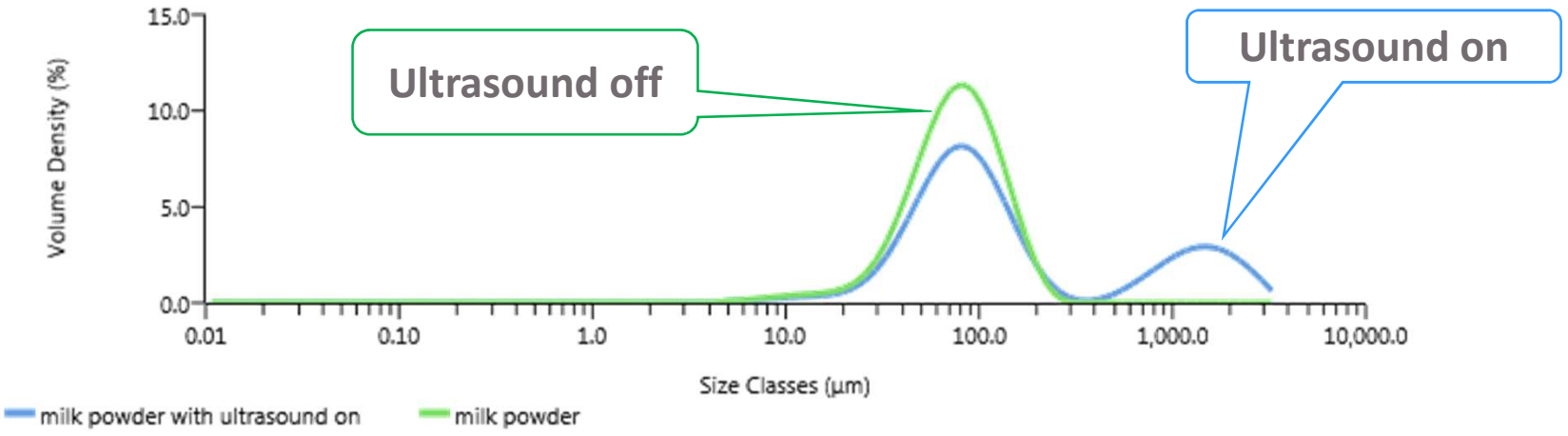
Unexpected large particles: Beam steering

- Ultrasound generates heat in the dispersant
  - Causing scattering signal on low angle detectors
  - Particularly in more volatile dispersants



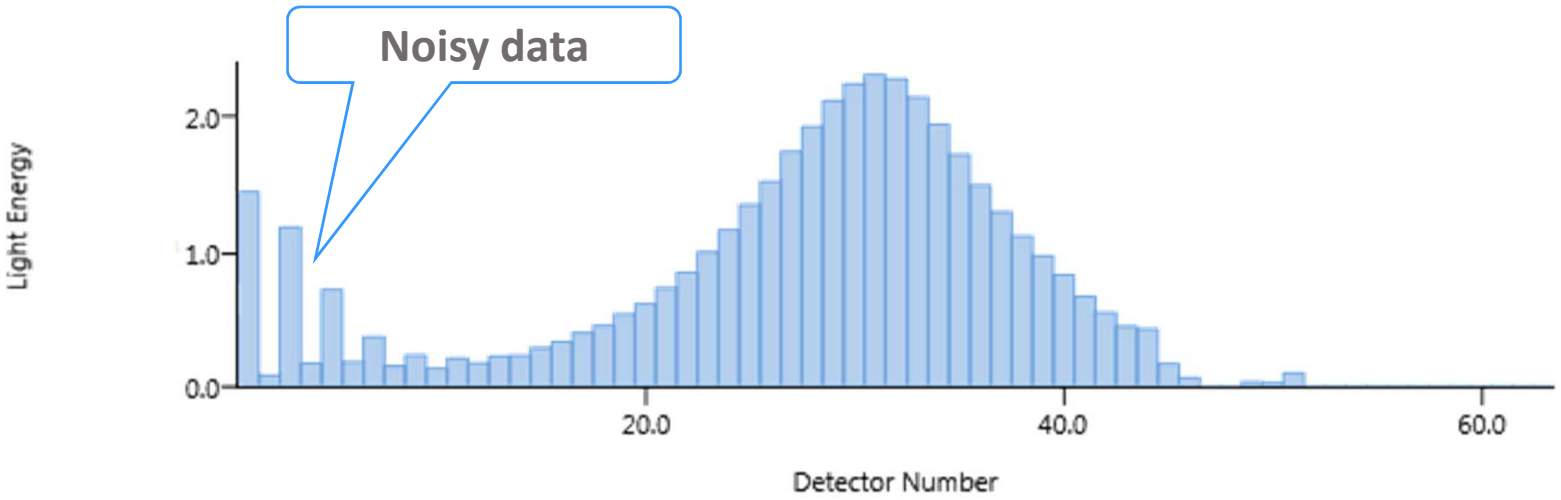
Unexpected large particles: Beam steering

- Ultrasound generates heat in the dispersant
  - Causing scattering signal on low angle detectors
  - Particularly in more volatile dispersants
  - This low angle scattering is interpreted as large particles
- Use a pre-measurement delay after ultrasound
  - Allows thermal gradients to dissipate



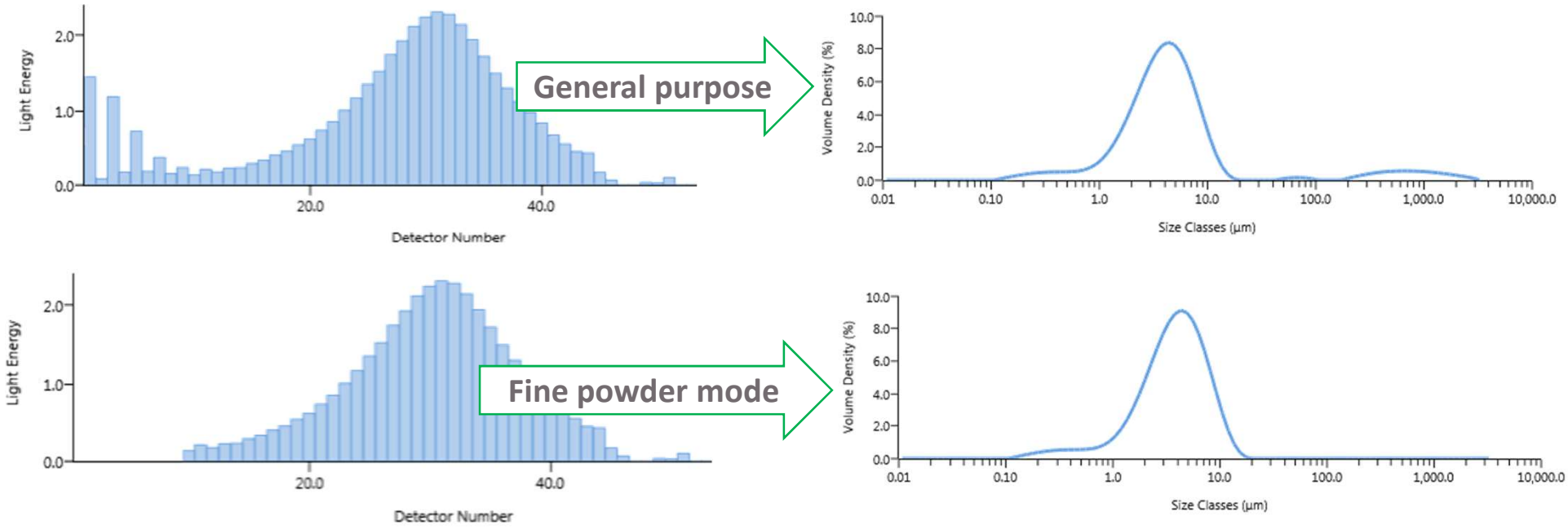
Unexpected large particles: Dry measurements

- Noise on the low angle detectors (1 to 10) can be significant when measuring fine particles dry
  - The noise is caused by thermal fluctuations in the air
  - This noise can be interpreted as large particles
- Measuring a longer background may help
  - Otherwise use the fine powder analysis mode.



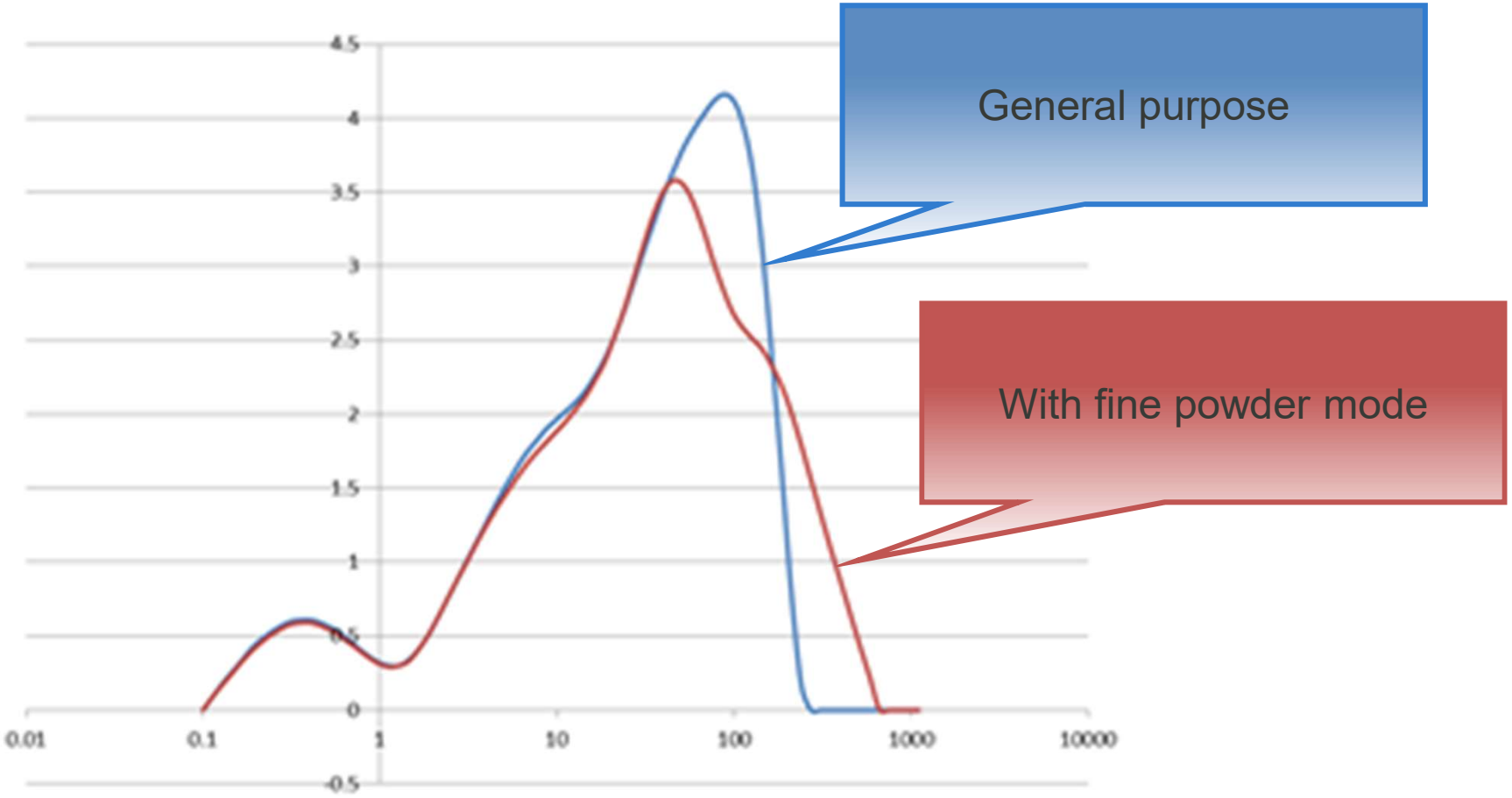
Dry measurements: Fine powder mode

- Fine powder mode removes the first 9 detectors
  - Eliminating the noise which may affect measurement of samples smaller than 10 micron
    - And removing the large modes that it produces
  - Limits the top end of the dynamic range to 600 micron



And when fine powder mode is not appropriate…

- … strange results can be generated.



Further considerations for dry measurements

- Check:
  - Are the cell windows clean?
  - Is the air pressure correct?
  - Does the air filter need changing?
    - Is there oil droplet contamination or moisture in the air supply?
  - Is the feeder earthed against static electricity ?
  - Is the vacuum bag full?

- Is the sample flow even?
  - if the sample obscuration is high, try lowering the feed rate or adjusting the hopper height
  - try changing the height of the hopper, different basket, ball bearing
  - try a different feed tray: often one tray design will deliver a more even sample flow for a particular material

- Use Fine Powder Mode when material less than 10 microns is present

Summary - data quality

- Background data
  - Make sure that:
    - Material is not stuck to the cell windows
    - There is no dispersant contamination
    - There are no thermal gradients
    - That the system has been properly aligned

- Sample data
  - Check that
    - There are reasonable signal to noise levels
    - There is no multiple scattering
    - There is no negative data
    - There is no noisy data
    - The inner detector data is free from castellation
    - There is no beam steering