Training Mastersizer 3000

For Attendees

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Outline

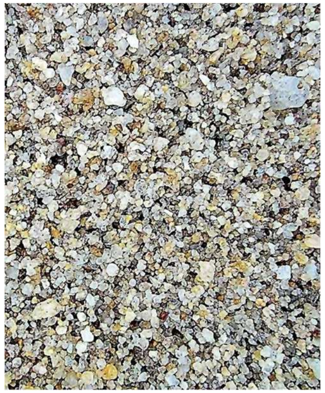
- Introduction to particle sizing
- Introduction to laser diffraction
- Light Scattering Theory
- Mastersizer 3000 component (liquid and dry dispersion unit)
- Mastersizer 3000 : Data quality
- Mastersizer 3000 : Understand size distribution-result reporting

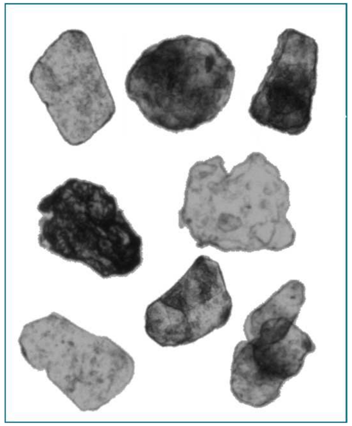
Introduction to particle size

What is a particle?

Particle size are everywhere.

- **A particle can be defined as** : a minute portion, piece, fragment or amount of matter
- **Naturally occurring examples include**: Beach sand, soil, clay, pollen, dust, smoke, fog



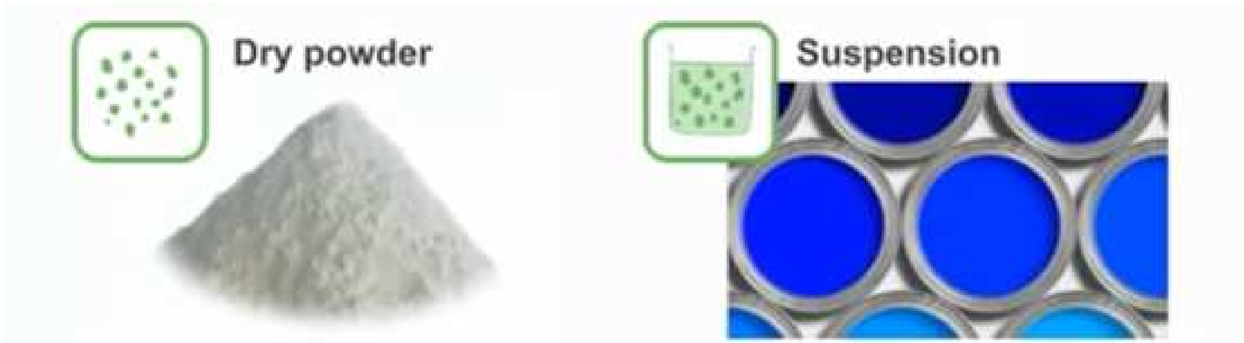


Introduction to particle size

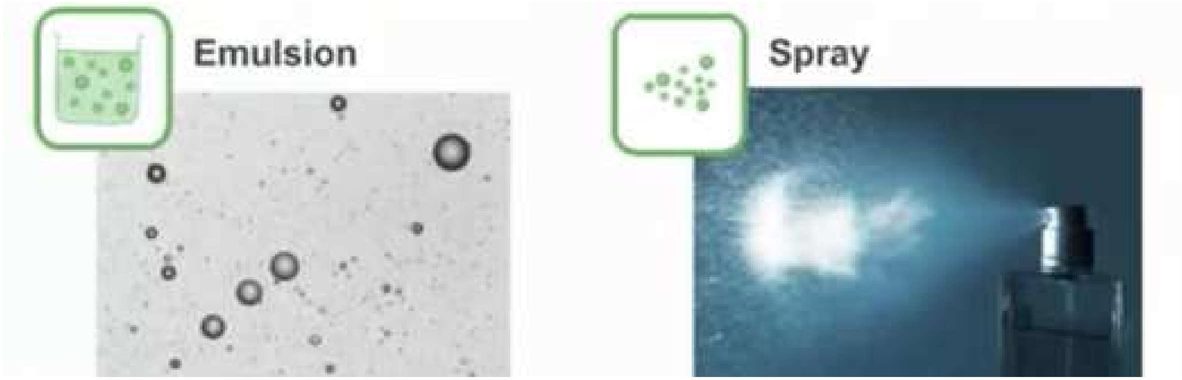
What is a particle?

In general particle are

- **Solid particle**



- **Liquid Droplet**



Light scattering phenomena

Why measure particle size?

Better control of product quality

- Charge higher premium for product
- Reduce customer rejection rates

Better understanding of products and processes

- Improve product performance
- Optimise efficiency of process to reduce costs eg. Milling/grinding
- Increase output/improve yield
- Stay ahead of the competition

Influenced by particle size







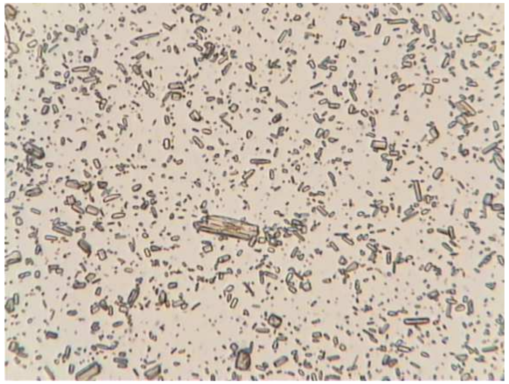
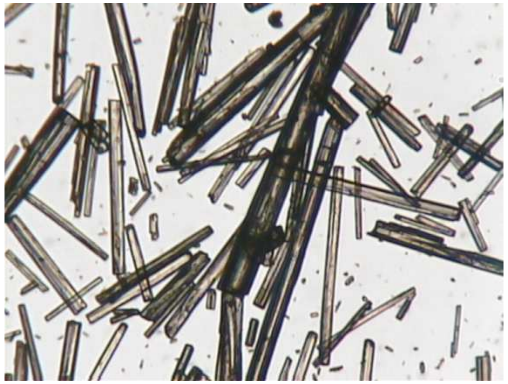
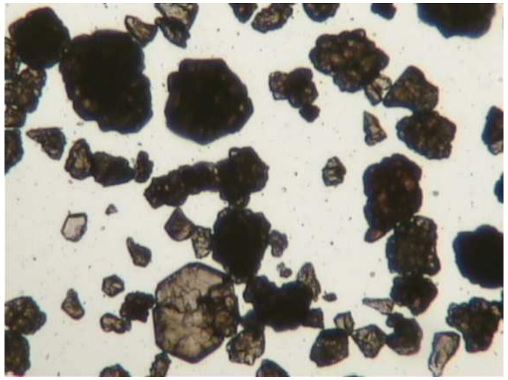
- Reactivity or dissolution rate - e.g. tablets
- Stability in suspension – e.g. paints
- Efficacy of delivery – e.g. asthma inhalers
- Texture and feel – e.g. food ingredients
- Appearance – e.g. toners and powder coatings
- Flowability and handling – e.g. granules
- Viscosity – e.g. suspensions and slurries
- Packing density and porosity – e.g. ceramics

1988 1998 2011

- Laser diffraction measures particles in the size range from **nanometres to millimetres**
- **MS3000 →** 10 nm – 3500 µm (**MS3000E** 0.1 micron – 1000µm)

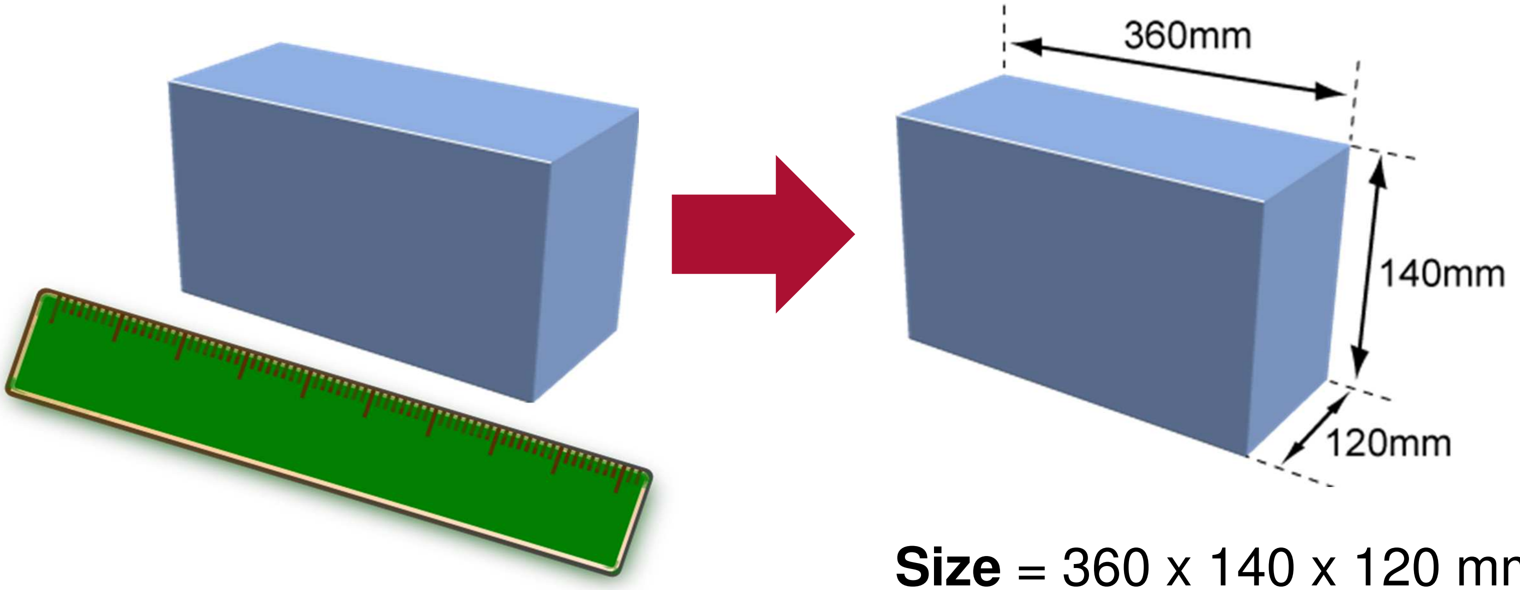
Introduction to particle sizing

People normally associate particles with images such as these…

…but what are their sizes?

Q : Which number is used to indicate the particle size of this box ?



Size = 360 x 140 x 120 mm

It is not possible to describe 3-dimensional object

with a single number except it is a sphere shape.

Concepts – Equivalent spheres

Malvern Panalytical DKSH

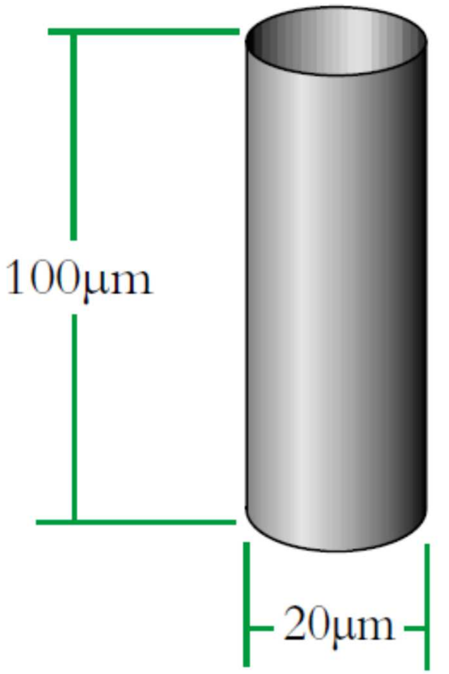


Figure Cylinder

Find Volume

$$\text{Volume}_{(cylinder)} = \pi r^2 h = \mathbf{10000\pi}$$

$$\mathbf{10000\pi} =$$

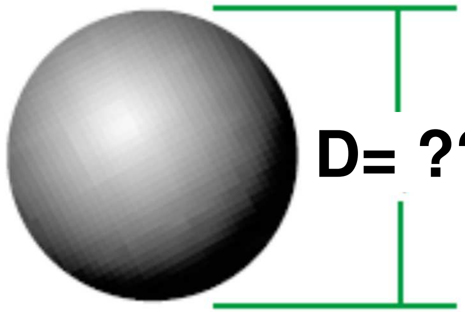


Figure Sphere

Find " r "

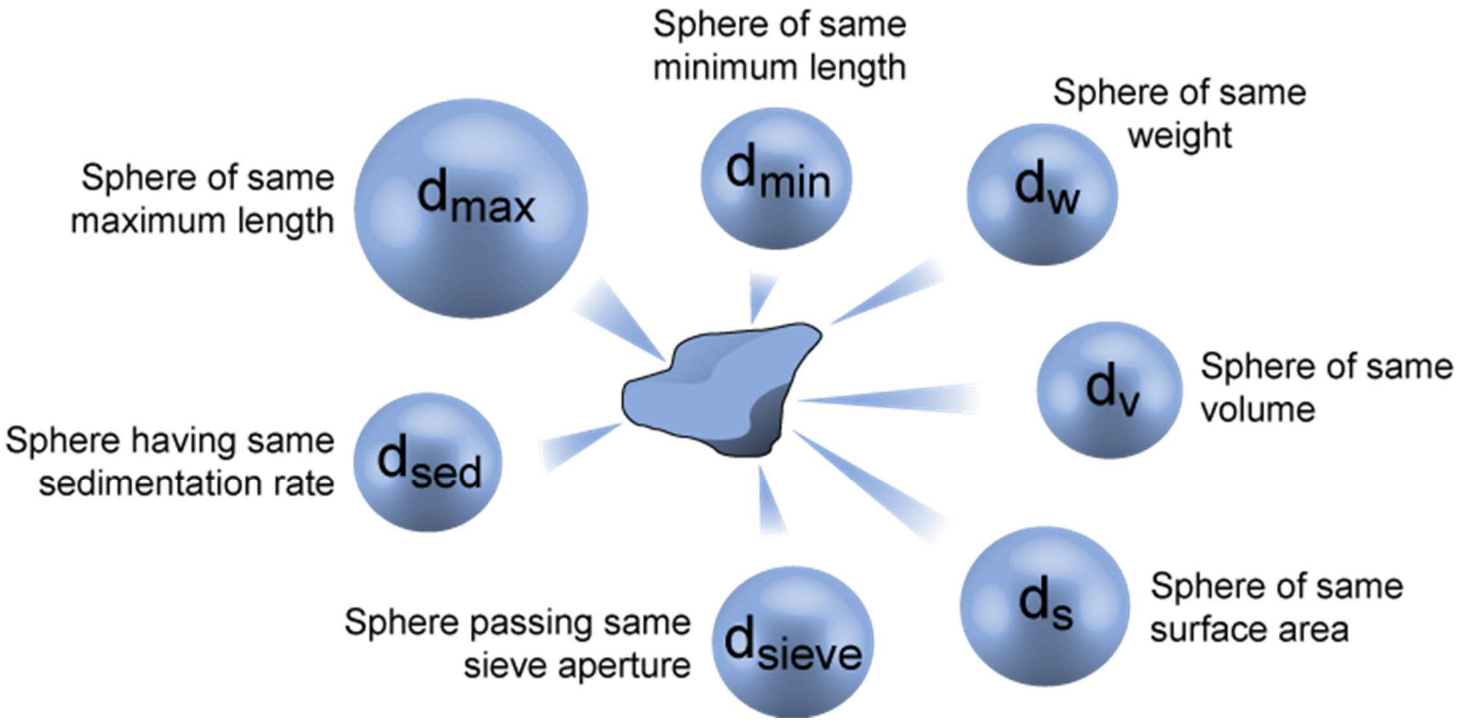
$$10000\cancel{\pi} = \frac{4}{3}\cancel{\pi}\, r^3 = 19.5\ \mu m$$

$$D = 2r = \text{"39 µm"}$$

Where; h = height of sample (m)
D = diameter of sample (m)
r = radius of sample (m)

Concepts – Equivalent spheres

All particle size analysis techniques measure some property of a particle and reports results as the equivalent spherical diameter based on this measured parameter

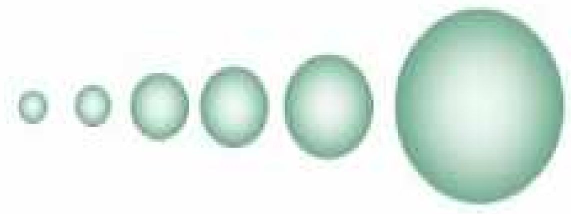


Introduction to particle sizing

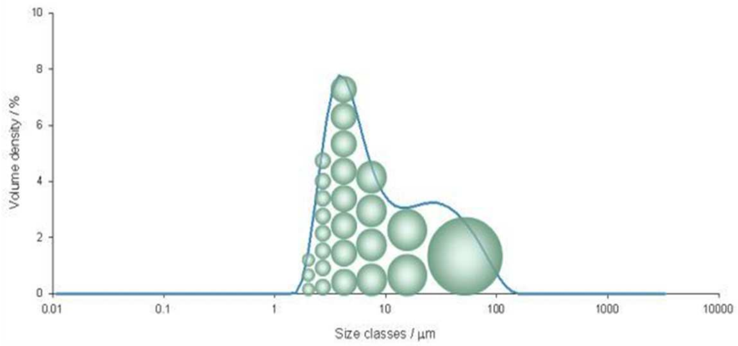
The particle size distribution

- Only a perfectly monodisperse sample will have particles all with exactly the same size

- Most real world samples will contain a distribution of particle sizes



- Laser diffraction measurements produce volume based particle size distributions

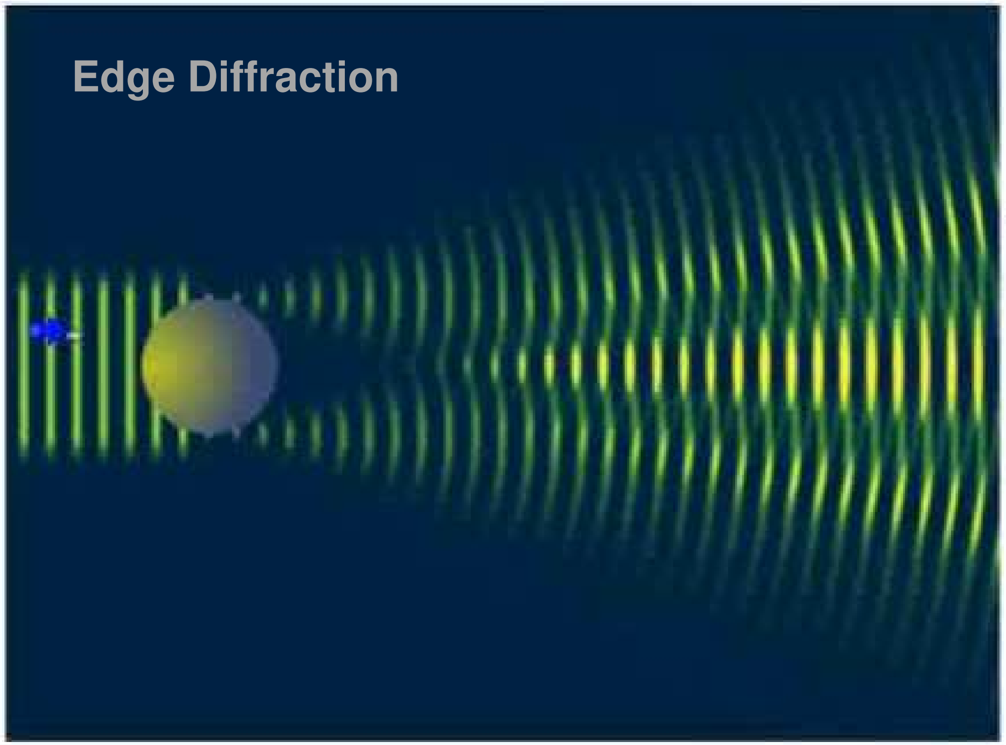


How does it work?

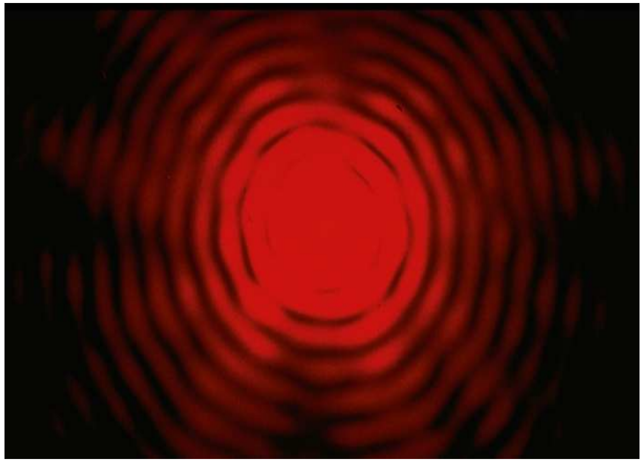
A laser diffraction instrument does not measure particle size!

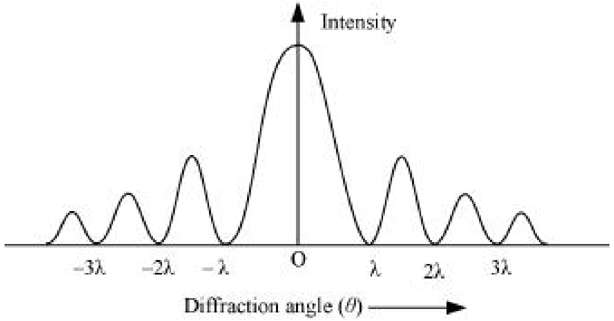
Laser diffracti particles!

Transmit a laser through a dispersion of particles



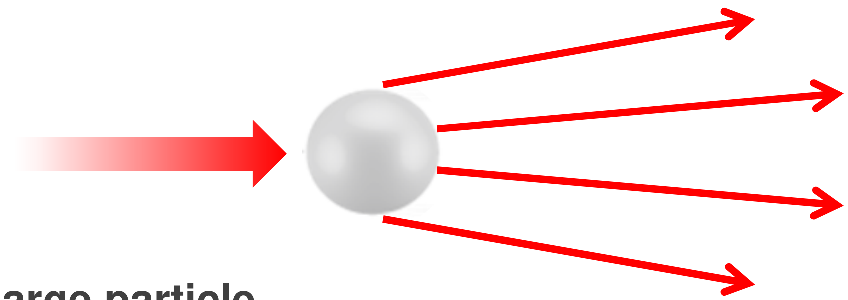
Scattering pattern



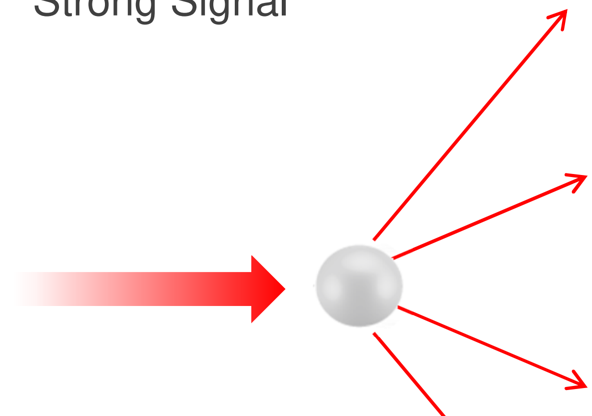


Intensity distribution

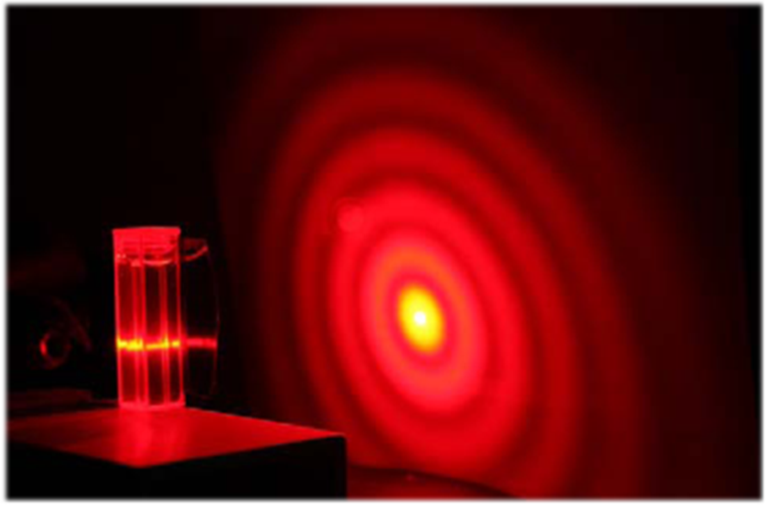
Dependence of light scattering on particle size



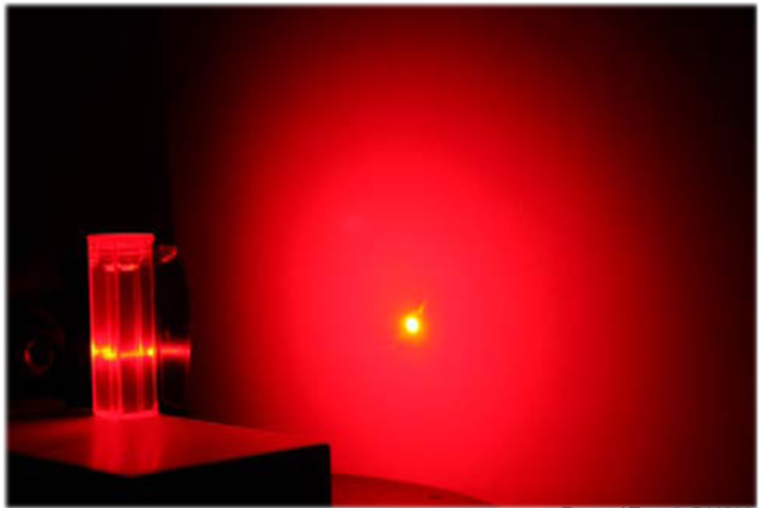
Large particle
- ✓ Low angle scattering
- ✓ Strong Signal



Small particle
- ✓ Large angle scattering
- ✓ Weak signal

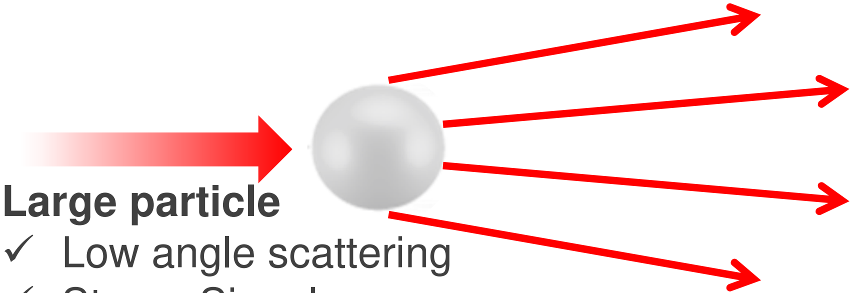


5 microns

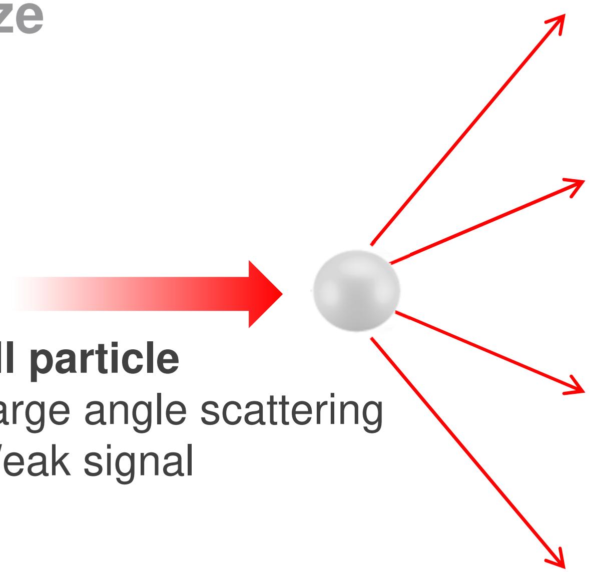


800 nanometres

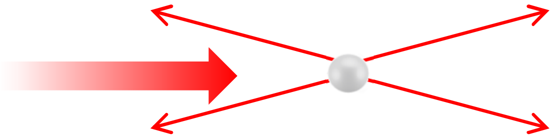
Dependence of light scattering on particle size



Large particle
- ✓ Low angle scattering
- ✓ Strong Signal



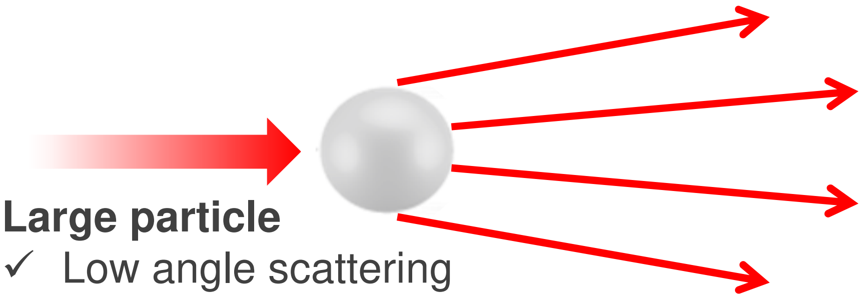
Small particle
- ✓ Large angle scattering
- ✓ Weak signal



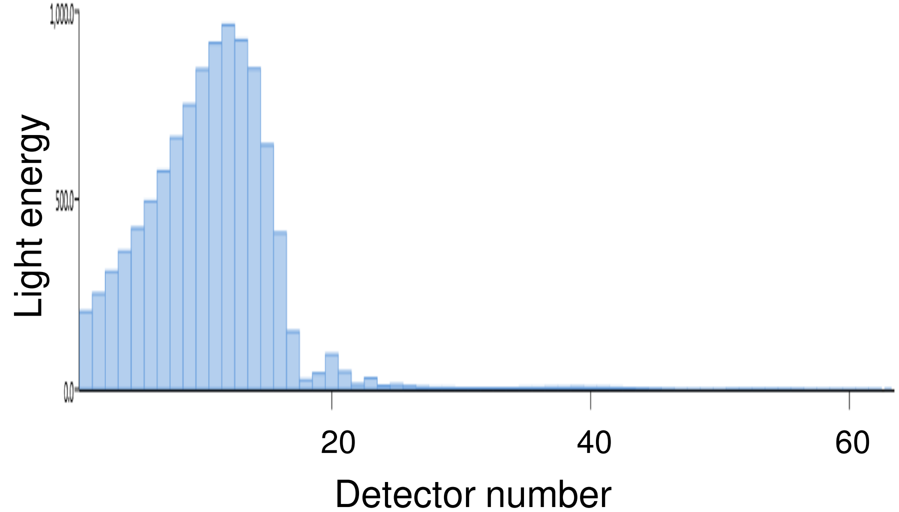
Particle diameter < λ/10
- ✓ Intensity is independant of scattering angle
- ✓ Very weak signal

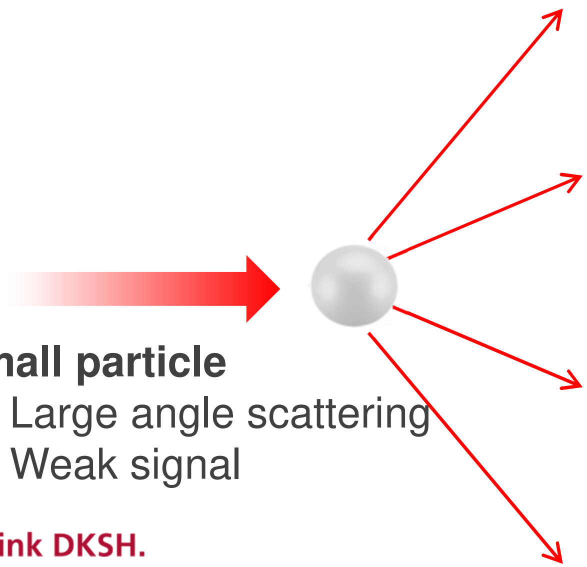
Malvern Panalytical DKSH

Dependence of light scattering on particle size

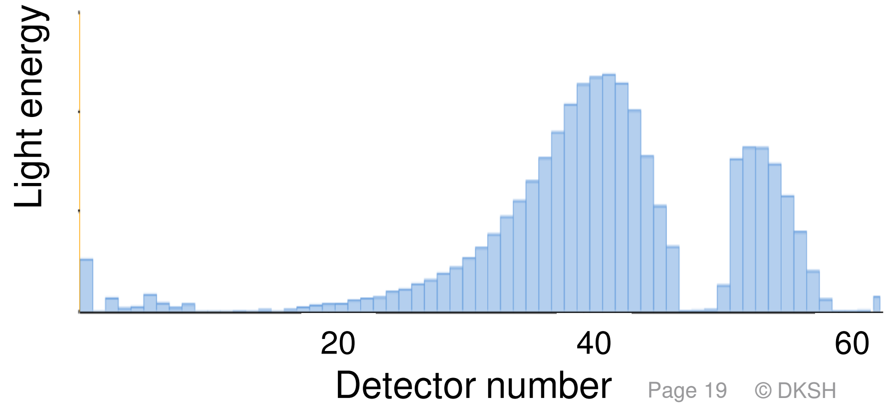


Large particle
- ✓ Low angle scattering
- ✓ Strong Signal

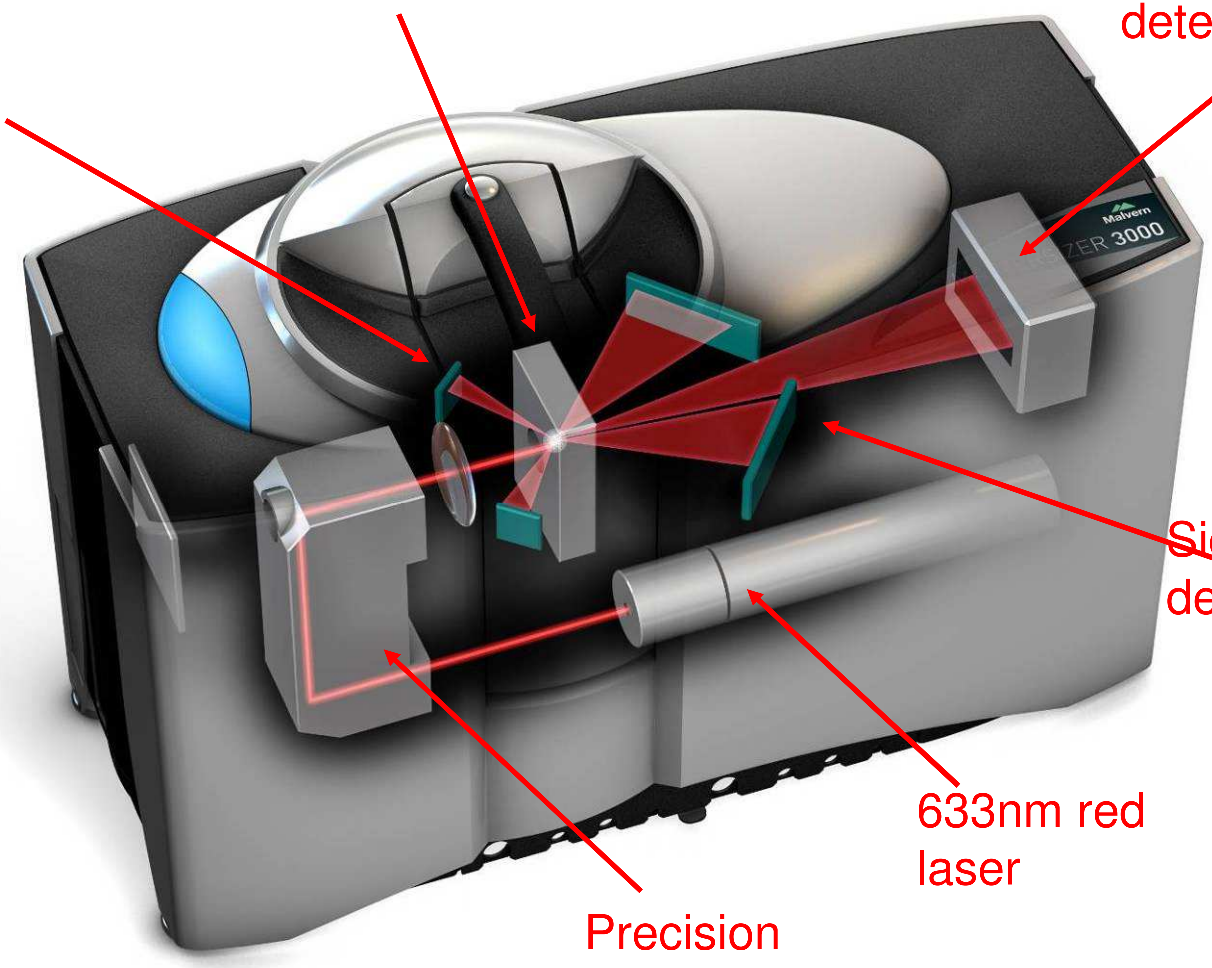




Small particle
- ✓ Large angle scattering
- ✓ Weak signal

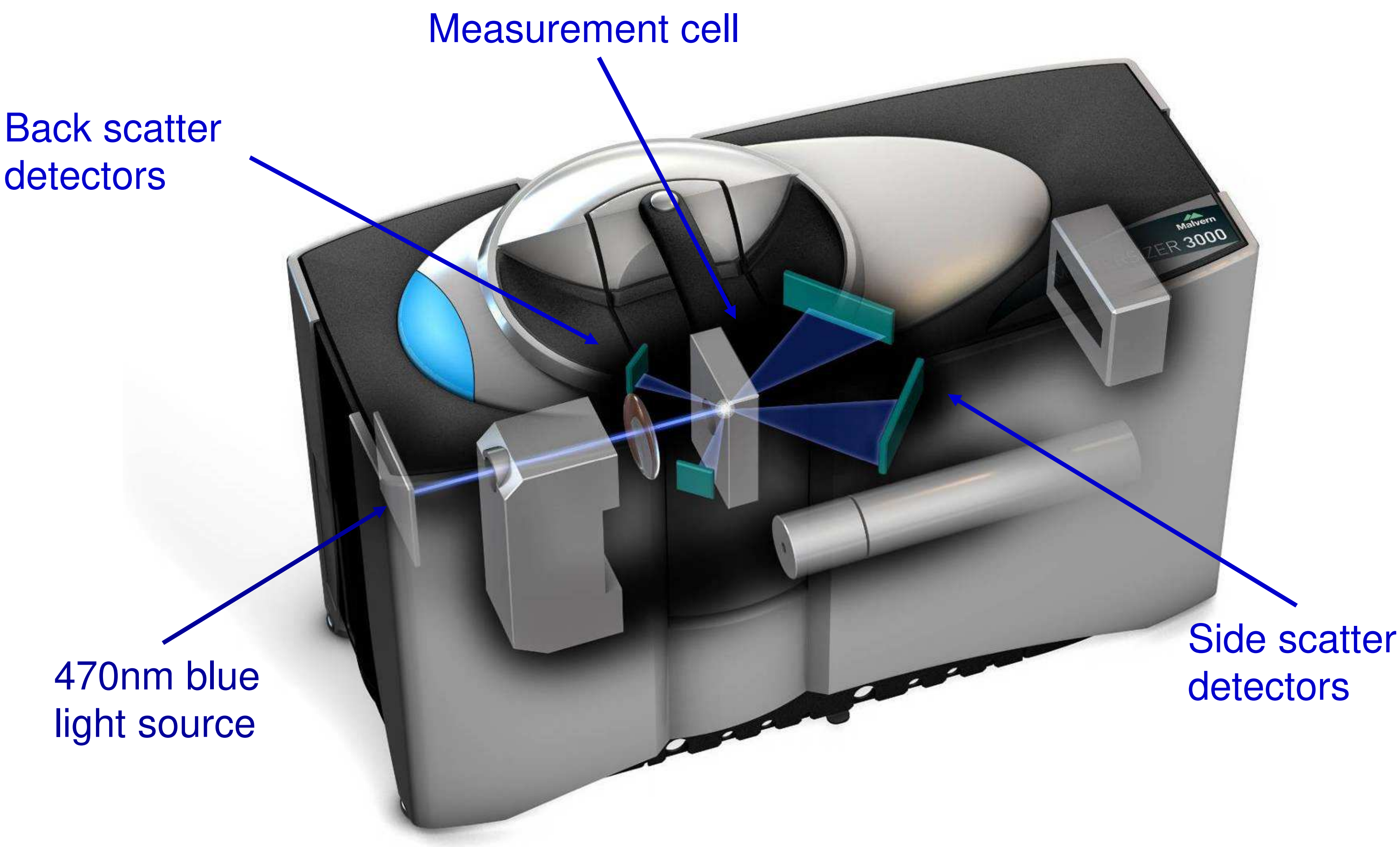


Mastersizer 3000 optics
Red light measurements

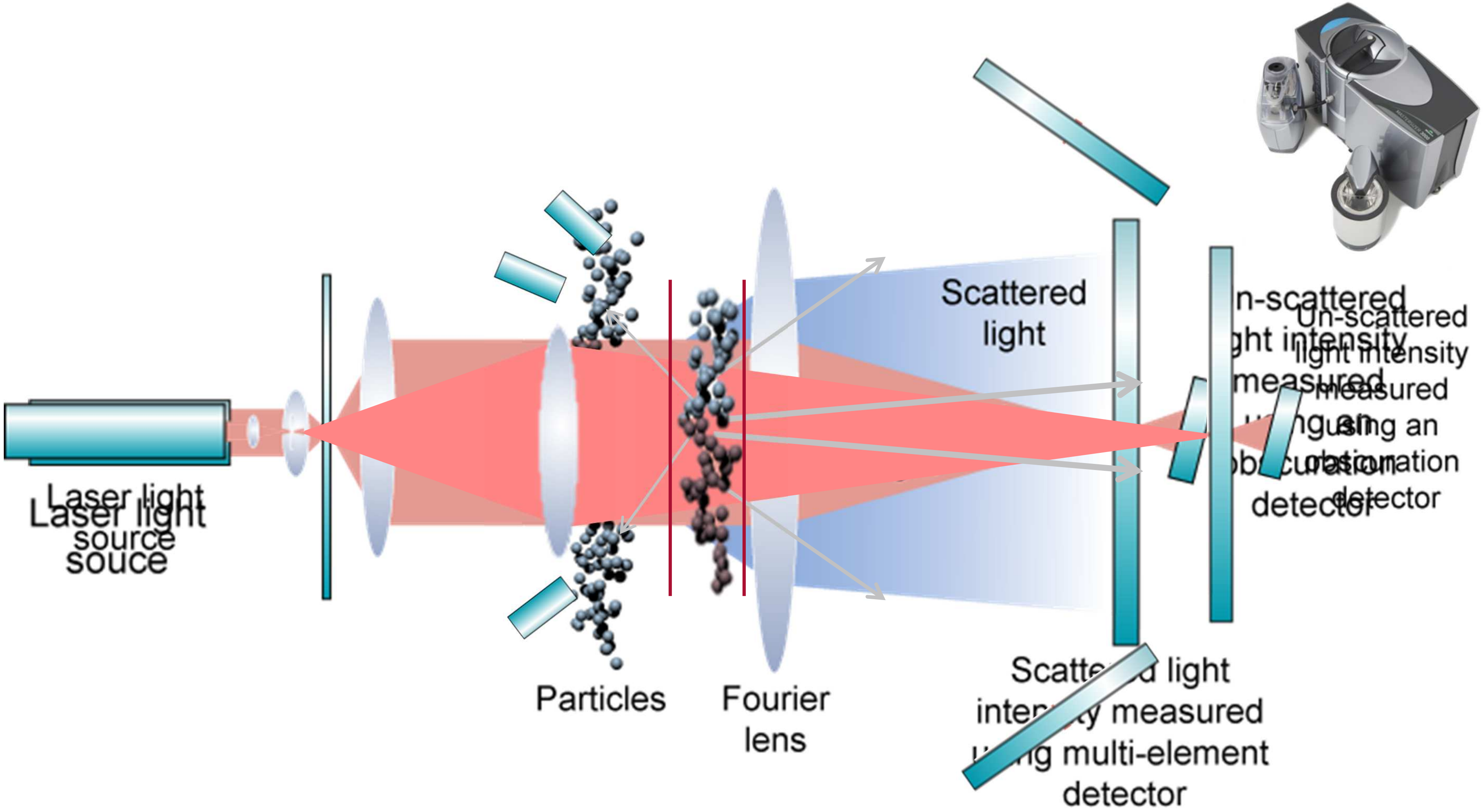


Mastersizer 3000 optics

Blue light measurements

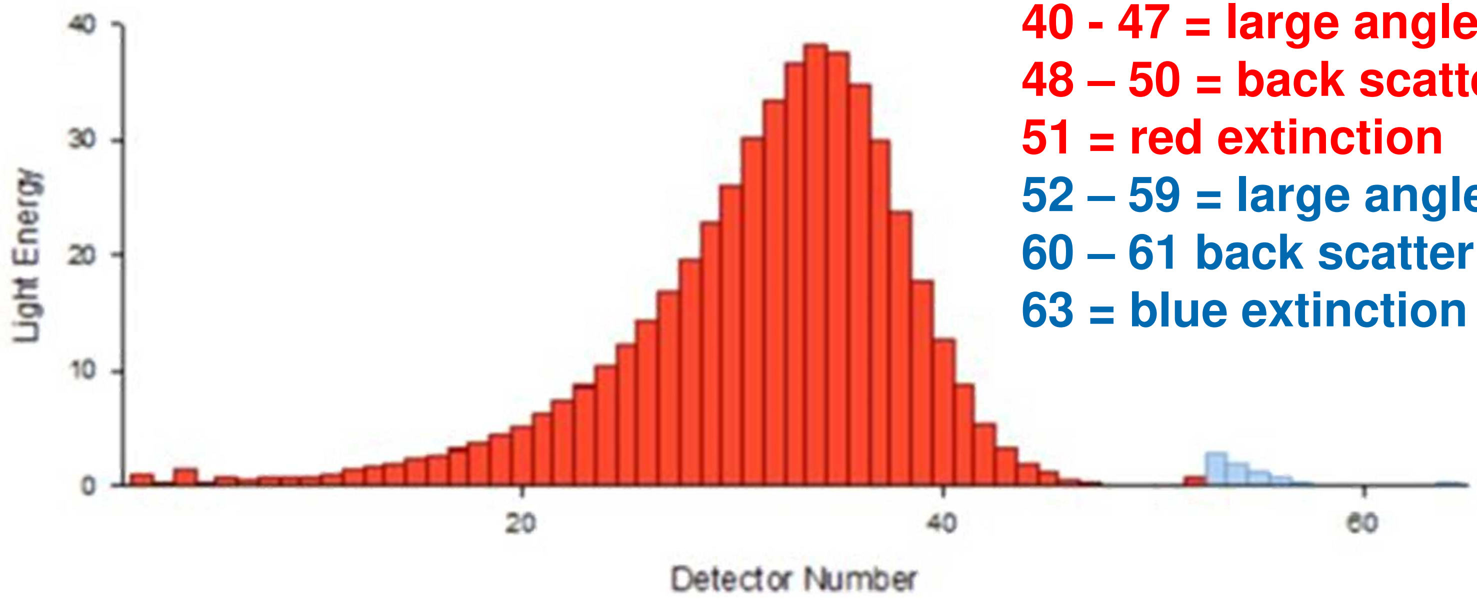


Laser diffraction – how does it work?



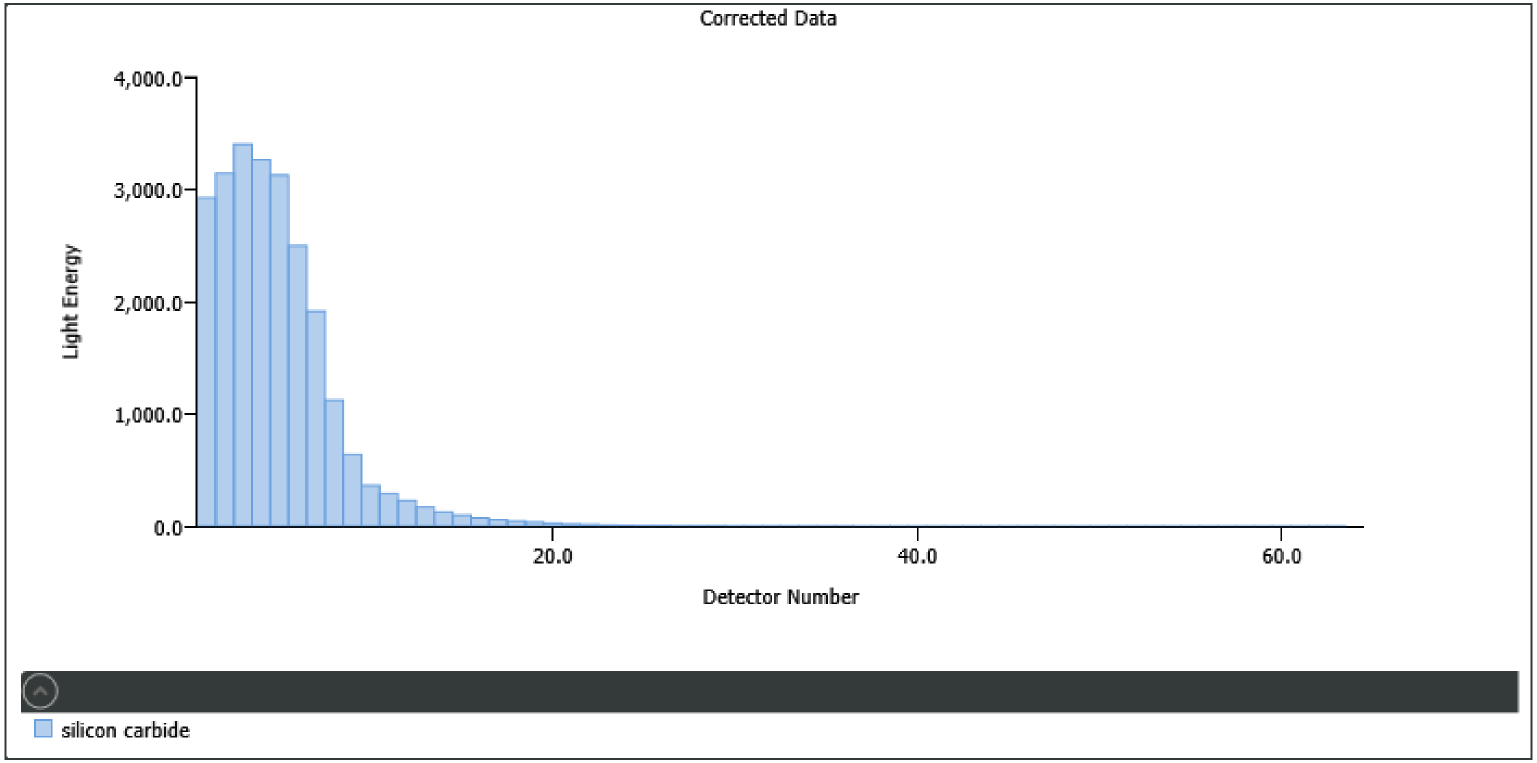
The measured scattering data

The angular scattering data is presented in real-time in the measurement window of the Mastersizer software.
Detector numbers correspond to increasing scattering angle…



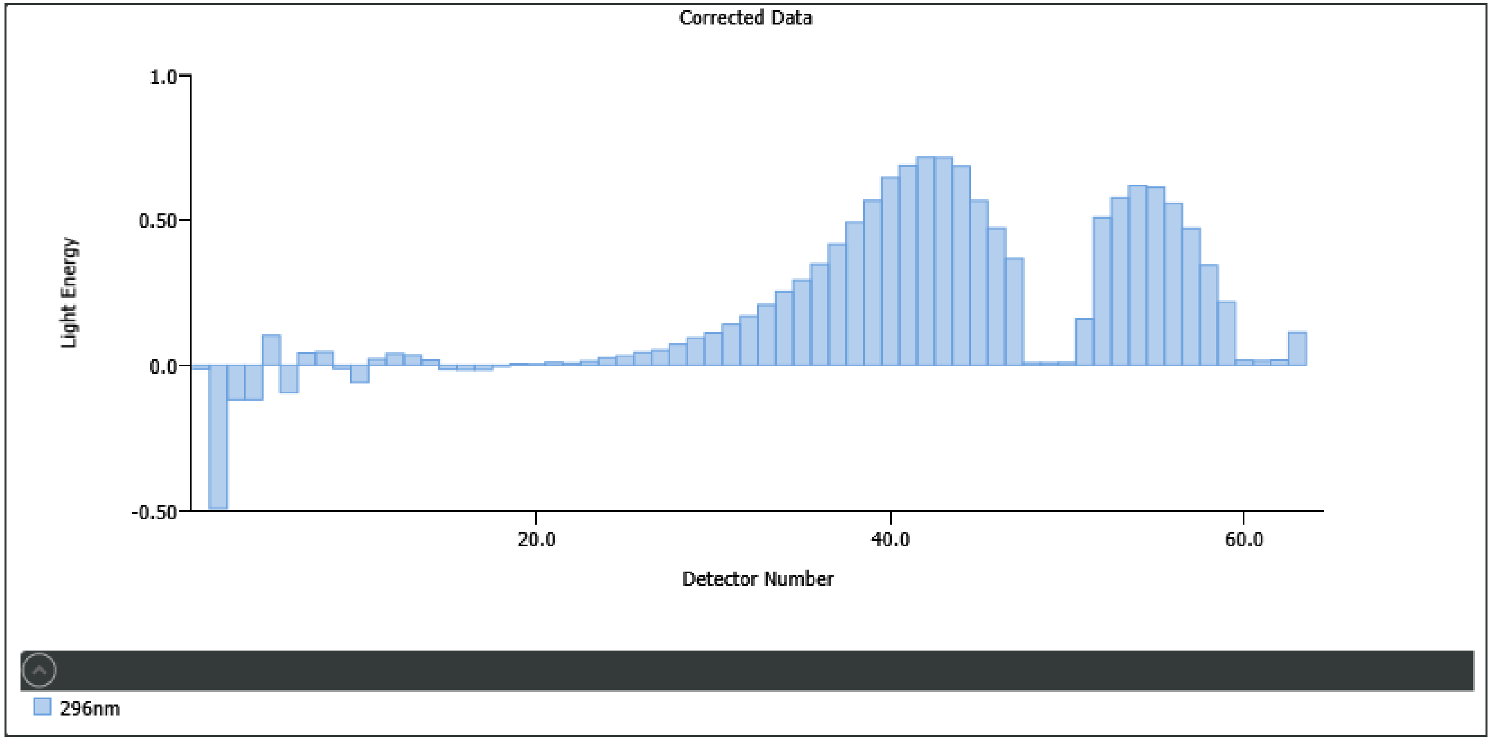
1-39 = focal plane
40 - 47 = large angle red
48 – 50 = back scatter red
51 = red extinction
52 – 59 = large angle blue
60 – 61 back scatter blue
63 = blue extinction

Typical Data Set – Coarse Particles



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

Typical Data Set – Sub-Micron Particles



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

Light Scattering Theory

How do we know the size from light scattering?

Scattering models

- **Laser diffraction** requires **a model** that accurately defines the light scattering behaviour of all particles
- There are currently two popular choices available

Fraunhofer Approximation | ***Mie Theory***

Joseph von Fraunhofer
(1787-1826)

Gustav Mie
(1869-1957)

Since 1986 the preferred model has been Mie Theory which correctly predicts the scattering at all wavelengths of light at all angles.

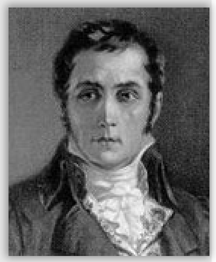
.

How do we know the size from light scattering?

Scattering models

Fraunhofer

simple and quick to calculate



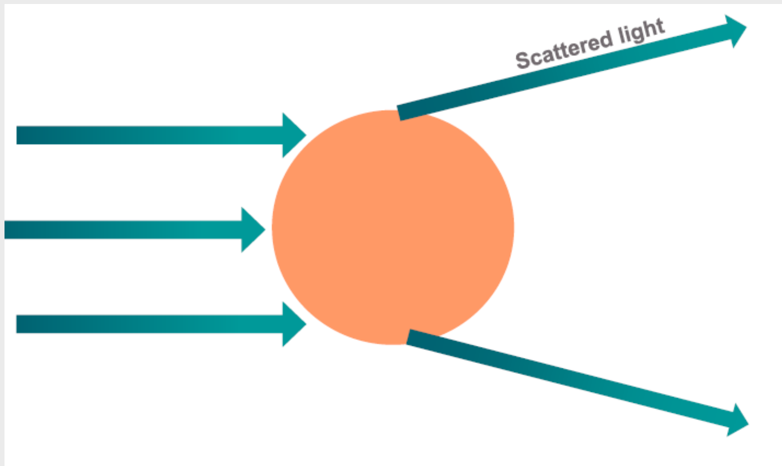
Assumption

- Assuming the particles are discs
- Assuming it is a two phase system

The particles are opaque

The angle of scattered light is small

No need optical parameter



Mie Theory

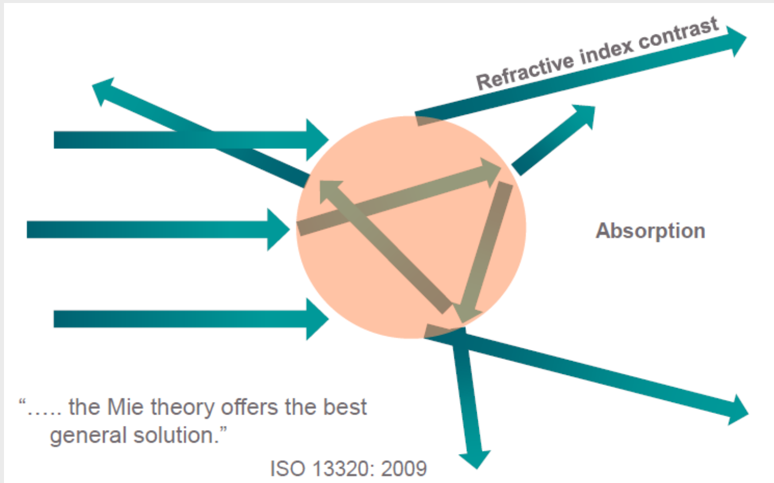
For particles smaller than about 25µm

Mie theory offers the best general solution

Assumption

- Assuming the particles are spherical
- Assuming it is a two phase system

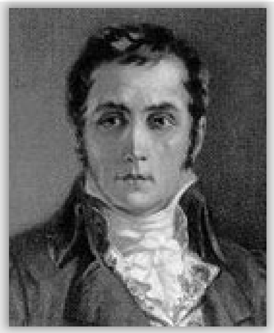
Need optical parameter : RI and AI



How do we know the size from light scattering?

Scattering models

Fraunhofer



Claimed Advantage

- *"No need to know the optical properties of your material." Which implies that optical properties are required for Mie theory.*

Disadvantages

- *Will produce incorrect answers when Particles are <25 µm*

Mie Theory

** 3 properties need to be known.

- **The Refractive index (RI) of dispersant.**
- **The Refractive index (RI) of sample.**
- **The Absorption index (AI)of sample.**

Claimed Advantage

- Valid for all wavelengths of light and all particle sizes
- Predicts the dependence of scattering intensity on particle size
- For particles < 25 µm the Mie theory offers the best general solution.

Disadvantage Optical parameters are needed.

Parameter : Shape

Particle Type

Opaque Particle (Fraunhofer Approximation)

The Fraunhofer Approximation can be used to calculate particle size distributions in cases where the particle size is large and where the particles can be assumed to be completely opaque. It is the easiest mode to use, as you do not have to provide any optical properties in order to calculate a size distribution. However, its use may lead to inaccurate results, particularly for small particles below 50 microns in size or for those which are transparent.

Fraunhofer Theory
No RI and AI need !!

Particle Type

Spherical

This particle type is applicable for particles which are perfectly spherical in shape. For example, it should be selected for polymer latex samples or for emulsions.

This type uses Mie Theory, and therefore requires input of the optical properties of your sample in order to calculate a particle size distribution. The advantage of this is that it provides the possibility of obtaining accurate size distributions for all particle sizes.

Particle Type

Non-Spherical

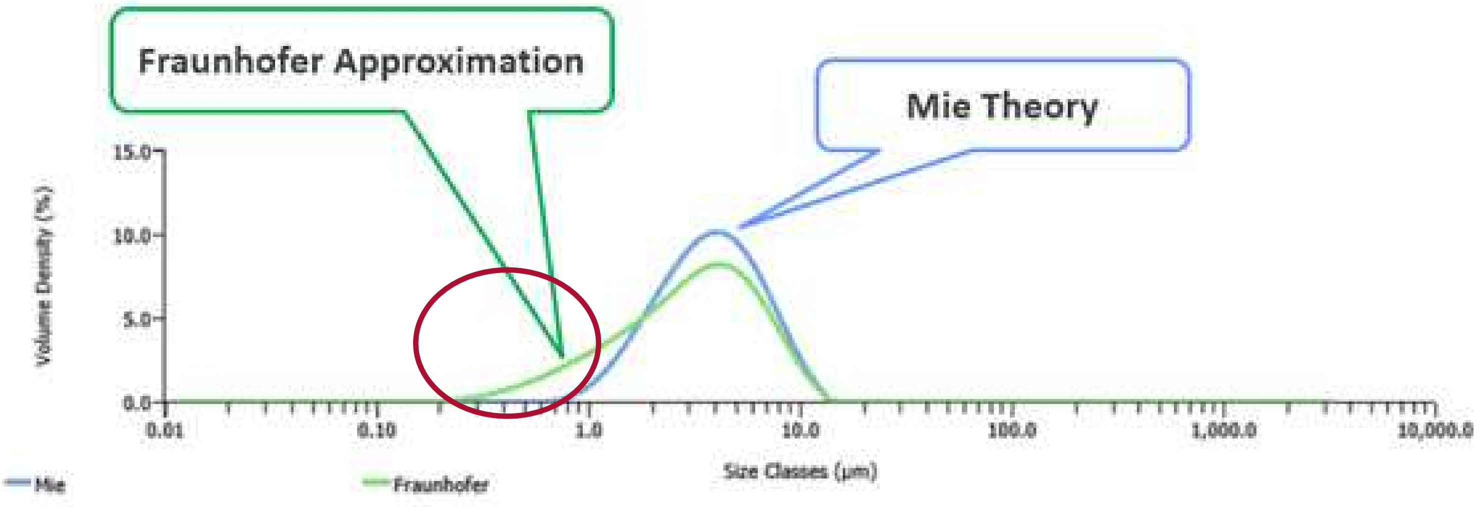
This particle type is applicable for particles which are irregular in shape, or have a rough surface structure. For example, it should be selected for milled or crushed materials.

This type uses Mie Theory, and therefore requires input of the optical properties of your sample in order to calculate a particle size distribution. The advantage of this is that it provides the possibility of obtaining accurate size distributions for all particle sizes.

Mie Theory
RI and AI need !!

How do we know the size from light scattering?

Comparing the results Fraunhofer Vs Mie



The **ISO13320-1** standard for laser diffraction states:

*For particles smaller than about **25 μm** Mie theory offers the best general solution…*

'If the Fraunhofer approximation is applied for samples containing an appreciable amount of small, transparent particles, a significantly larger amount of small particles may be calculated.'

Optical properties
What are they?

For the application of **Mie theory**, 3 properties need to be known.

These are:

- **The Refractive index of the dispersant**.
- **The Refractive index of the sample material**
- **The Absorption index of the sample material**
    - Often referred to as the absorption

'Good understanding of the influence of the complex refractive index in the light scattering from particles is strongly advised in order to apply the Mie theory of the Fraunhofer approximation appropriately.'

ISO 13320-1

Absorption index

- The absorption can be determined by looking at the dispersed sample under a microscope by looking at its
  - **Shape**
  - **Transparency**
  - **Internal structure.**
- Absorption is generally only required to a factor of 10
  - E.g. 0.1 or 0.01 (not 0.023)

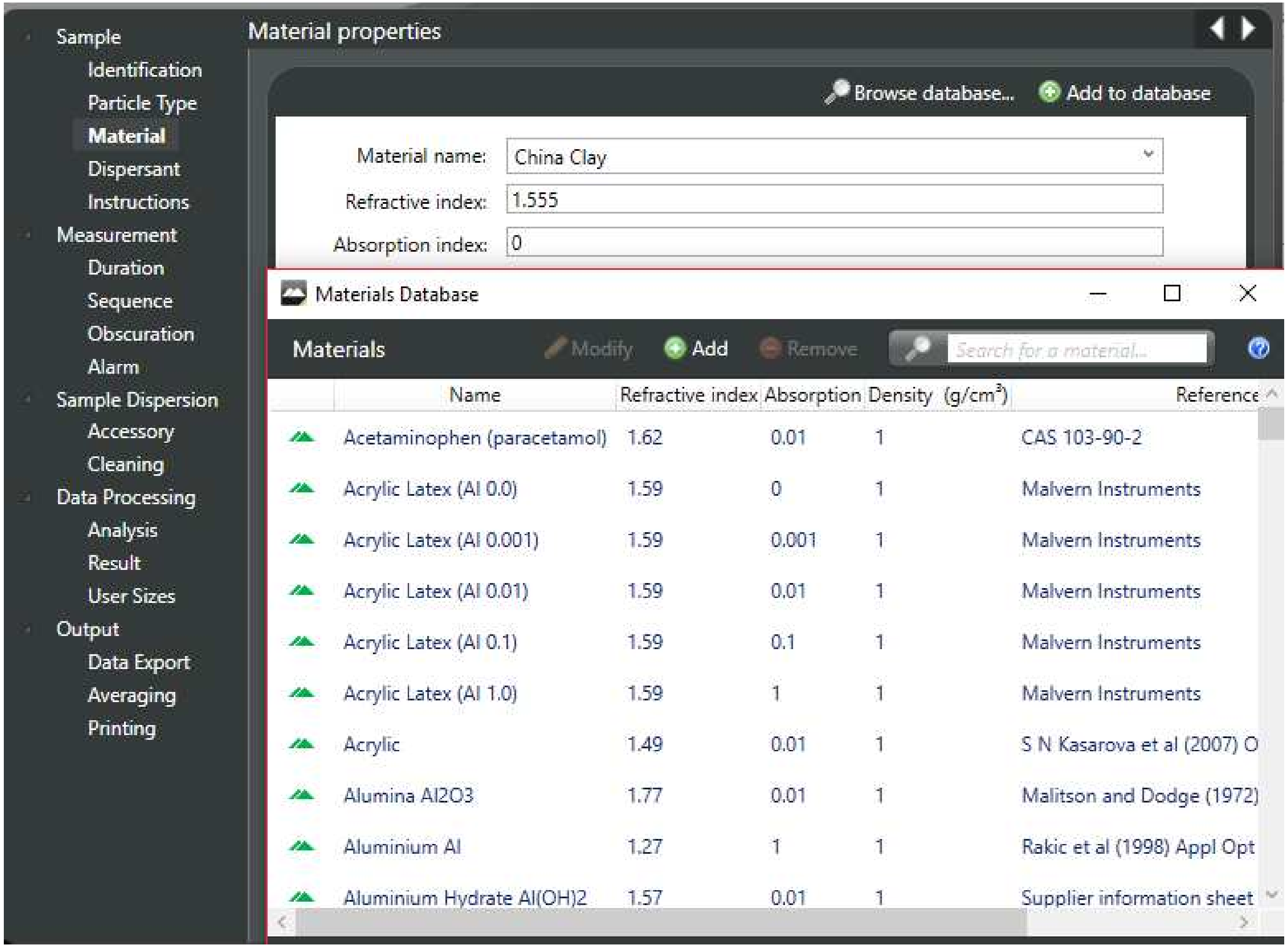
Estimating absorption from particle appearance

Appearance	Imaginary RI	Example
	0	Latices
	0.001	Emulsions
	0.01	Crystalline milled powders
	0.1	Slightly colored powders
	1.0+	Highly colored (complementary) and metal powders

Choosing optical properties

- You can estimate the optical properties based on typical values of similar materials.
  - **A Refractive Index is generally only required to 2 decimal places e.g. 1.42 not 1.4234**
- Some families of similar materials are:
  - **Plastics and elastomers = 1.38 - 1.57**
  - **Organic compounds = 1.4 - 1.7**
  - **Inorganic salts = 1.52 - 1.8**
  - **Metal Oxides = 1.6 - 2.5**
- Use the estimated refractive index as a starting point and examine the fit to confirm the suitability of the value chosen.

Optical properties
Material database



Methods for determining RI

- Four main routes to refractive index information
  - **Reference books and the internet**
    - Appendix of ISO 13320
    - Malvern materials database
    - CRC handbook
    - Manufactures label (for dispersants)
    - Online info
  - **Refractometer measurements**
  - **Microscope observations**
  - **Empirical/semi-empirical models**

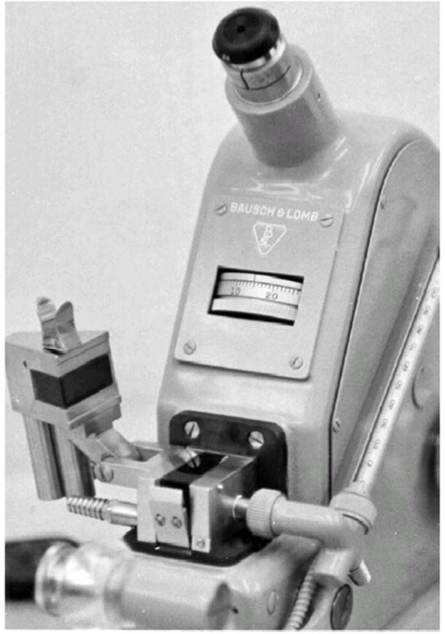
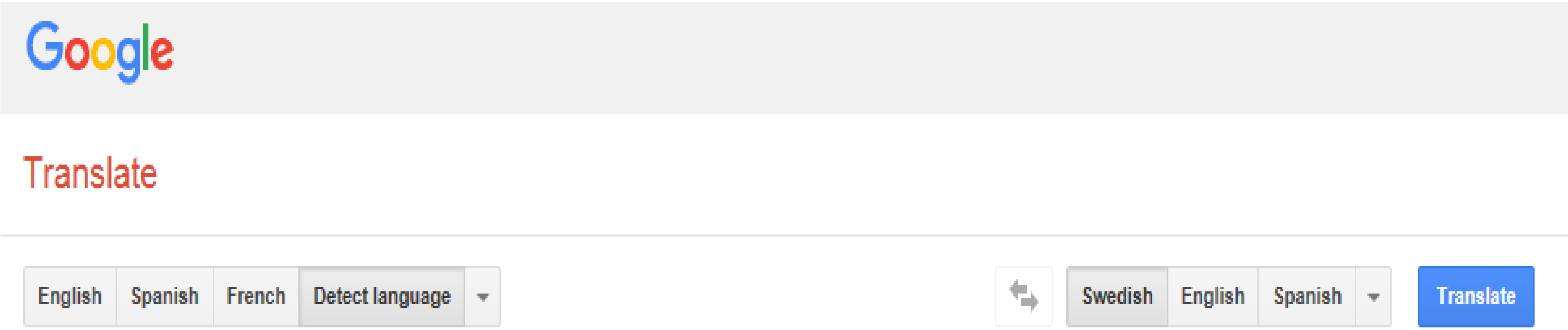
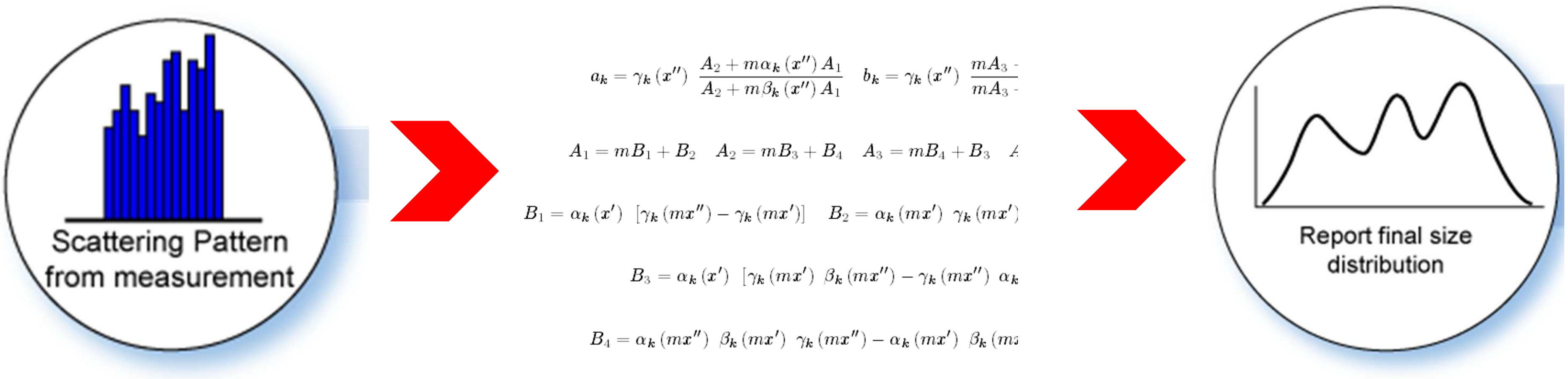


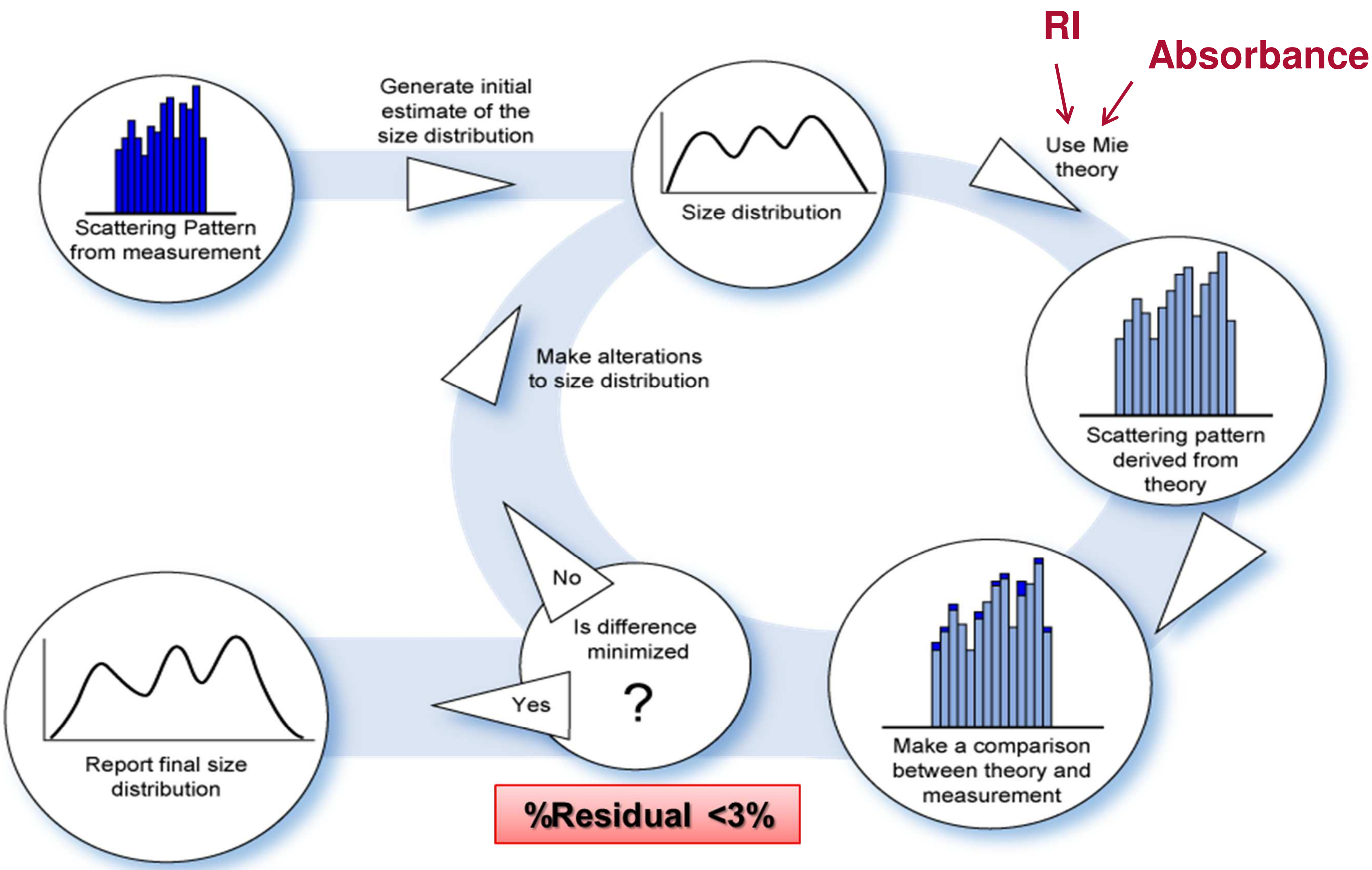
Fig. 2-6. Typical classical Abbe refractometer opened to show prism system.

Translate scattering data to particle size





Calculation of size distribution

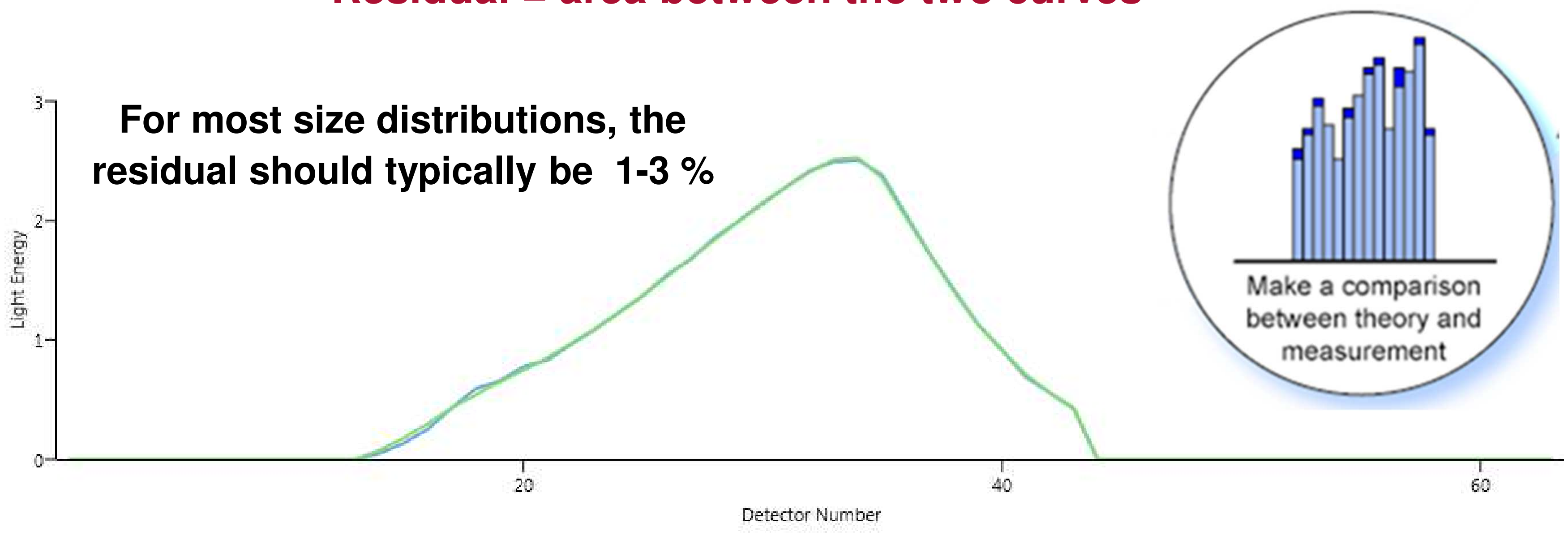


Using the data fit

- Here we see an overlay of the measured scattering data and the data predicted based on the optical properties.
- The precision with which these two curves overlay is known as the “data fit”
- The residual quantifies how good the fit is

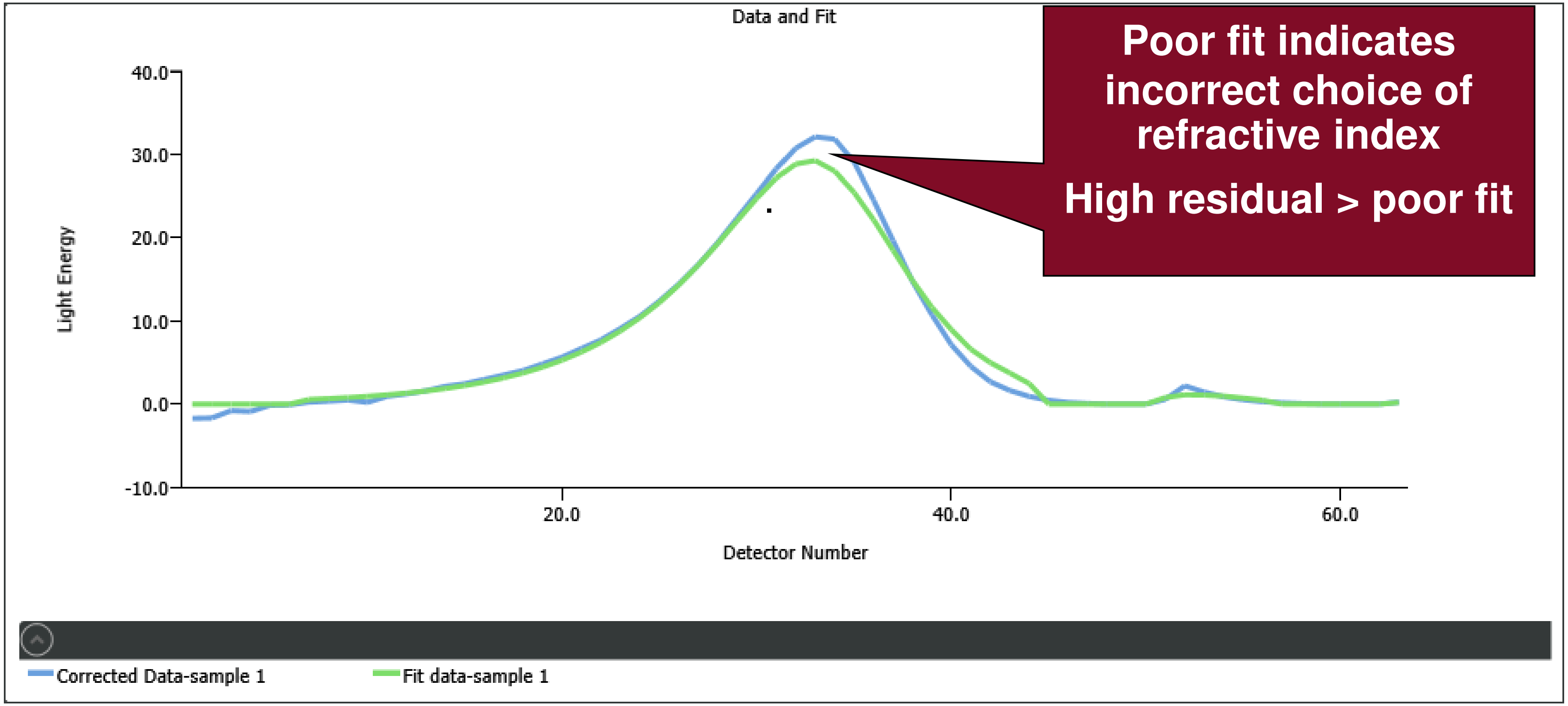
Residual = area between the two curves

For most size distributions, the residual should typically be 1-3 %



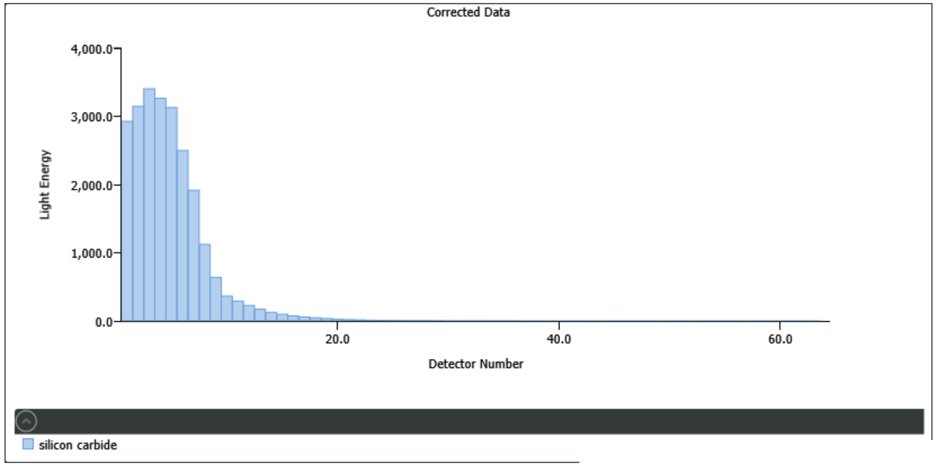
Inspecting fit data
refractive index

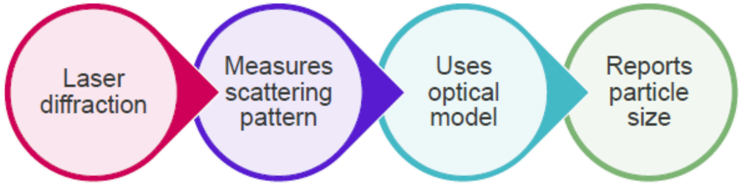
A poor fit in the low detectors (<40) indicates an incorrect choice of refractive index

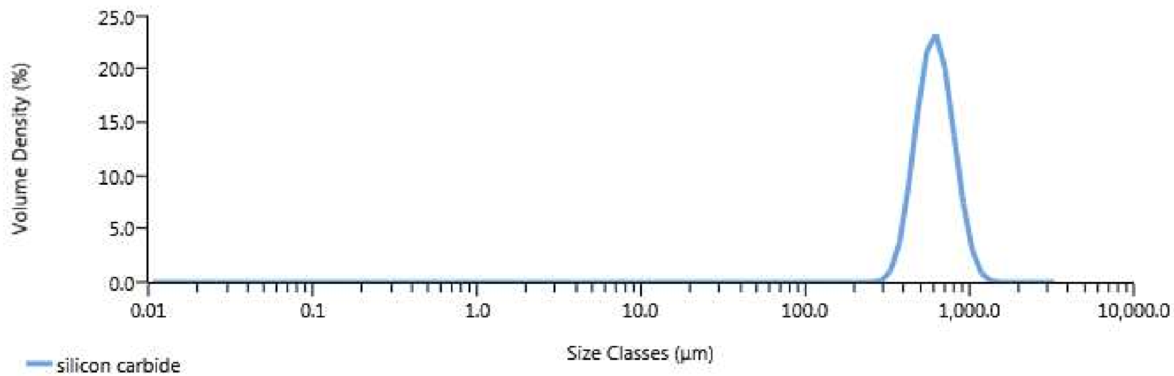


Laser diffraction to particle size

Typical Data Set – Coarse Particles



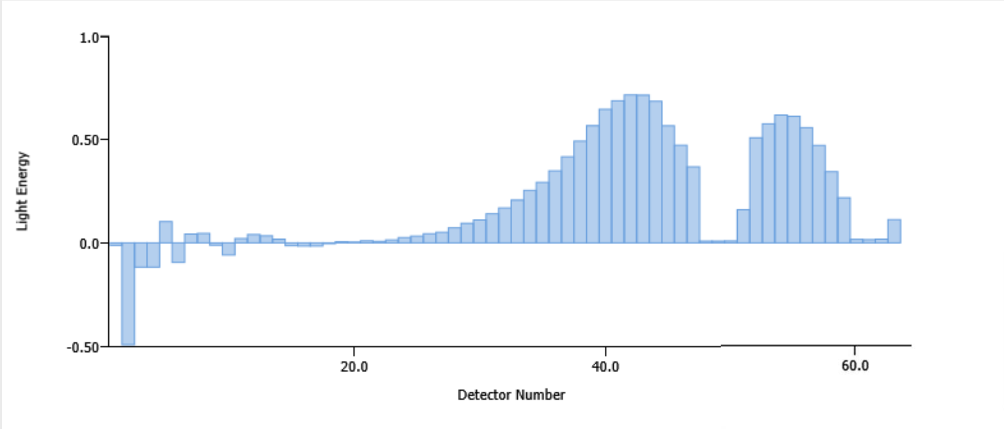


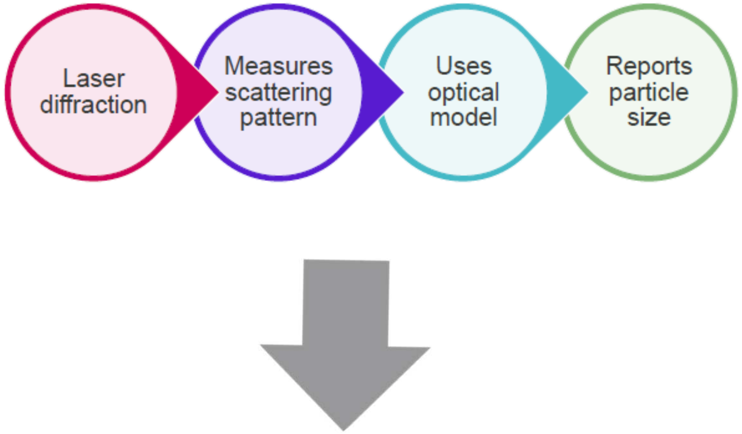


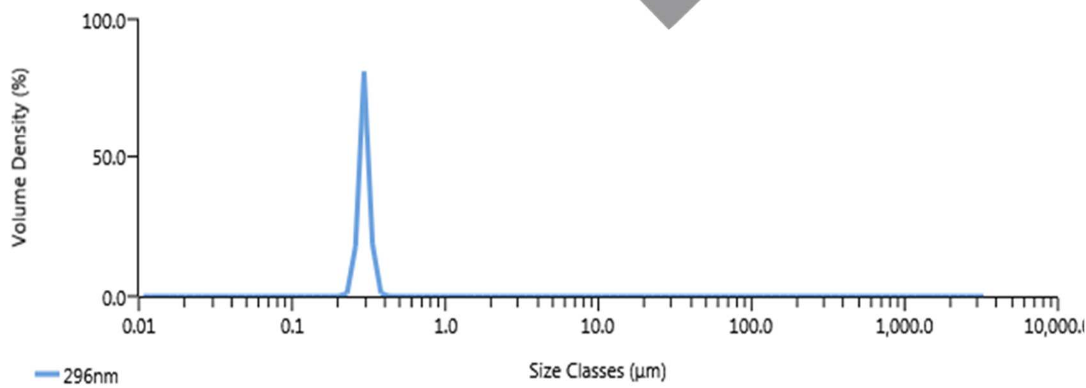
- Large particle scatter at low angles.
- Scattering data is concentrated in the low angle detector
- Region which corresponds to low detector numbers

Laser diffraction to particle size

Typical Data Set – Sub-Micron Particles







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

Mastersizer 3000

Sample Dispersion unit

Wet Dispersion

Dry Dispersion

Wet Dispersion

Mastersizer3000

Wet Dispersion unit

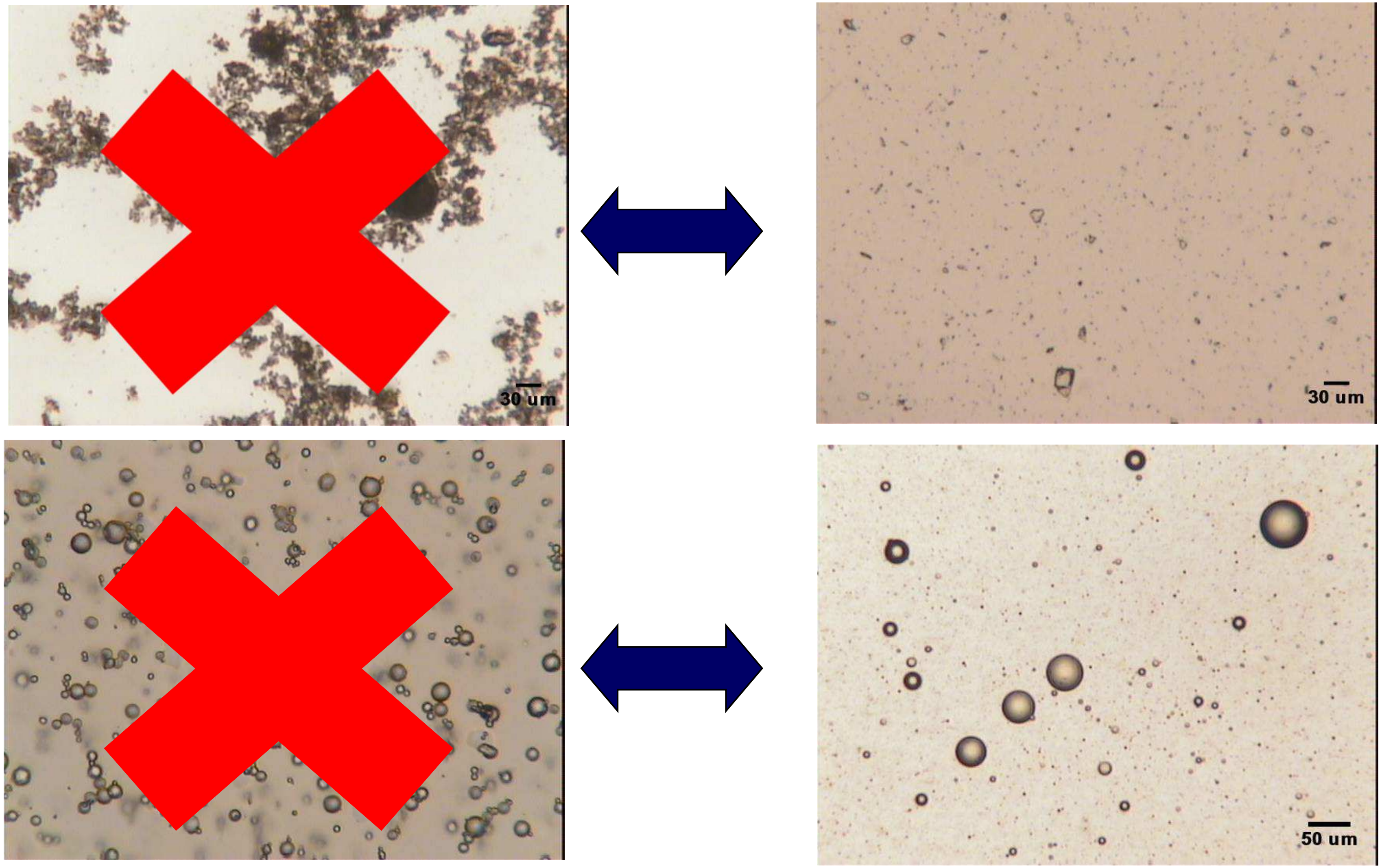
- Automatically locked into measurement position
- System recognises cell type
- Windows can be quickly removed without needing special tools
- Jacketed heating/cooling provided for rapid temperature stabilisation

Mastersizer3000

Wet Dispersion unit

Malvern Panalytical DKSH

Sample Dispersion unit : suitable dispersed form



Mastersizer3000
Wet Dispersion unit

Sample Quantity Decreases

HYDRO **EV**
600 – 1000ml

HYDRO **LV**
600ml

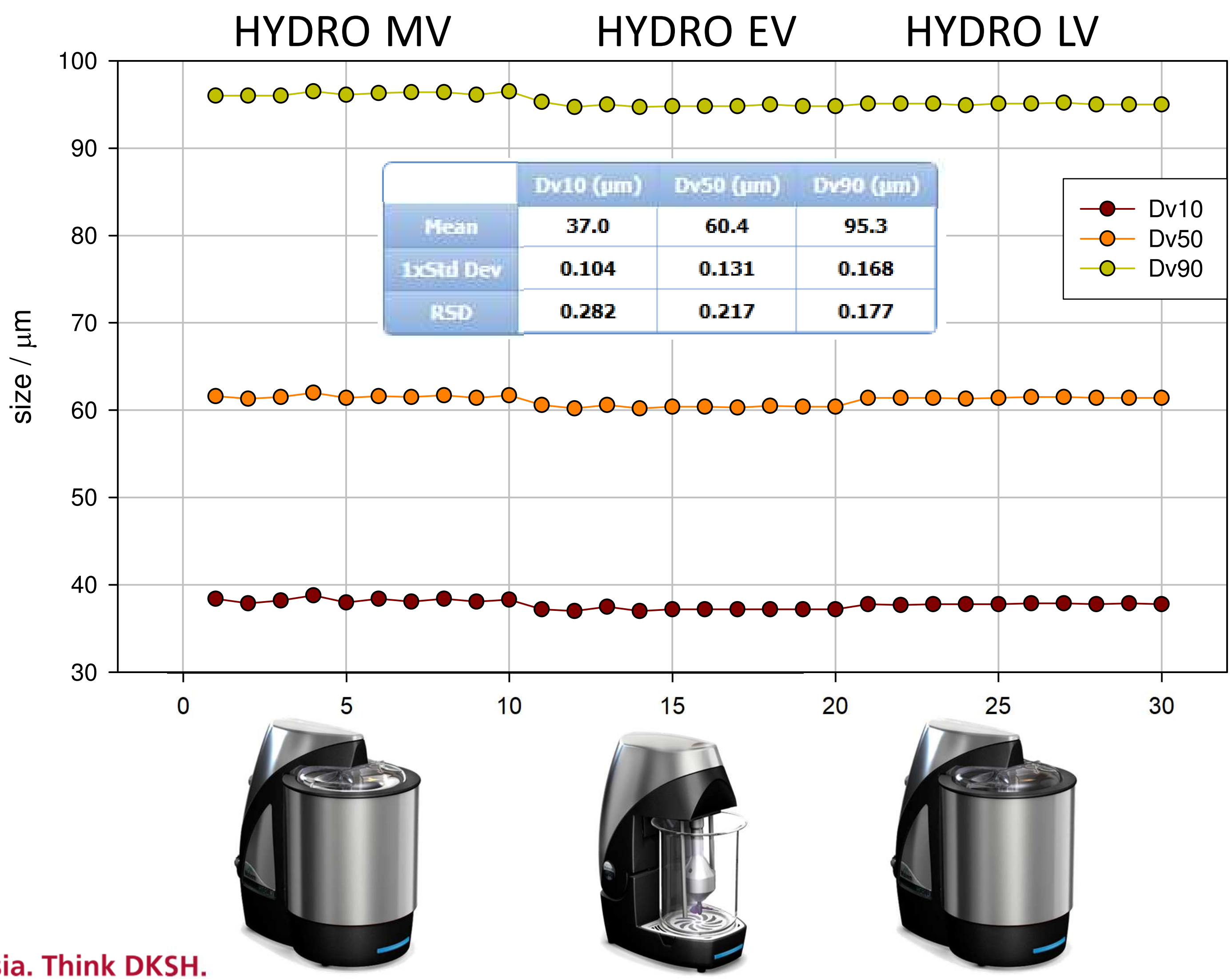
HYDRO **MV**
120ml

HYDRO **SM**
120ml

HYDRO **SV**
5.6-7ml

Mastersizer3000

Hydro unit reproducibility



	Dv10 (µm)	Dv50 (µm)	Dv90 (µm)
Mean	37.0	60.4	95.3
1xStd Dev	0.104	0.131	0.168
RSD	0.282	0.217	0.177

Malvern Panalytical DKSH

Very small volume liquid dispersion measurements are also possible using the Hydro SV

- 6-7ml volume
- 0.01 – 200 µm range
- Removable measurement cell
  - **Provides ease of cleaning**
  - **Wash station provided as standard**
- Uses a magnetic stirrer bar
  - **Manual or SOP speed control**
  - **Local speed reporting on cell**
- Pipette port provided for sample addition
- High chemical compatibility

Hydro SV demo video

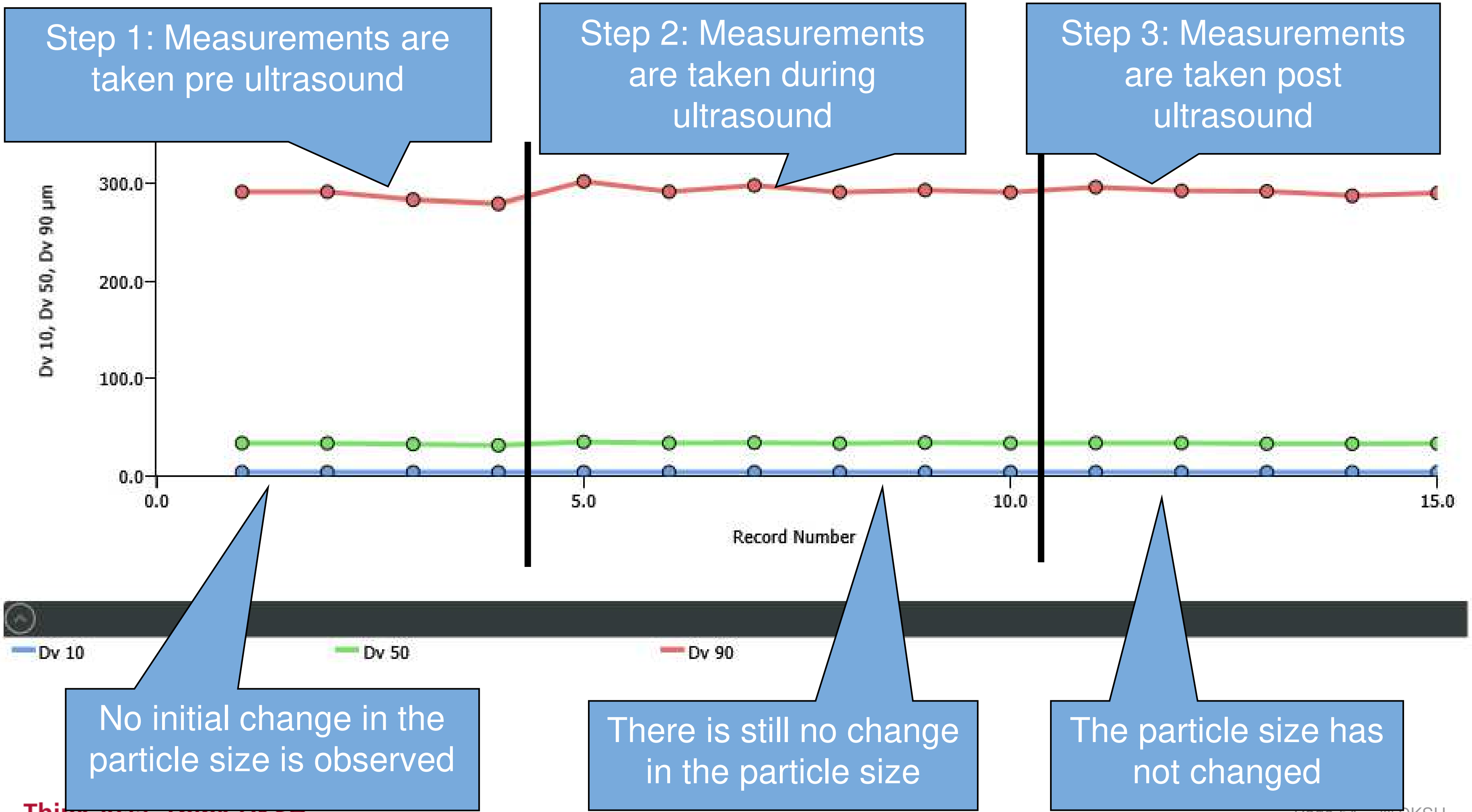
Good wet dispersion

- Wet dispersion allows the user to maximise measurement stability by **speed stirrer**, **changing dispersants, ultrasonication** and **surfactants/additives**.

- Start by selecting what you feel will be the most appropriate dispersant **and performing a set of repeat measurements**

- **For example** before, during and after ultrasonication.

Good wet dispersion - Trend graph Ultrasound



Good wet dispersion - Records view

Look at the records view and ensure that obscuration is stable and not increasing or decreasing with time

Record View

*Measurement file 5

Drag column header here to group by that column

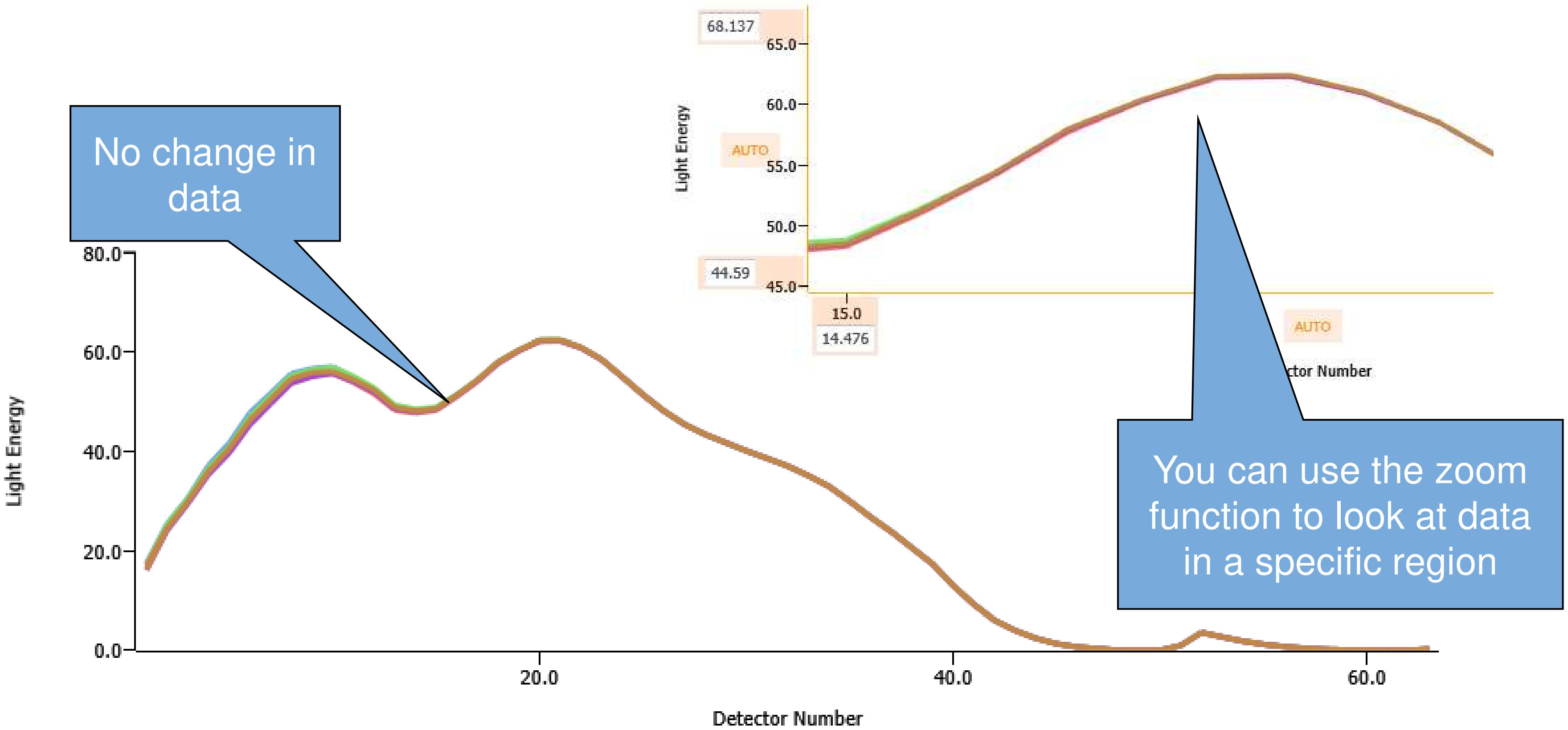
Record Number	Sample Name	Laser Obscuration(%)	Dv 10(µm)
1	sample 2	10.42	3.62
2	sample 2	10.42	3.61
3	sample 2	10.43	3.58
4	sample 2	10.41	
5	sample 2	10.45	3.68
6	sample 2 ultrasound	10.44	3.63
7	sample 2 ultrasound	10.44	3.64
8	sample 2 ultrasound	10.43	3.60
9	sample 2 ultrasound	10.44	3.65
10	sample 2 ultrasound	10.44	3.62
11	sample 2 after ultrasound	10.45	3.63
12	sample 2 after ultrasound	10.45	3.62
13	sample 2 after ultrasound	10.45	3.60
14	sample 2 after ultrasound	10.45	3.59
15	sample 2 after ultrasound	10.45	3.61

No changing obscuration

Note: you can also plot obscuration on the trend graph

Good wet dispersion - Data overlay

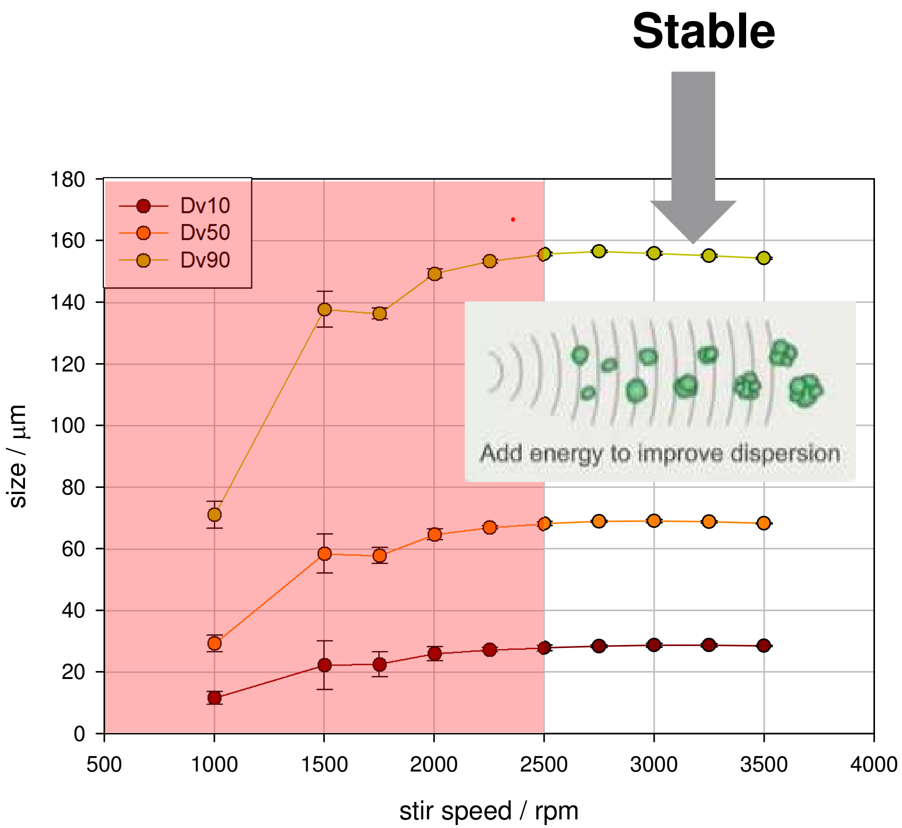
Overlay the data plots to verify that the data is stable and that poor optical property choices are not masking subtle changes.

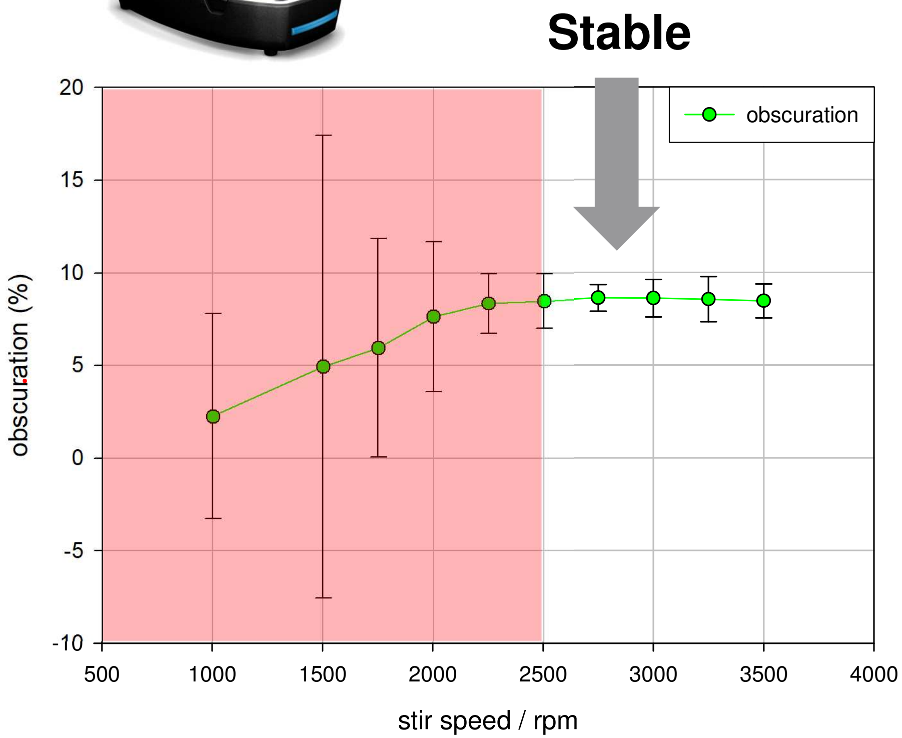


Hydro EV: sampling performance

Copper powder - density 8.92 g/cm^3

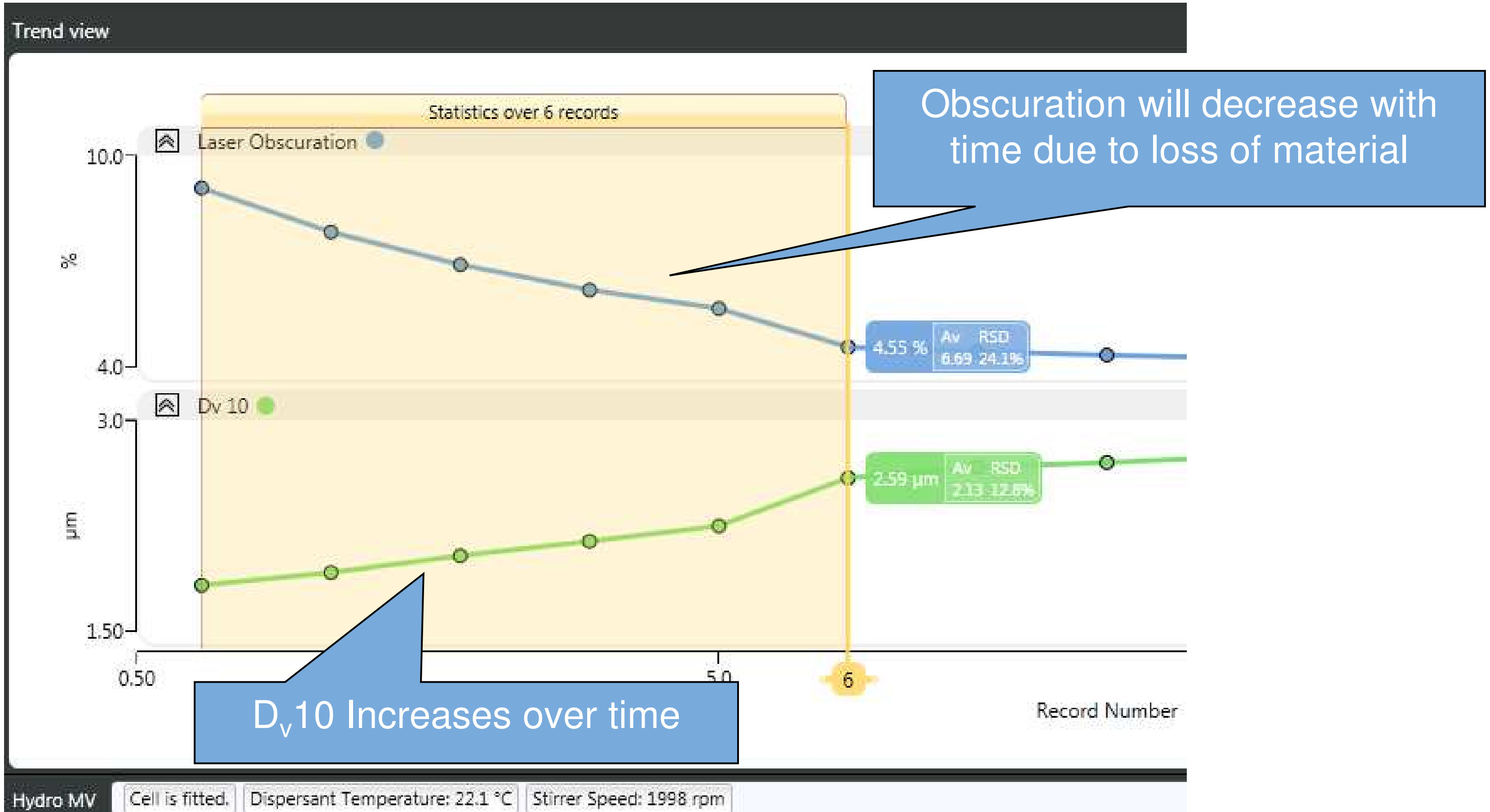
Stir speed titration – stable >2500 rpm



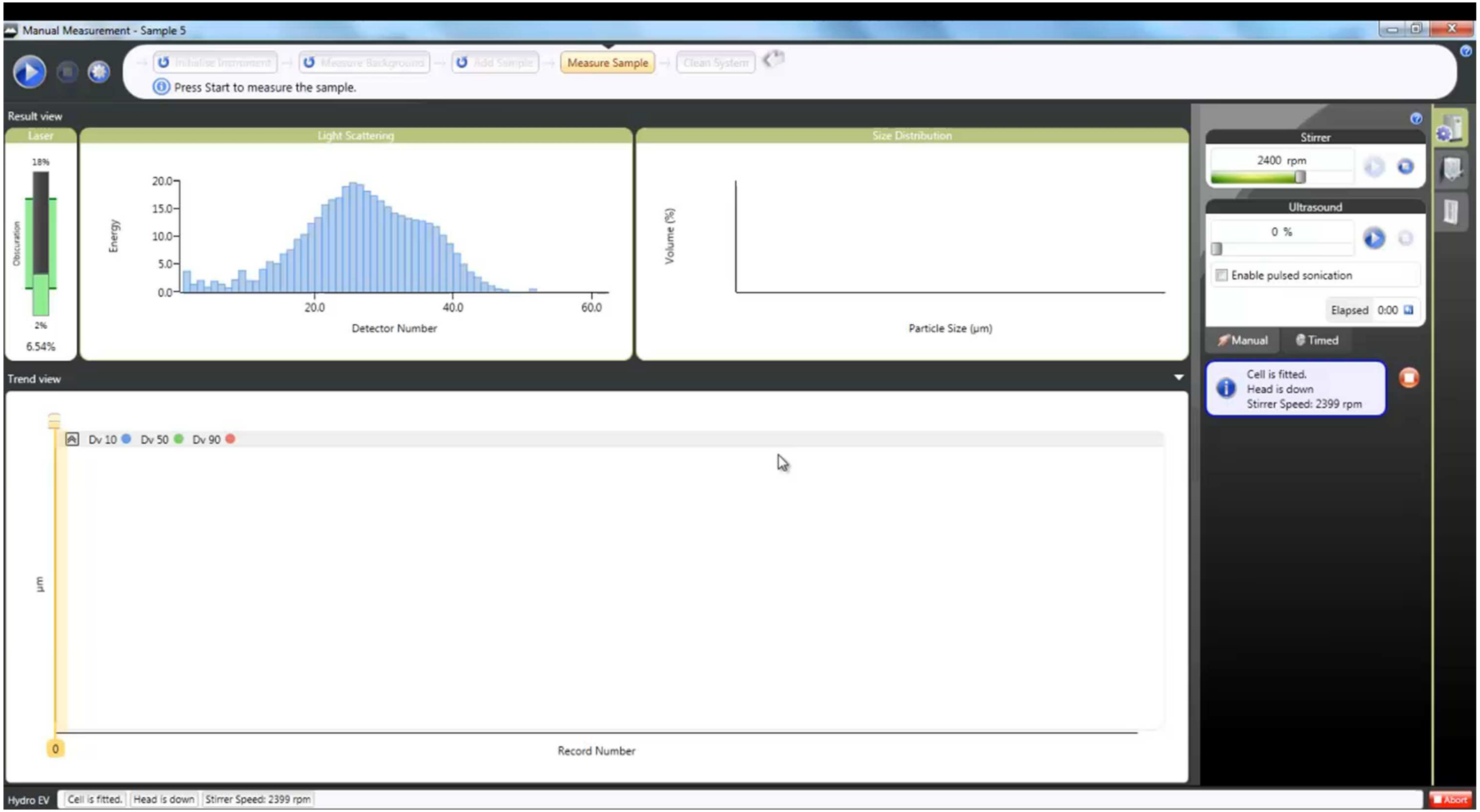


Dissolution – Using trend graphs

The Trend graph shows size increasing with time and obscuration decreasing – we are losing small particles



Using trend display to detect particle dissolution



Dissolution – Inspecting the records view

The Records view helps you to see what is happening

Record View

indigestion liquid

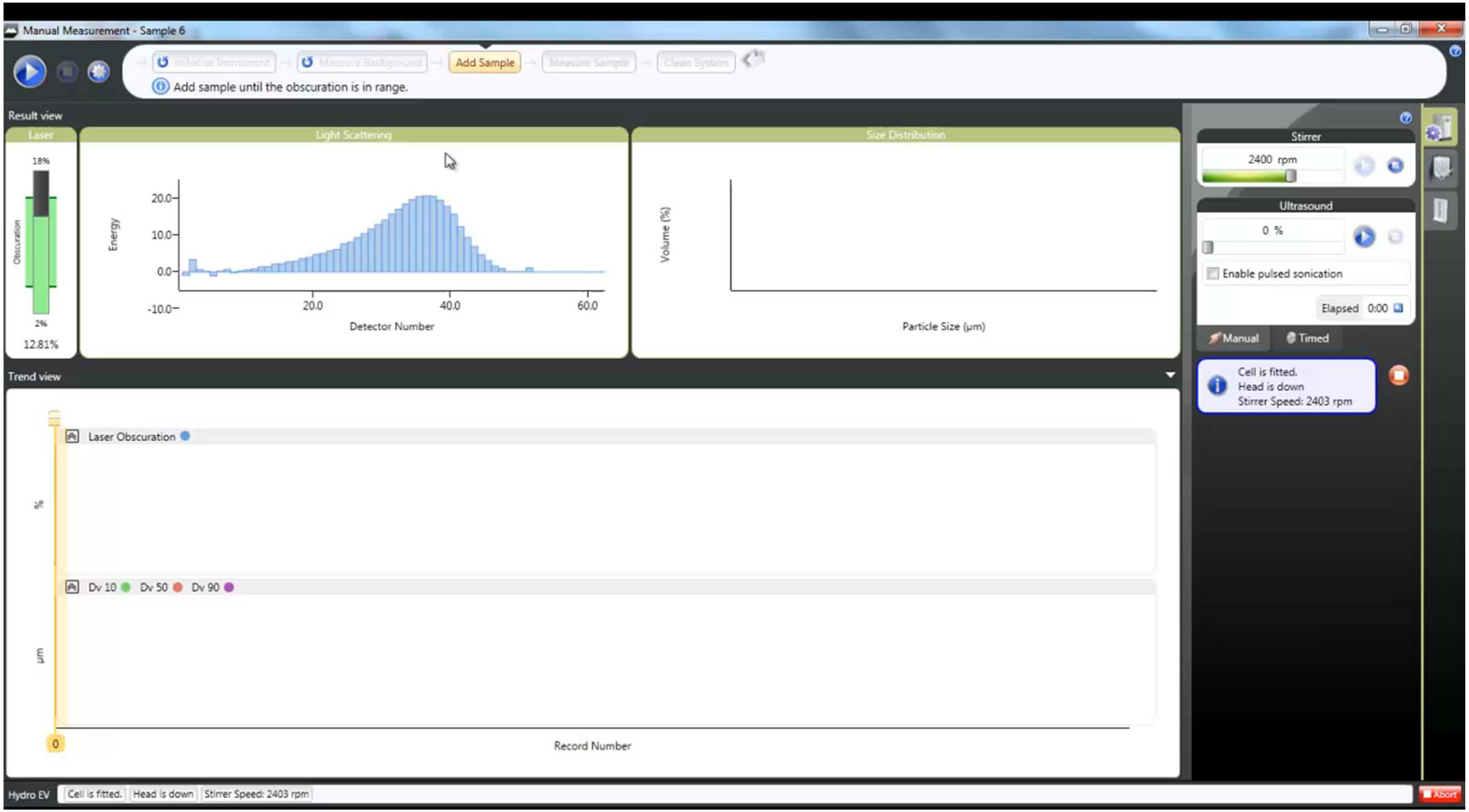
Drag column header here to group by that column

Record Number	Sample Name	Laser Obscuration(%)	Dv 10(µm)
1	sample 5	9.07	1.83
2	sample 5	7.83	1.92
3	sample 5	6.89	2.04
4	sample 5	6.17	2.14
5	sample 5	5.64	2.25
6	sample 5	4.55	2.59
7	sample 5	4.42	2.67
8	sample 5	4.32	2.70

Obscuration is dropping quickly with time.

D(0.1) is increasing. Fines disappear first.

Agglomeration – Size increases



Agglomeration – Inspecting the records view

Looking at the record view indicates that the obscuration has dropped slightly

Record View

*Measurement file 5

Drag column header here to group by that column

Record Number	Sample Name	Laser Obscuration(%)	Dv 90(µm)
1	Sample 6	8.02	10.5
2	Sample 6	7.84	10.5
3	Sample 6	7.70	10.7
4	Sample 6	7.58	11.1
5	Sample 6	7.49	11.0

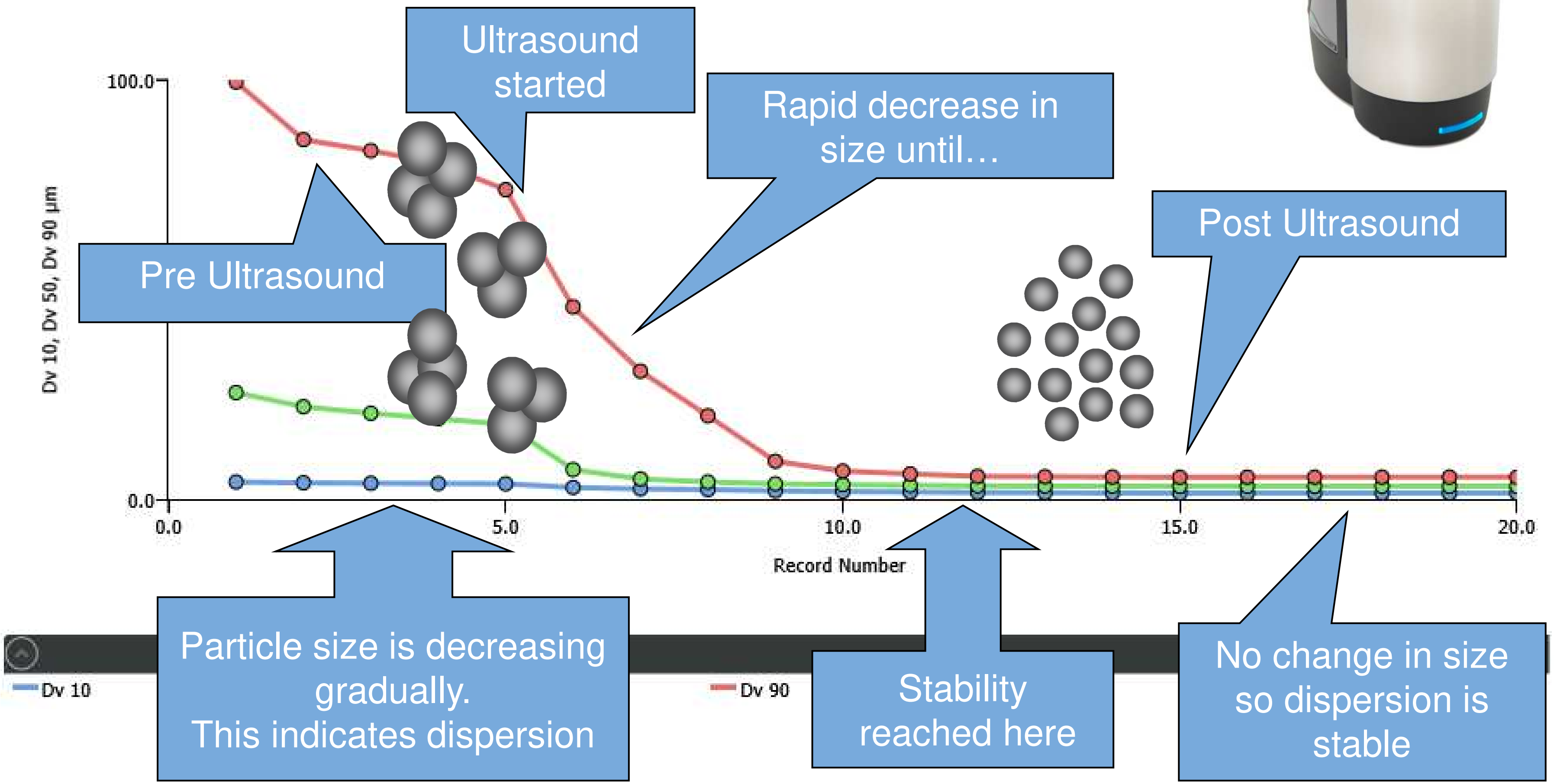
Slight decrease in obscuration

Increase in Dv90

Wet dispersion with ultrasound

Evaluating a dispersion process

What happens when the sample requires ultrasonic dispersion?



Evaluating a dispersion process– inspecting the obscuration

The Records view shows that the obscuration increases when ultrasonication is introduced

Record Number	Sample Name	Laser Obscuration(%)	Dv 10(µm)
1	sample 3	5.70	0.833
2	sample 3	5.81	0.815
3	sample 3	5.88	0.809
4	sample 3	6.03	0.830
5	sample 3	6.14	0.772
6	sample 3	6.26	[illegible]
7	sample 3	6.41	0.838
8	sample 3	6.44	0.711
9	sample 3 US	12.51	0.403
10	sample 3 US	12.57	0.397
11	sample 3 US	12.58	0.360
12	sample 3 US	12.86	0.374
13	sample 3 US	13.03	0.374

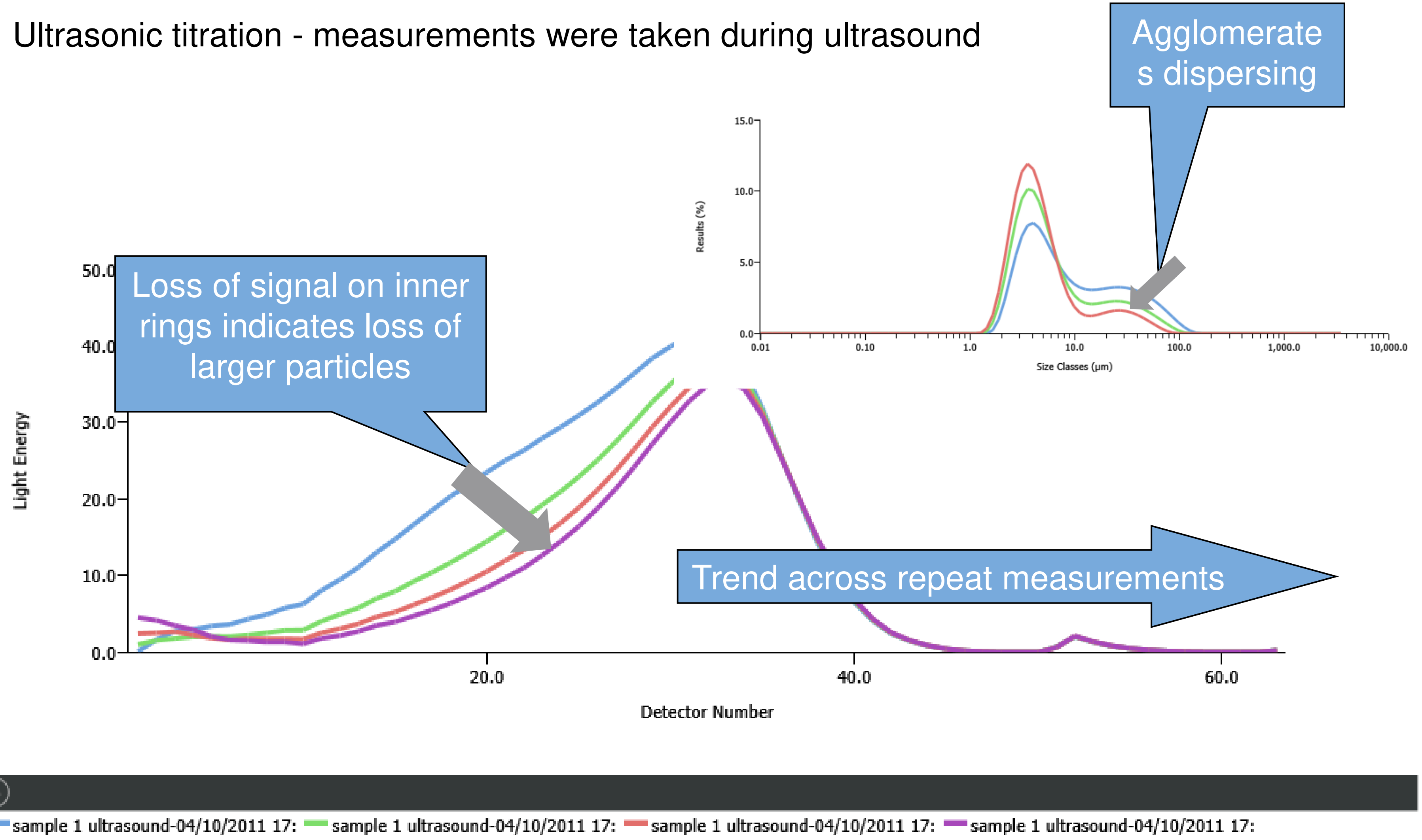
Ultrasound started. Note the increase in obscuration as more and smaller particles become present due to the dispersion of agglomerates

Dispersion starts with the break-up of agglomerates by:
- Wetting the sample by adding surfactant
- Stirrer action
- Ultrasonication

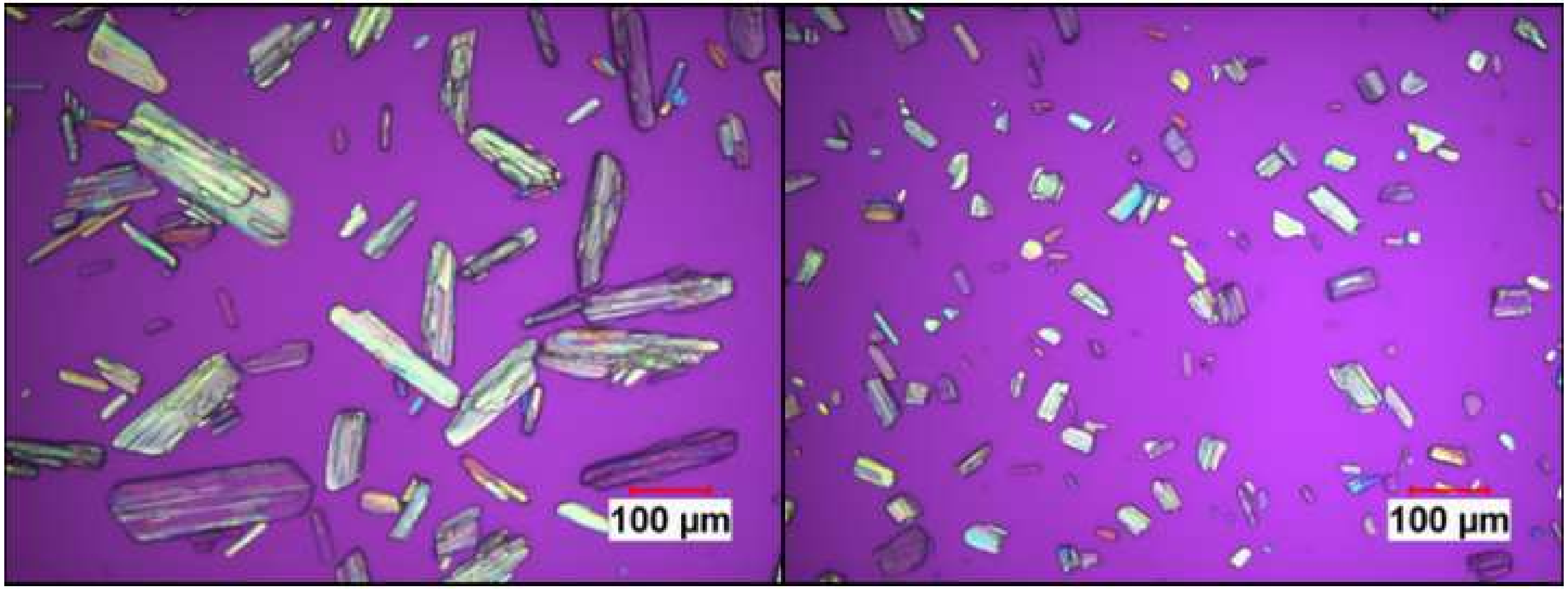
What are the signs of dispersion?
- **Particle size** reduction
- An increase in **obscuration**
- Increased **light scatter on the higher detectors** on the data plot
- **Coarse fractions** in the distribution reduce and may disappear

Evaluating a dispersion process– Inspecting light scattering data

Ultrasonic titration - measurements were taken during ultrasound



Beware: Too much Ultrasound



Dry dispersion

Mastersizer3000

Dry Dispersion unit

❖ **Rapid measurement**

Very simple, rapid workflow

❖ **Precise dispersion control**

❖ **Modular disperser design**

Options for fragile, cohesive or abrasive samples

❖ **Modular powder handling capability**

Hopper unit for large sample quantities

Micro and Macro trays for smaller quantities

❖ **Fully automated sample feed and dispersion**

Mastersizer3000

Dry Dispersion unit : Window Cell (Dry)

- Automatically locked into the required measurement position
- Windows can be quickly removed for cleaning without needing any tools
- Vacuum connection is attached to the optical bench
- Sheath air flow is filtered to aid measurement robustness

Dry dispersion unit

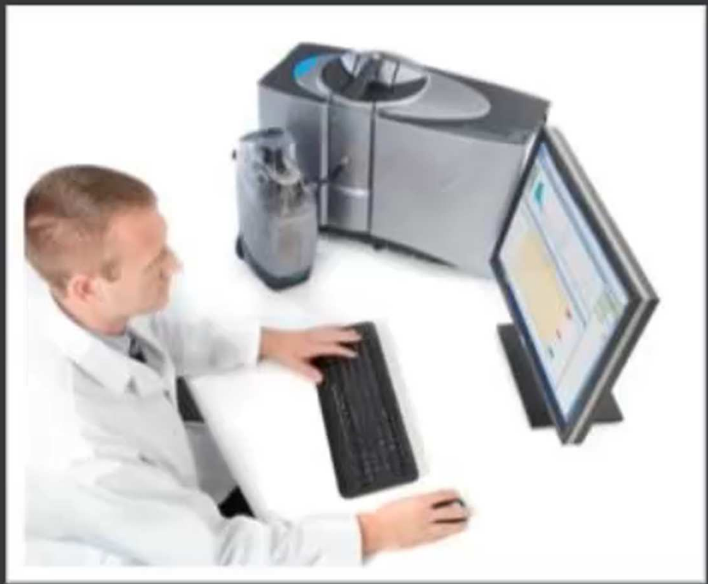
Aero S (For Mastersizer3000)

Aero M (For Mastersizer3000E)

Dry Dispersion unit

Malvern

Dry Measurement Demo
Aero S



Dry Dispersion unit : Sample tray

Micro tray

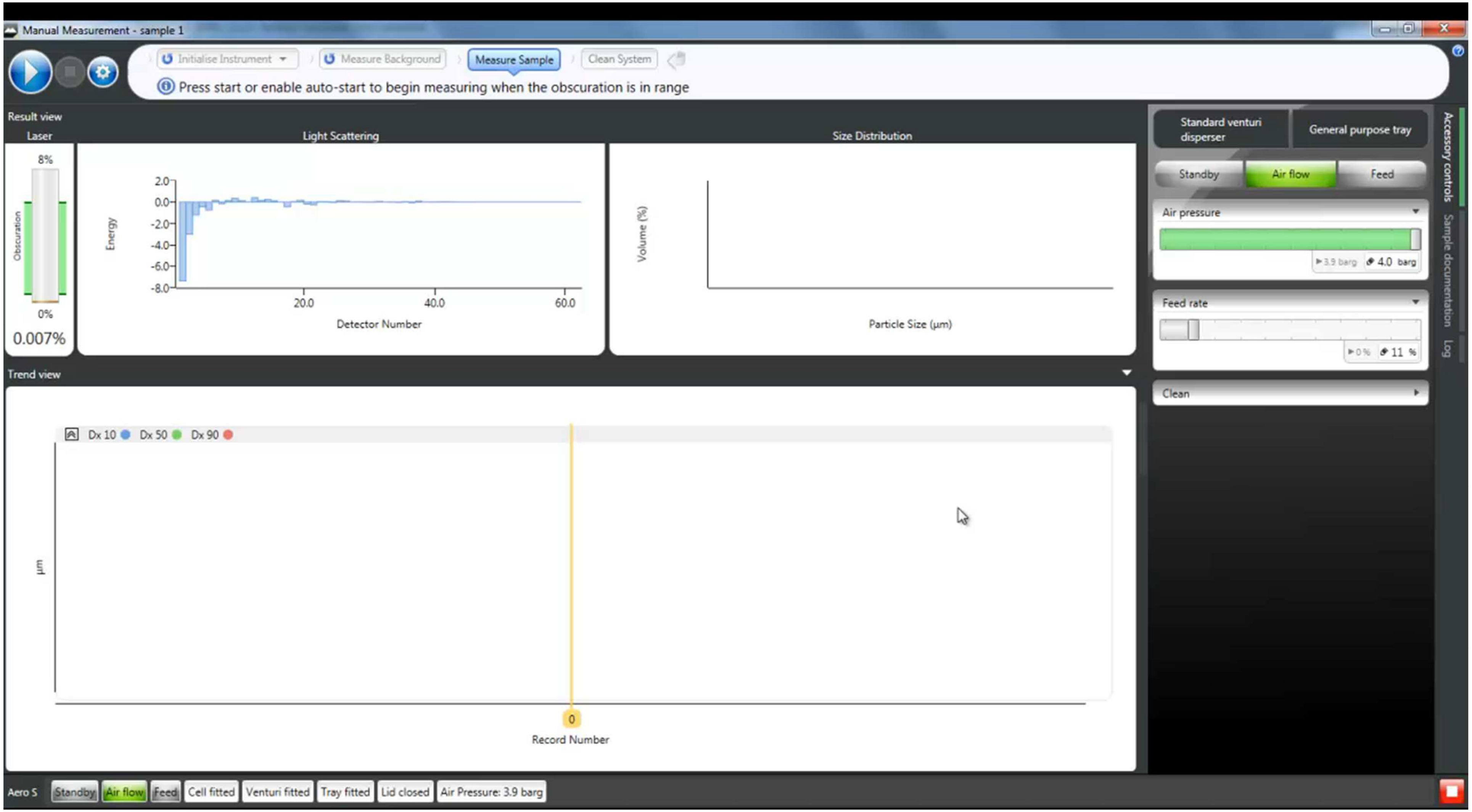
Macro tray

General purpose tray
with hopper

Large volume tray

Funnel sample feeder

Set up feed rate



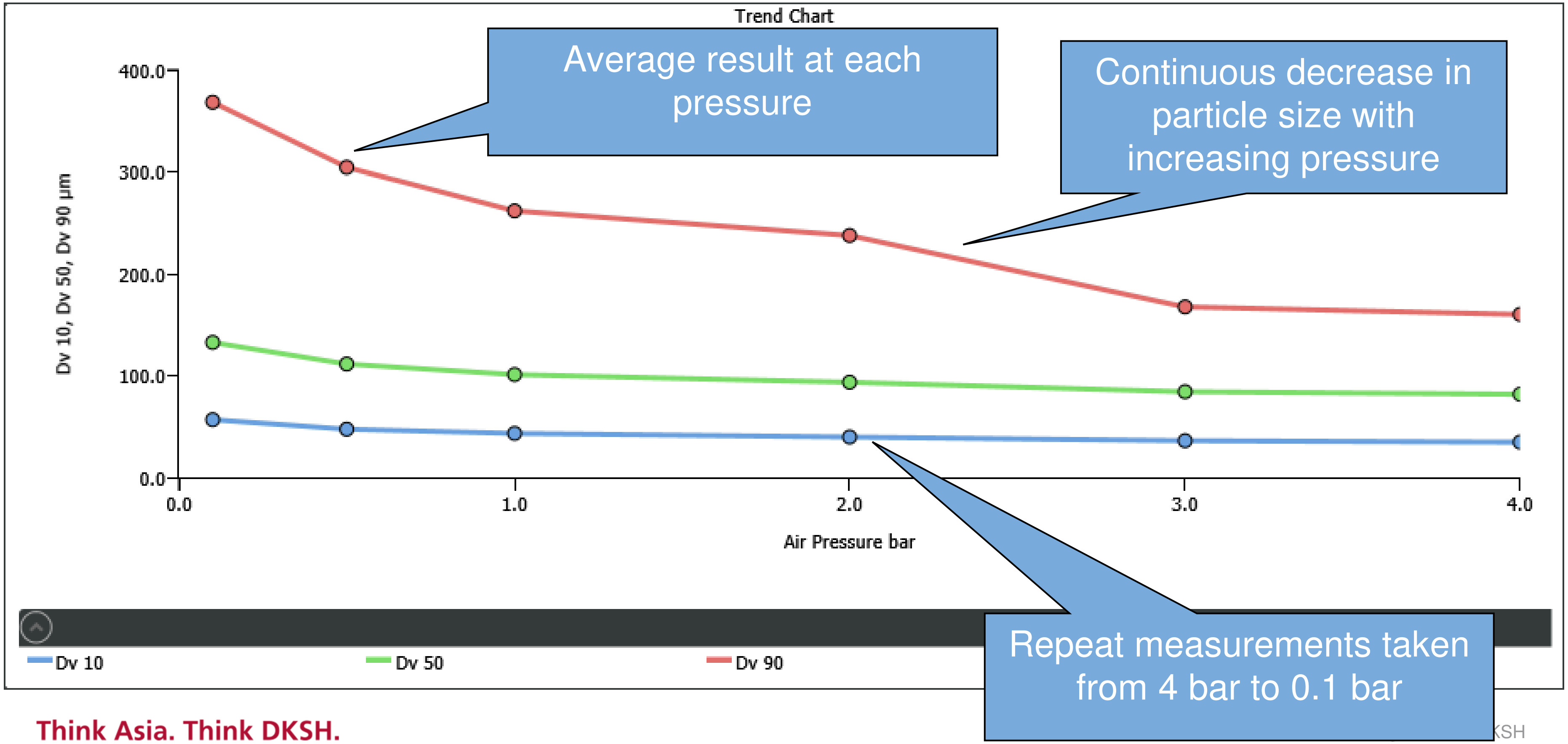
Dry dispersion - obscuration filtering

Obscuration filtering is used to ensure that noise (no particles present) and occasional spikes in obscuration are not recorded - good flow control is still **important**

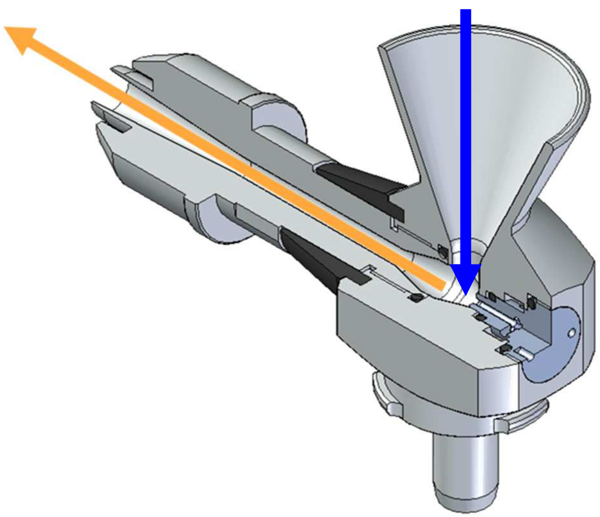
Dry dispersion – pressure titration

Pressure titration –identifies optimal conidiation

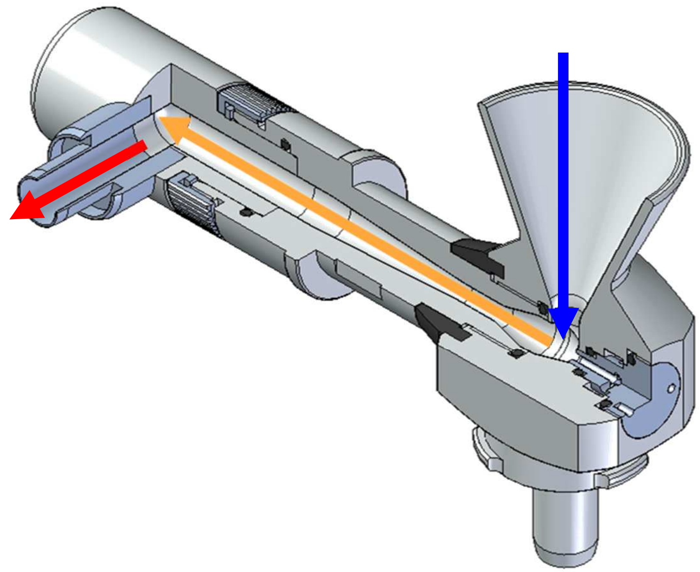
Generally particle size decreases with increasing air pressure, to a stable value where agglomerate are dispersed.



Dry Dispersion unit : Venturi



Standard Venturi

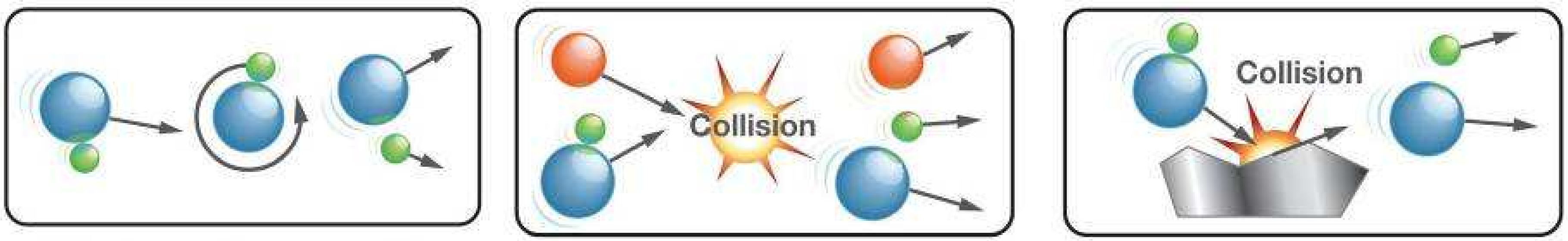


High-Energy Venturi

Dry Dispersion unit : Venturi

Performance : Versatile Dry Powder Dispersion

Dry powder dispersion mechanisms



Velocity gradients caused by shear stress

Particle-to-particle collisions

Particle-to-wall collisions

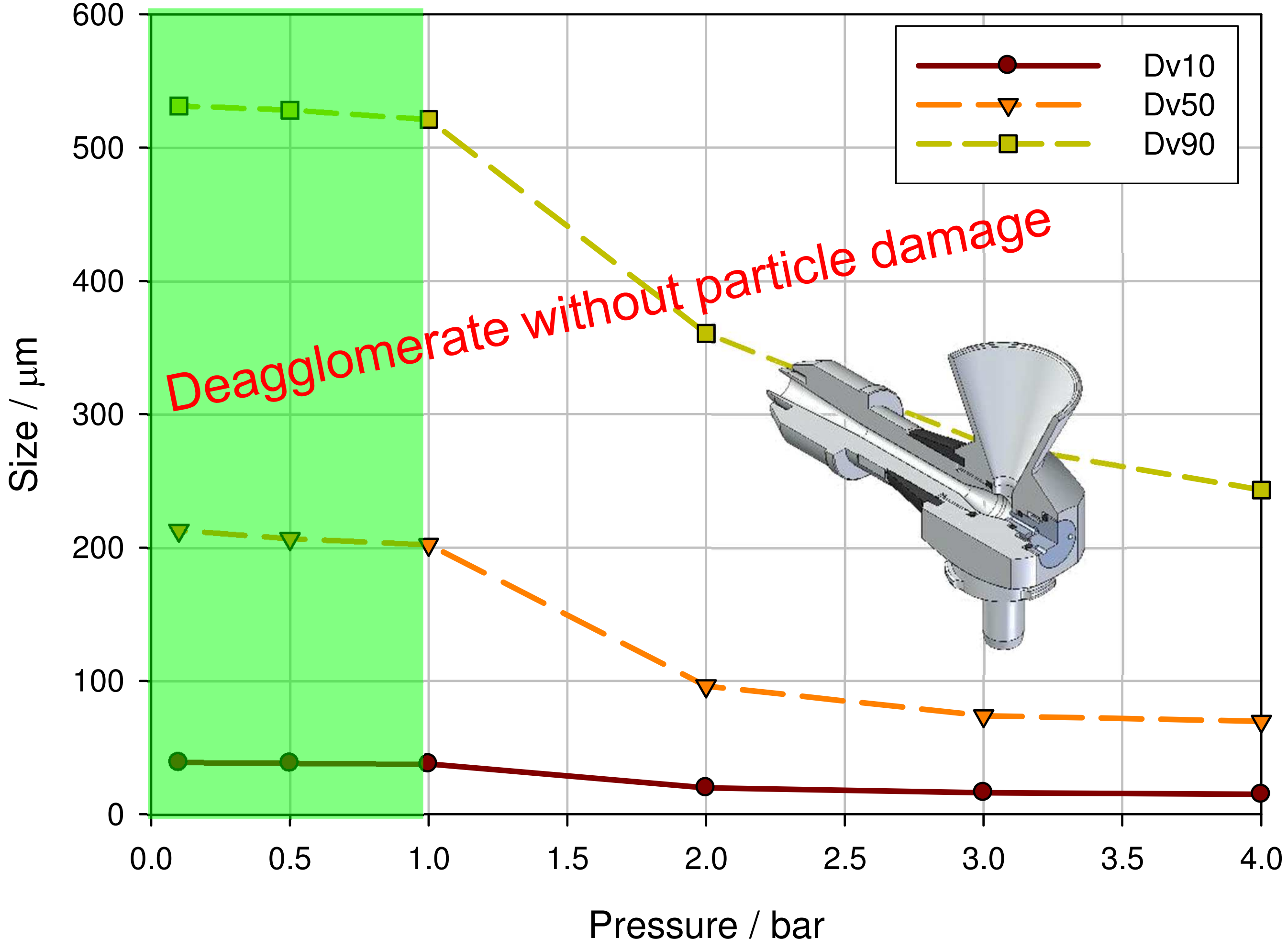
Energy/aggressive

Modular disperser design

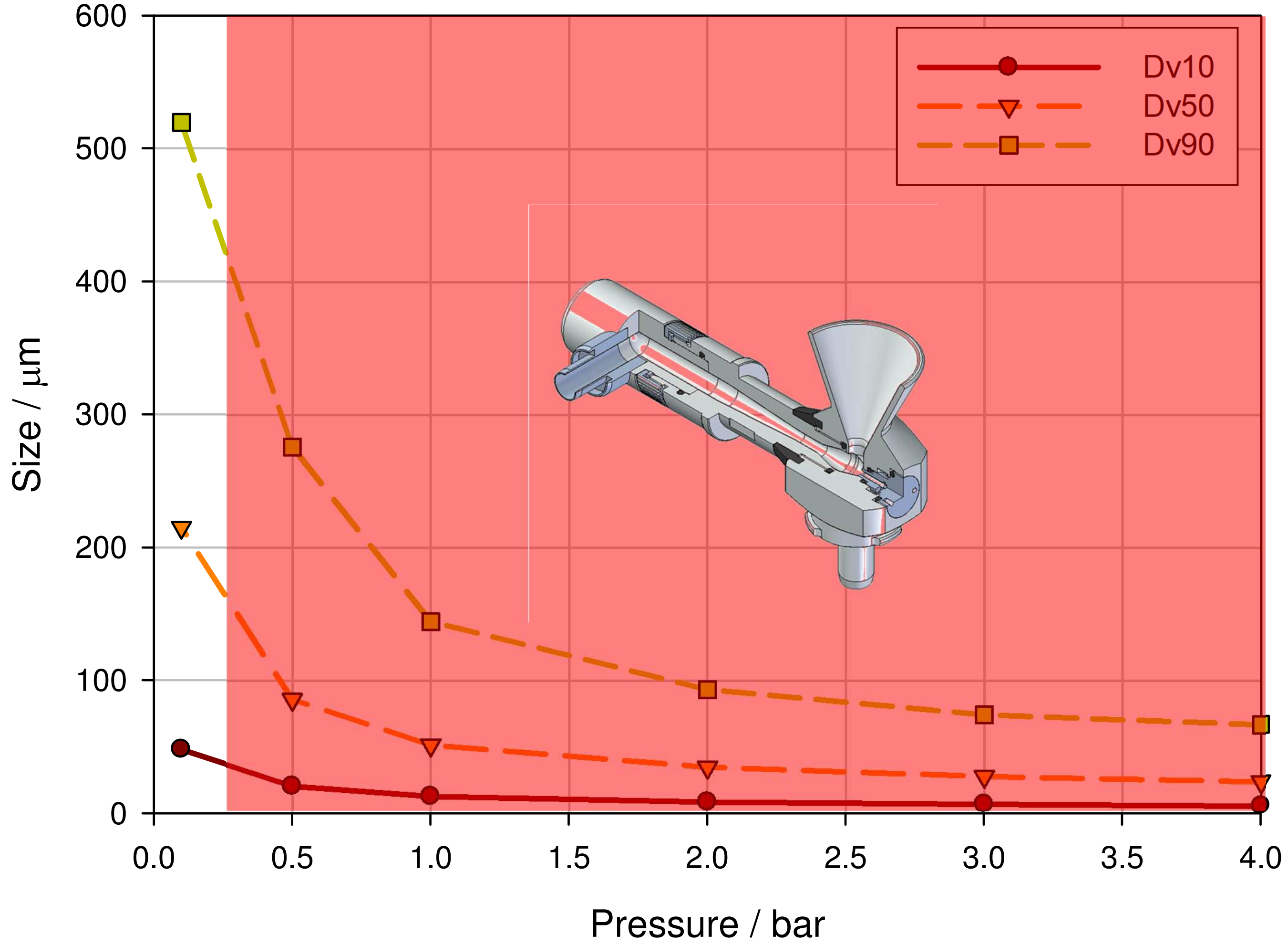
- Quick changeover for application versatility
- Match the dispersion mechanism to application

Pressure Titration in Dry dispersion unit

Standard disperser:Fragile crystalline powder



High energy disperser: fragile crystalline powder



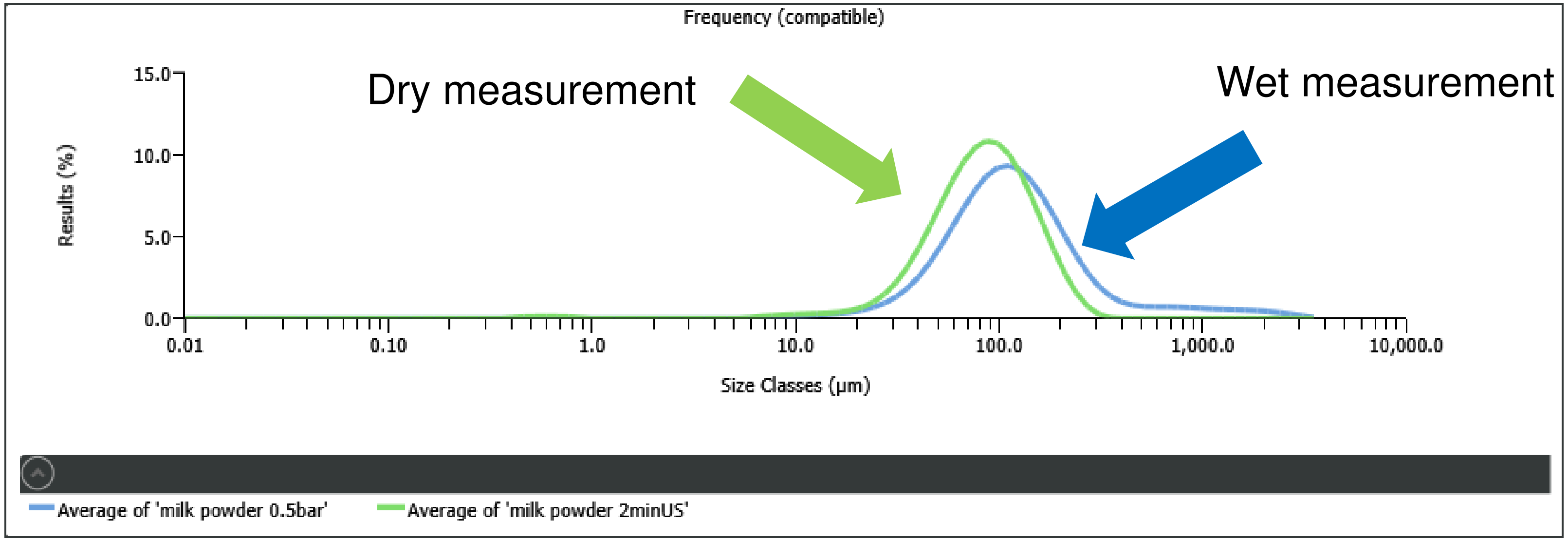
Dry dispersion – which pressure should we select?

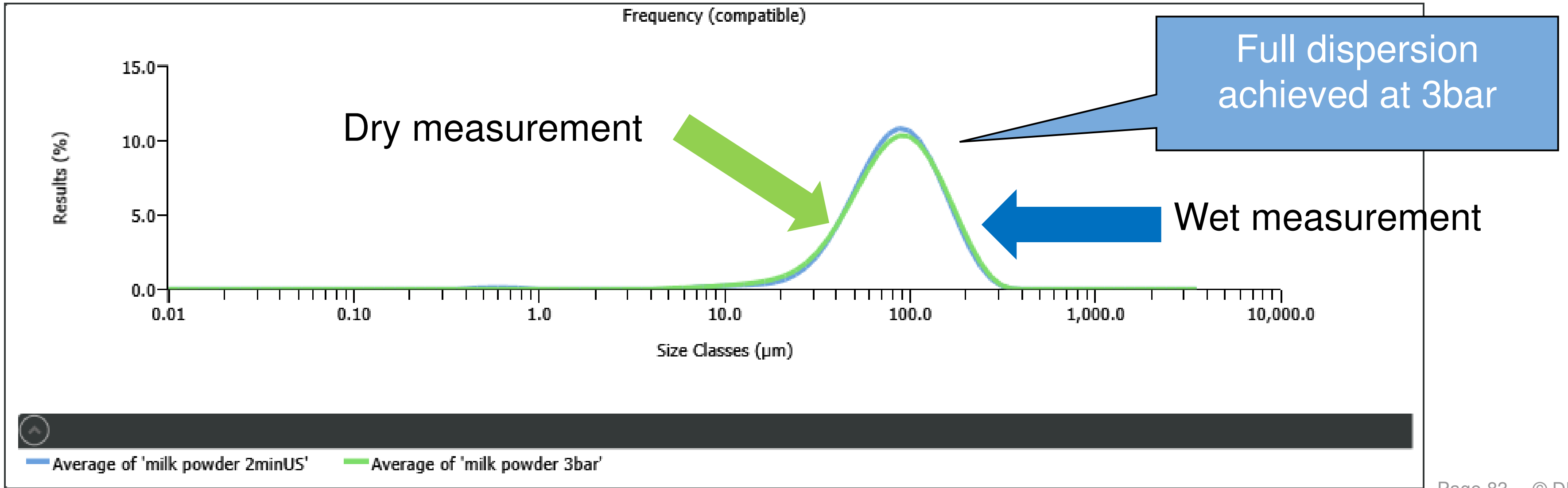
ISO13320-1, sec 6.2.3.2 states that…

It is necessary to check that comminution of the particles does not occur and conversely that a good dispersion has been achieved. This is usually done by direct comparison of dry dispersion with a liquid one: ideally, the results should be the same.

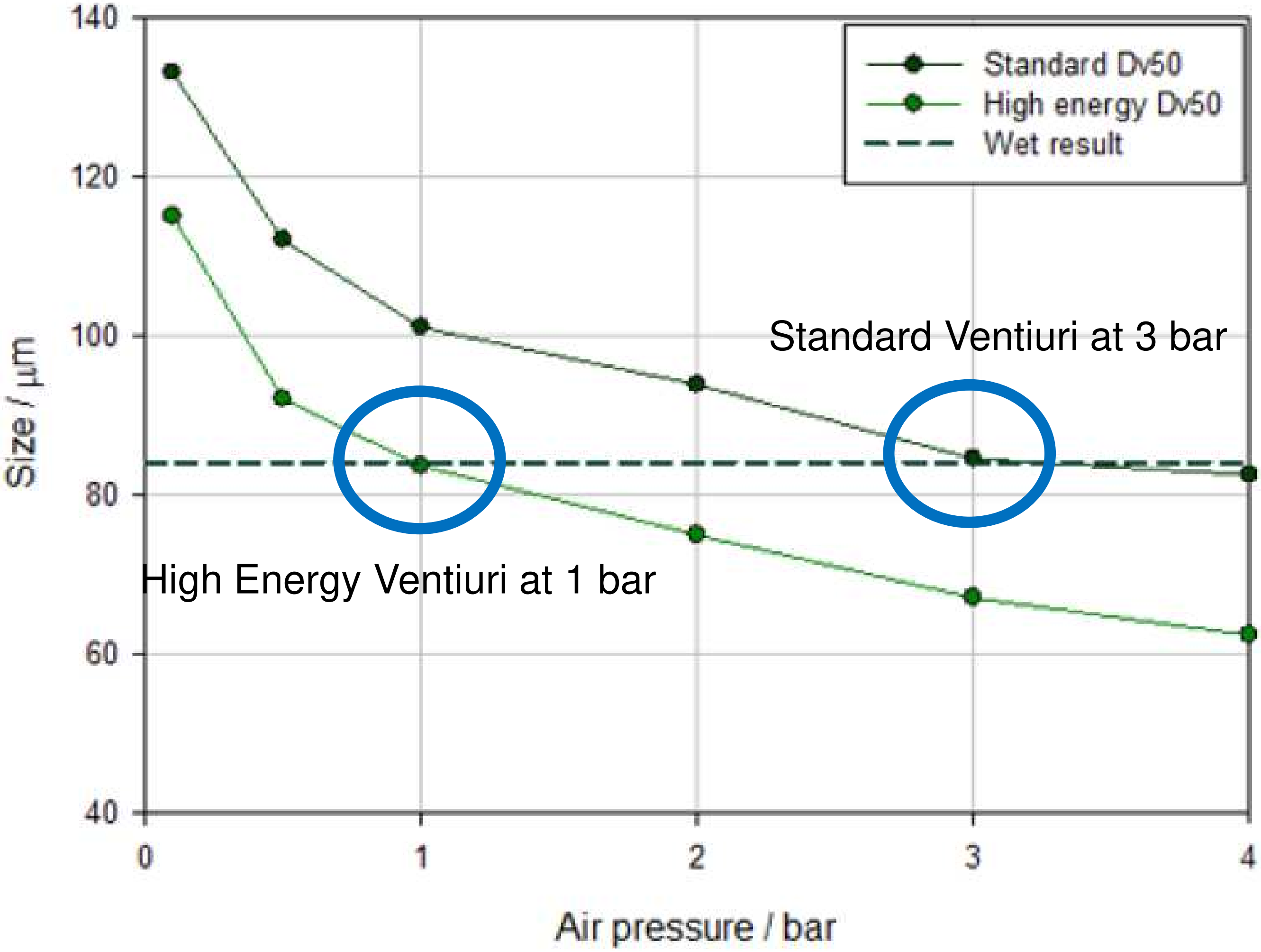
To determine which is the correct pressure, compare the dry analysis results to those obtained by an optimised wet dispersion if possible

Dry dispersion – which pressure should we select?





Dry dispersion – which pressure should we select?

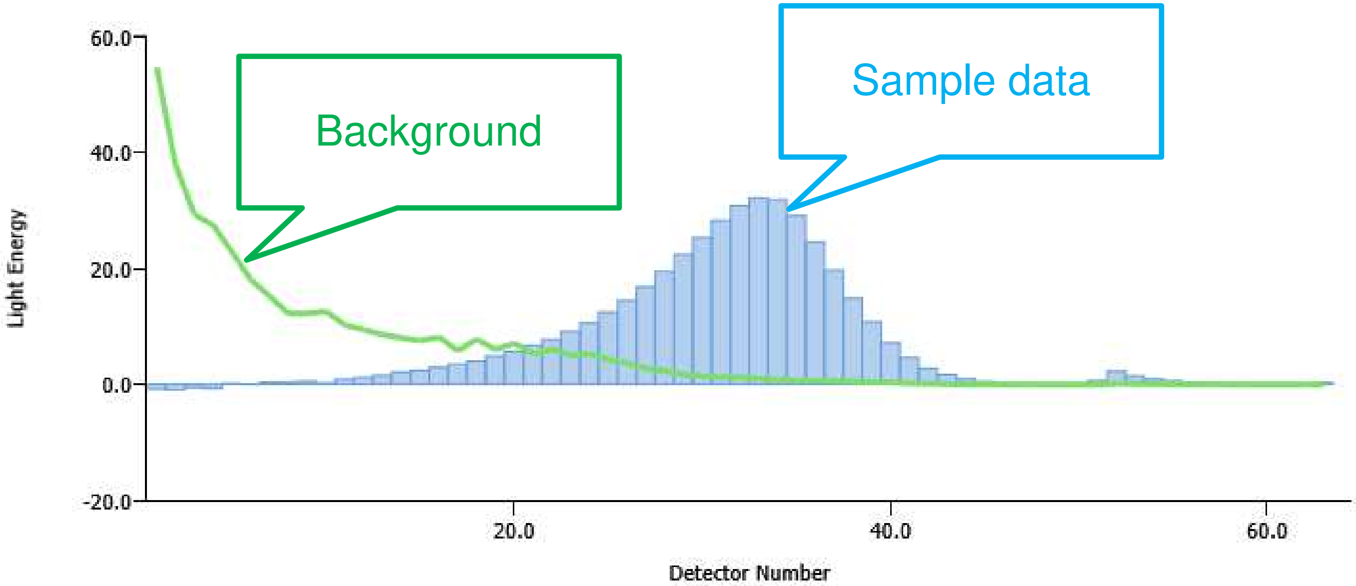


Data Quality

Way to get the best result.

Data quality - introduction

What do we mean by data?



- Data is the fundamental **light scattering** caused by your sample
  - It is not the particle size result
  - It is independent of the optical model.
- A stable result requires stable data

Data quality - introduction

What Constitutes Quality Data?

A good background measurement, taking into account

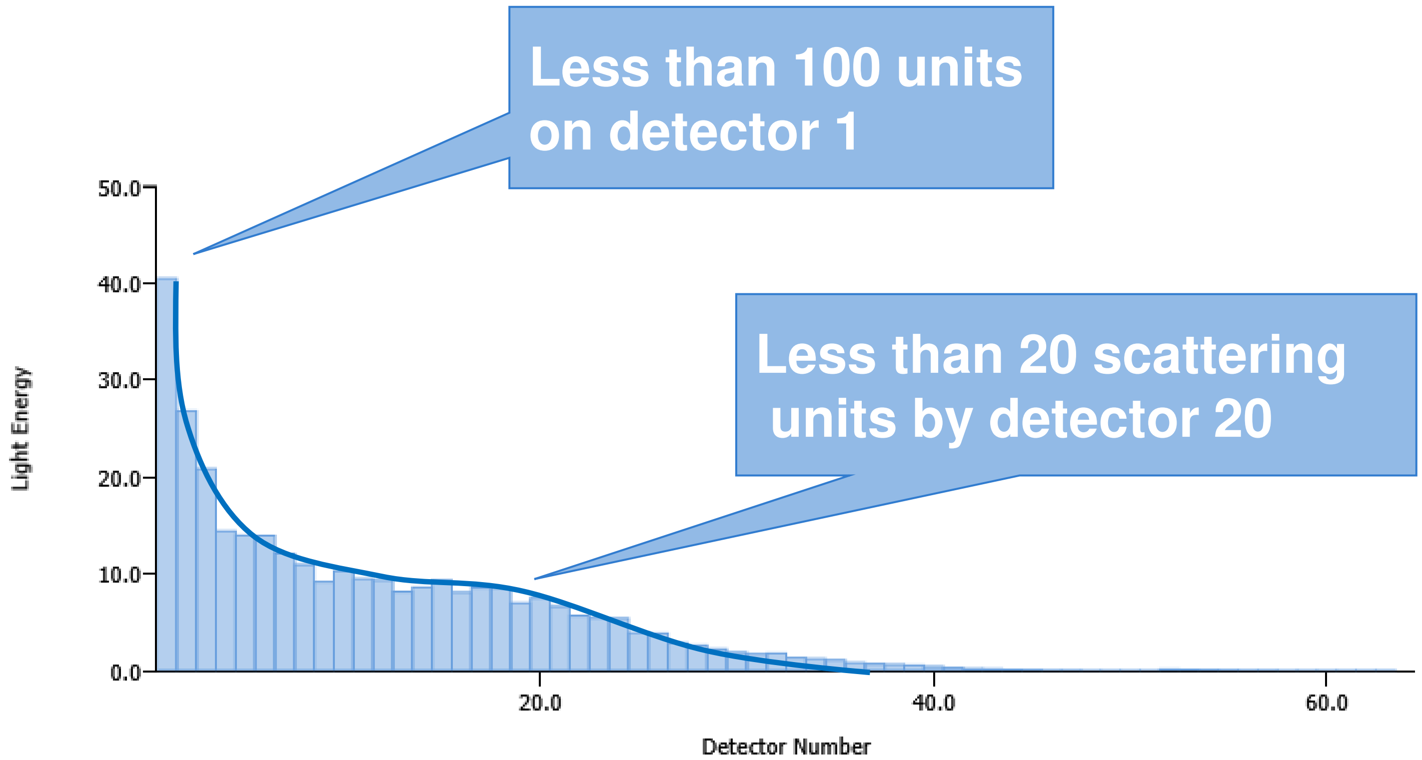
- cleanliness of cell windows and dispersant
- alignment of the system
- stability over time

Sample data

- gaining sufficient signal to noise
- avoiding negative data
- limiting the effect of multiple scattering
- avoiding beam steering

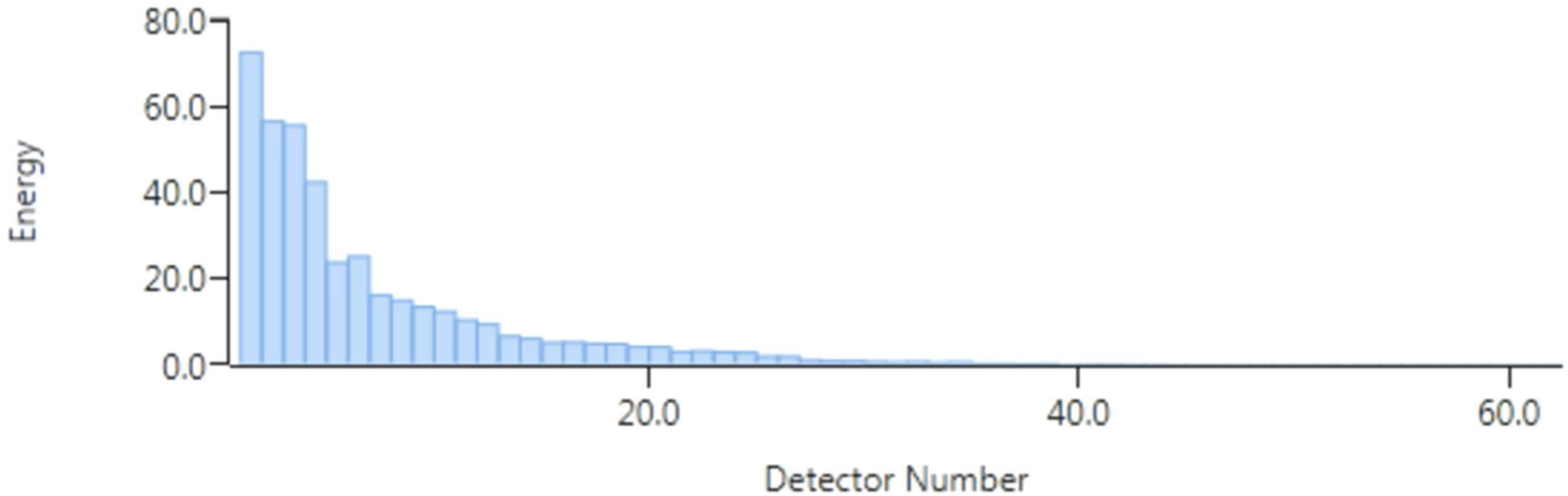
In general this applies to all laser diffraction instruments

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



A clean background – wet system

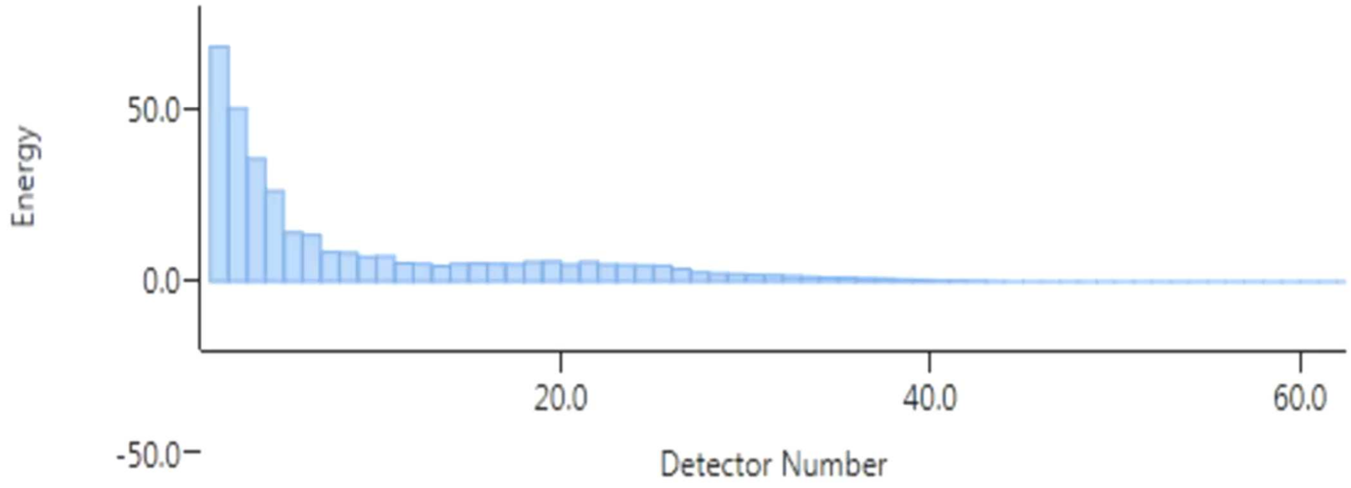
A good clean background on a wet system should look very similar to this...



- Less than 100 units on the 1st detector
- Limited fluctuations over time

A clean background - dry system

- The flow of air through a dry cell will cause a slightly higher degree of fluctuation over time than seen in a wet system
  - The same decrease in the signal is observed across the detector range.
  - **The same maximum acceptable value of 100 applies**



Data Quality

Poor background

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

Adding the right amount of sample

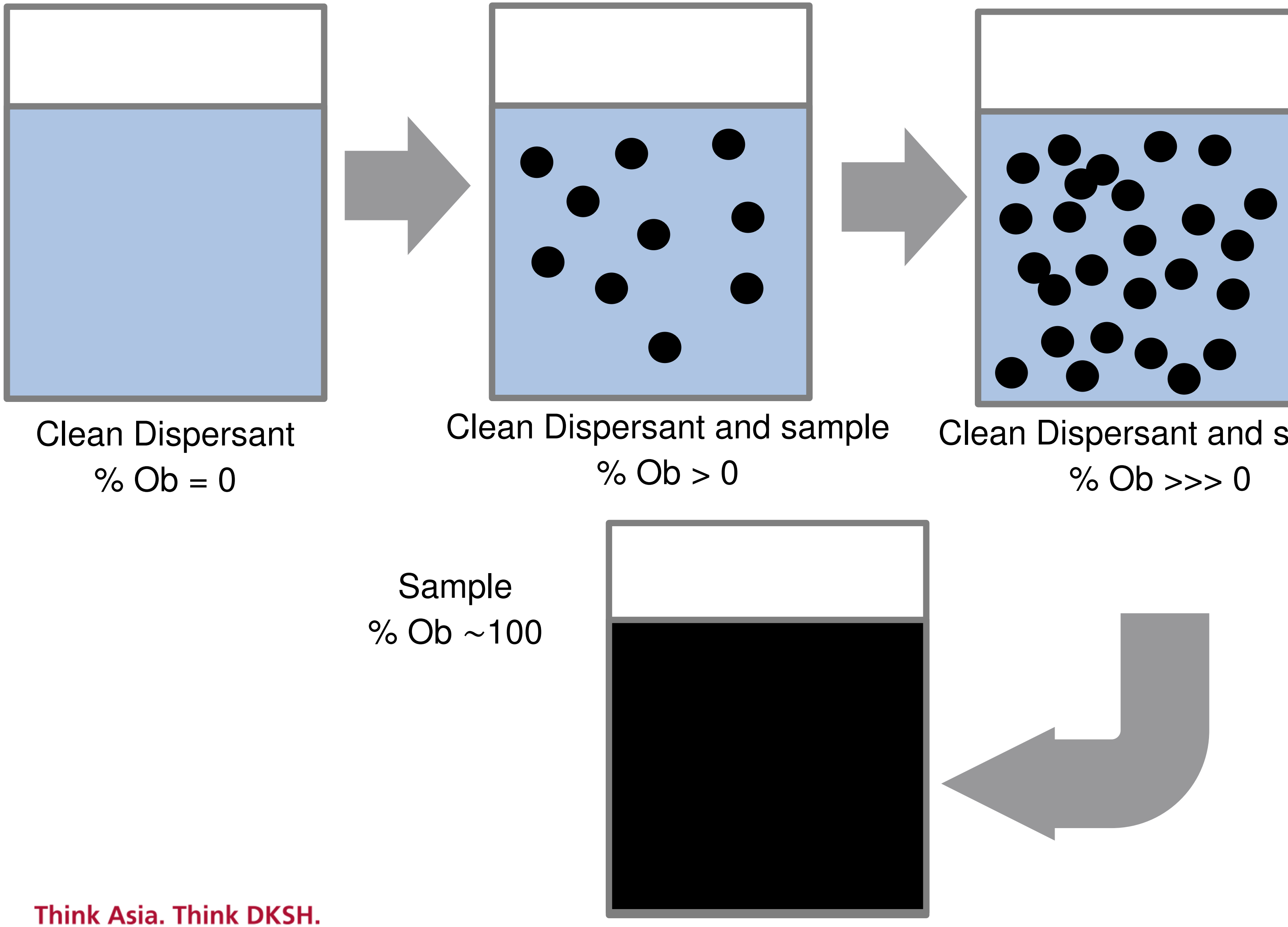
Terms used in this section:

- **Obscuration**: The amount of laser light blocked and /or scattered by the sample: a guide to the sample concentration

- **Signal-to-noise ratio**: The magnitude of the sample data in relation to the background data

- **Multiple scattering**: The phenomenon caused by light scattered by a particle being scattered by other particles when too much sample is added

Data Quality
Obscuration



Adding the right amount of sample

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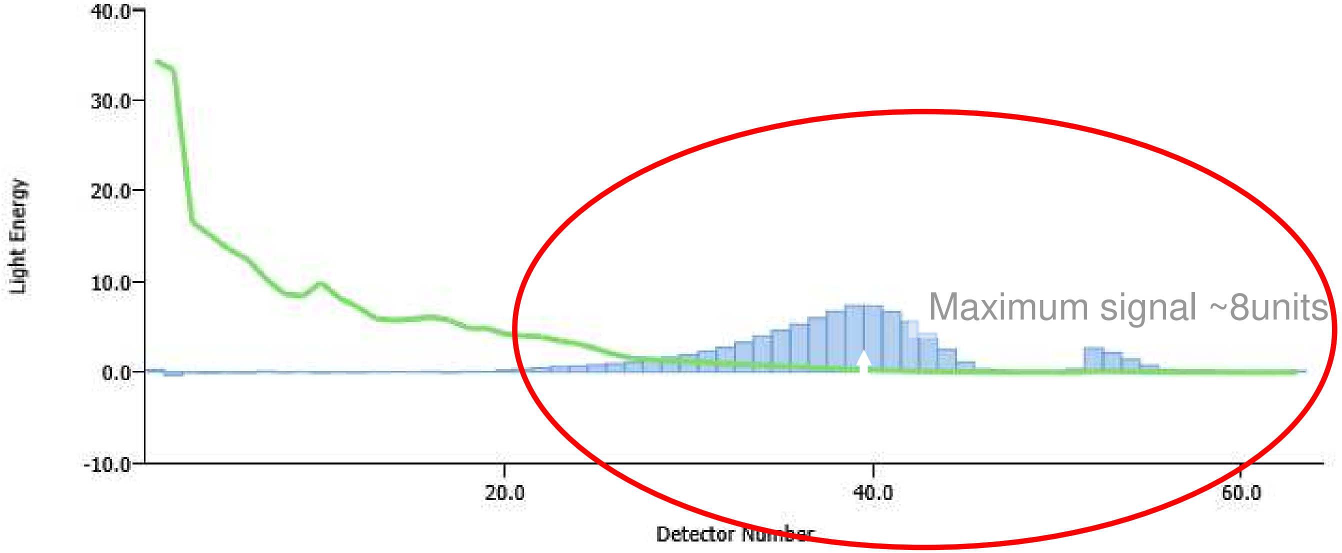
Data Quality
Signal to noise ratios - small particles

(1 micron Latex measured at **an obscuration of 5%**) in wet system



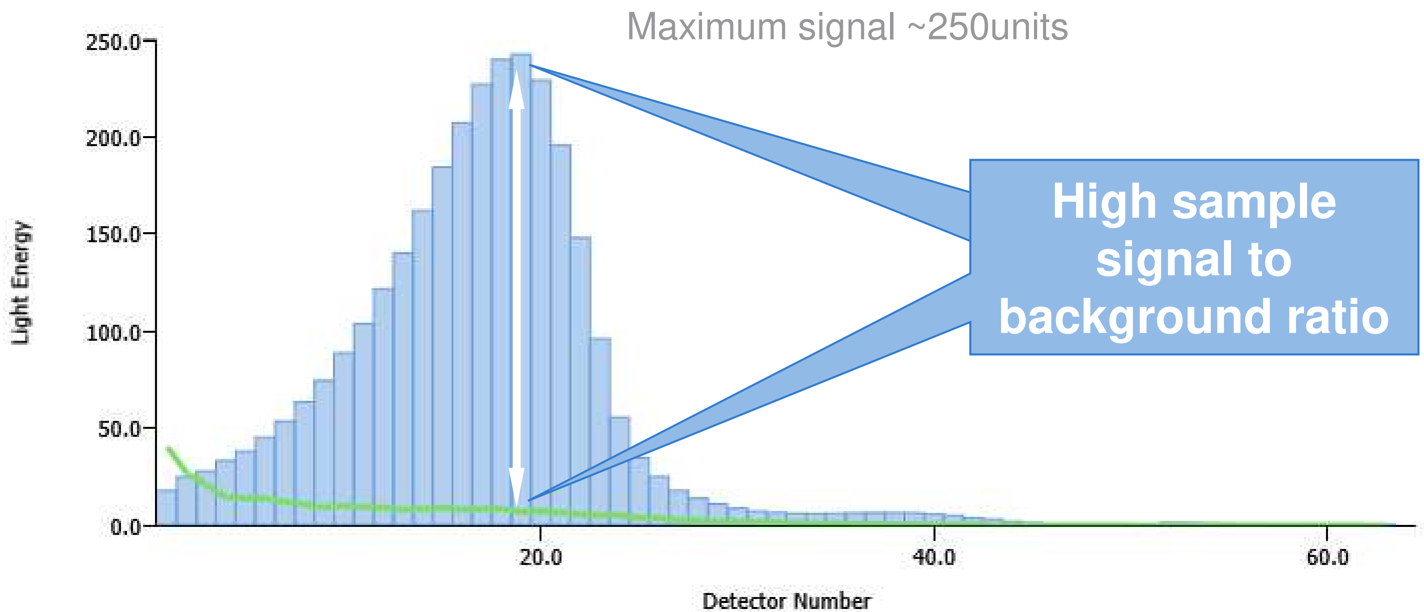
The signal to noise ratio is the amount of sample data relative to the background data.

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.

Signal to Noise ratios – large particles

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



Note that 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.

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)

Data Quality

Guide for adding the right amount of sample

THE RECOMMENDED OBSCURATION RANGES FOR WET ANALYSIS

Particle Size	Obscuration Range
0.01 – 1 µm	1% – 5%
1 – 100 µm	5% – 10%
100 – 3500 µm	10% – 20%
Poly-dispersed samples	Choose based on largest

HERE'S FOR DRY ANALYSIS:

Particle Size	Obscuration Range
Cohesive, fine particles	0.5% – 5%
Easily dispersed, coarser particles	1% – 8%

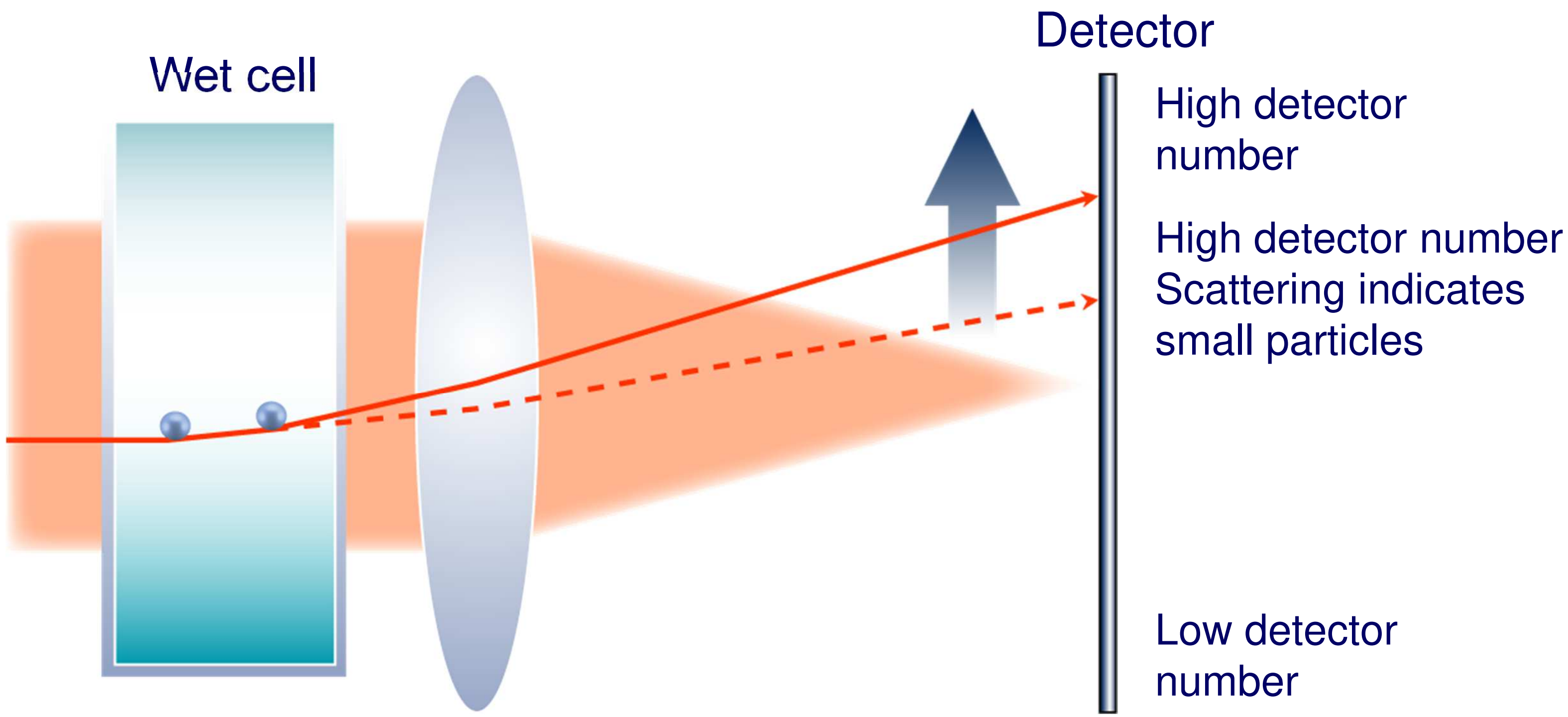
Data Quality

adding the right amount of sample

Terms used in this section:

- **Obscuration**: The amount of laser light blocked and /or scattered by the sample: a guide to the sample concentration
- **Signal-to-noise ratio**: The magnitude of the sample data in relation to the background data
- **Multiple scattering**: The phenomenon caused by light scattered by a particle being scattered by other particles when too much sample is added

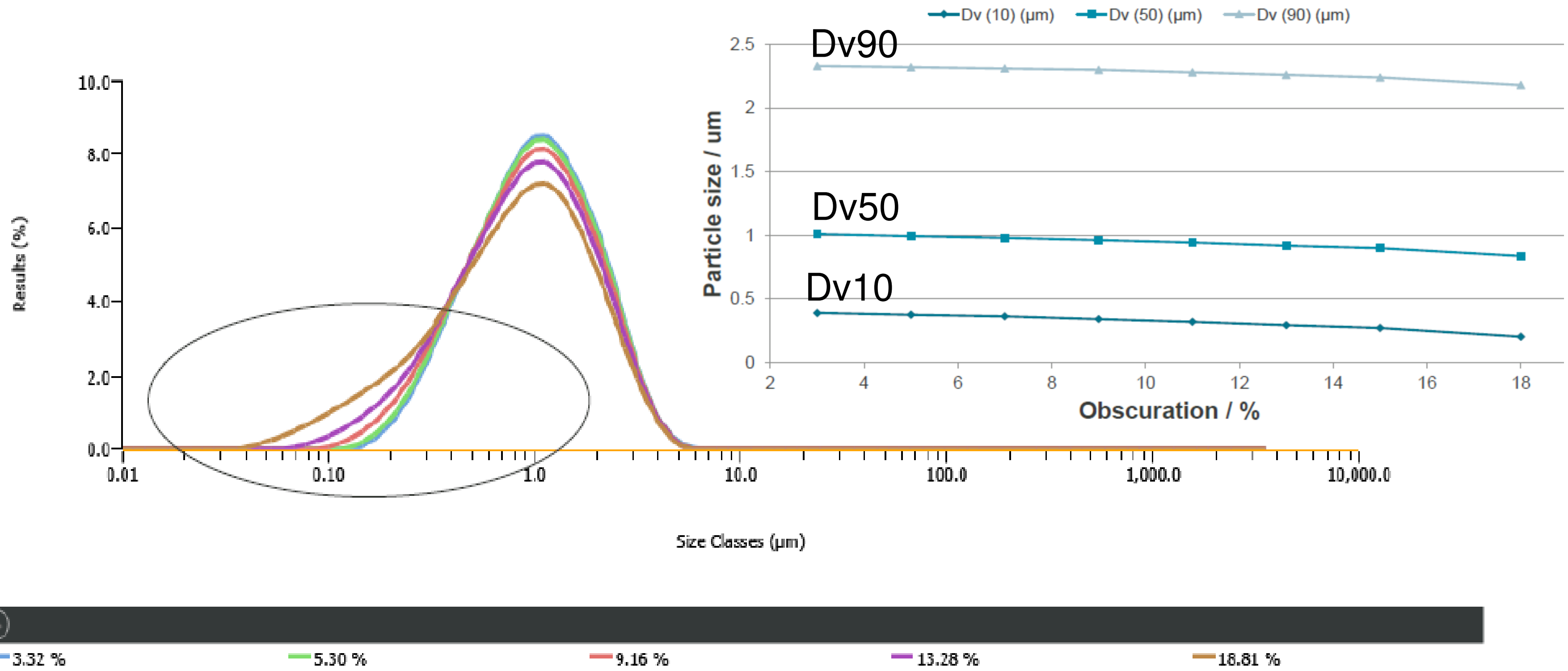
Multiple Scattering



Increasing the obscuration (concentration) makes multiple scattering more likely.
This leads to **higher angle scattering which corresponds to smaller particles.**

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 obscuration on the particle distribution.

Data Quality

How to improve data quality in dry measurement?

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?

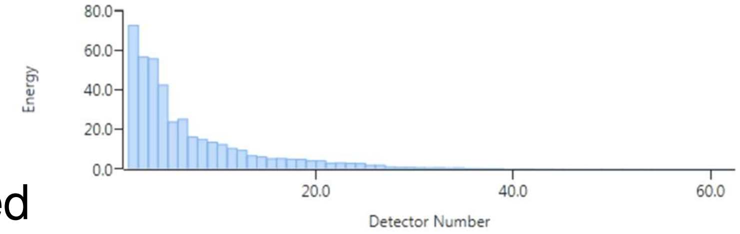
- if the sample obscuration is high, try lowering the feed rate or adjusting the hopper height
- try changing the height of the hopper, different basket, ball bearing
- try a different feed tray: often one tray design will deliver a more even sample flow for a particular material

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

Data Quality- Wet System

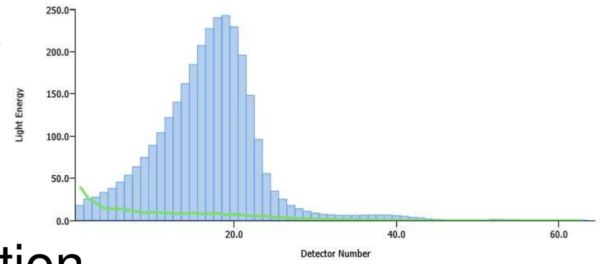
Background data

- 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

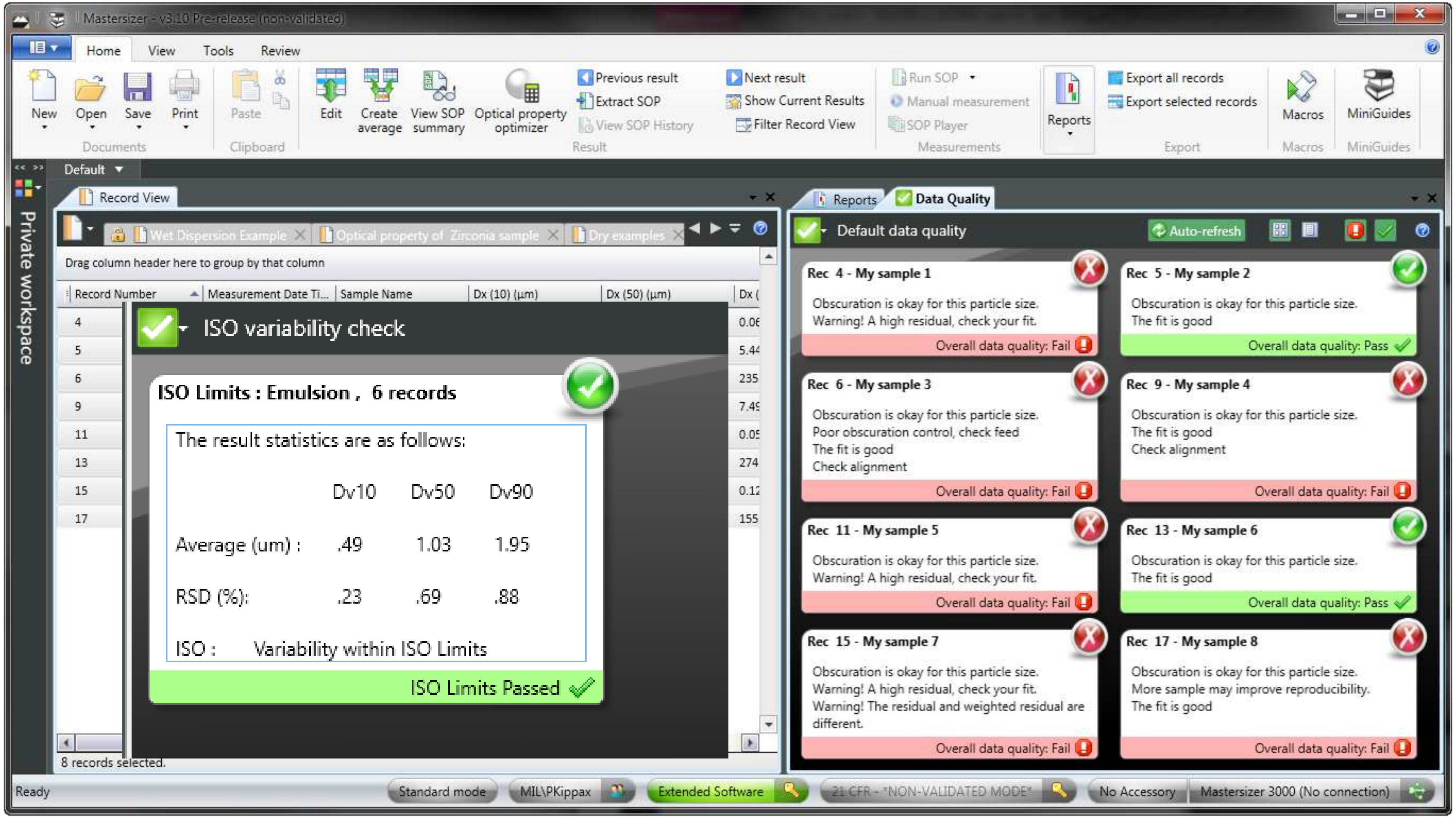


Sample data

- 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



Software :Data quality



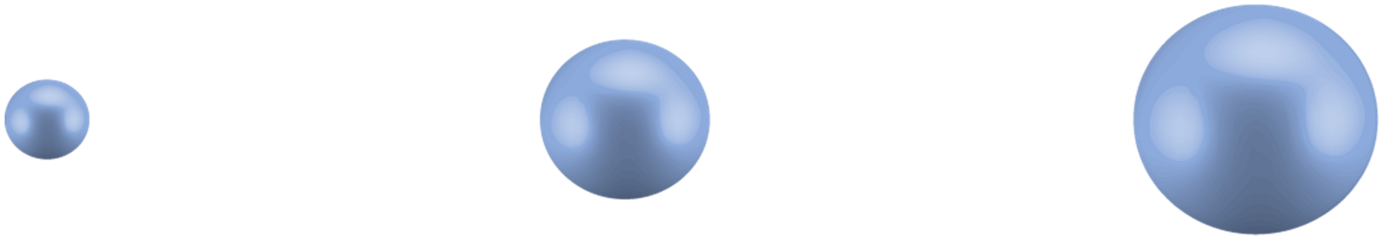
Understanding the size distribution

- The Mastersizer 3000 system is designed such that equal volumes of particles of different sizes produce a similar measured scattering intensity.

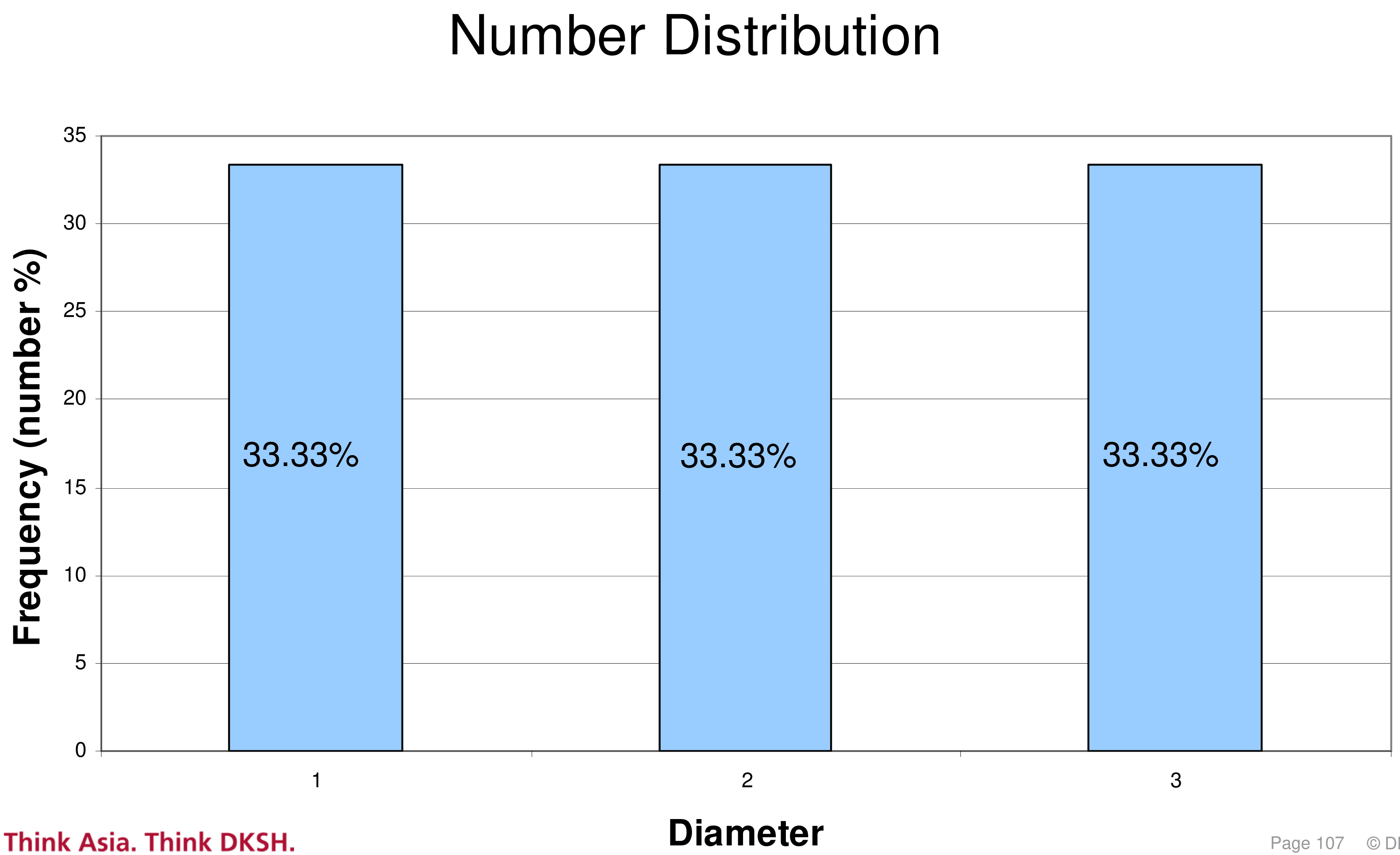
- *The size distribution is reported as a volume distribution as this best reflects the sensitivity of the system.*

- What does this mean in practice?

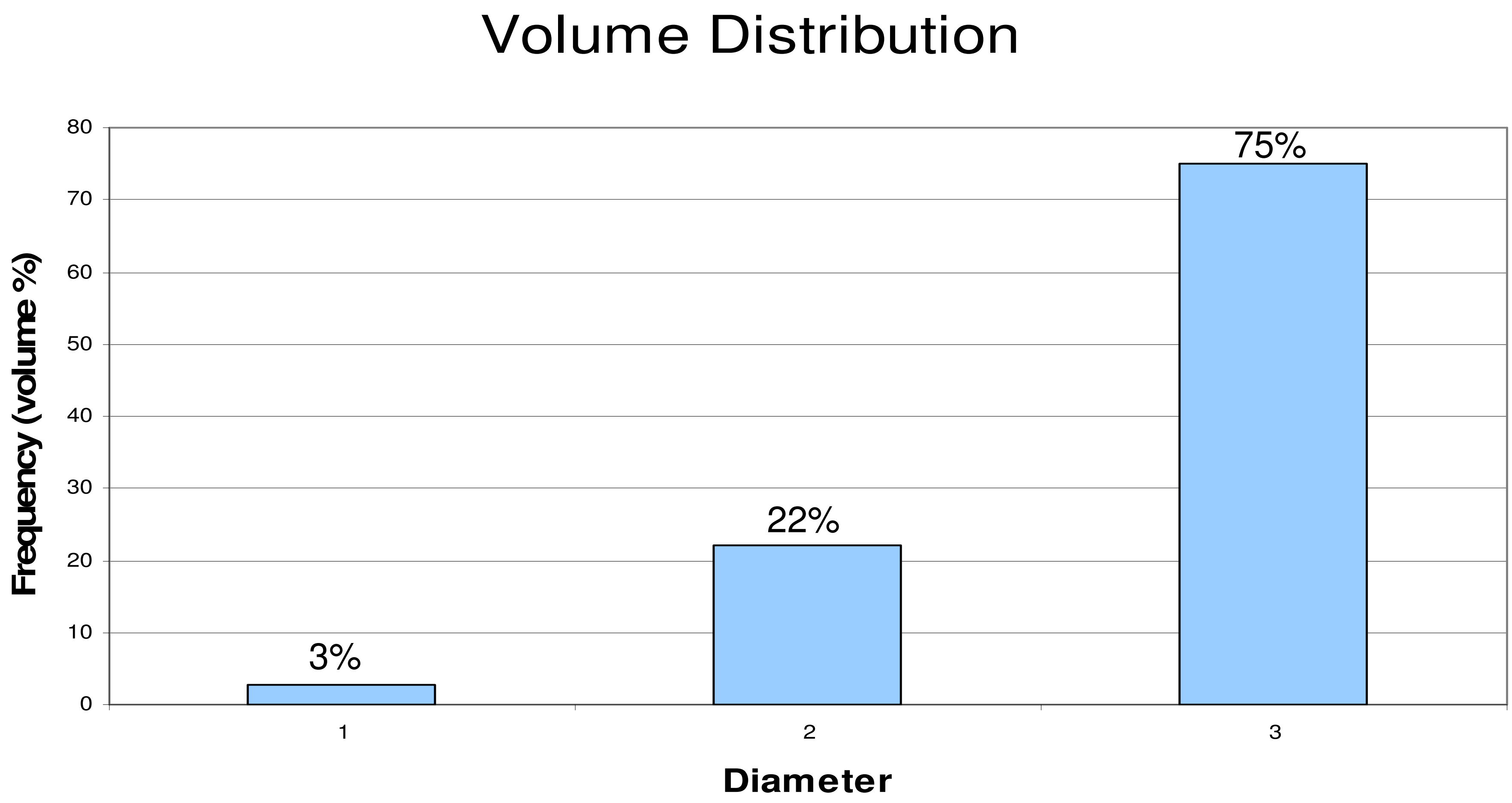
Imagine we have 3 particles with diameters of 1,2,3 units.



If these were measured by microscopy – a technique that produces a number distribution - then we would produce the following distribution…


Number Distribution
Frequency (number %)
35
30
25
20
15
10
5
0
33.33%
33.33%
33.33%
1
2
3
Diameter

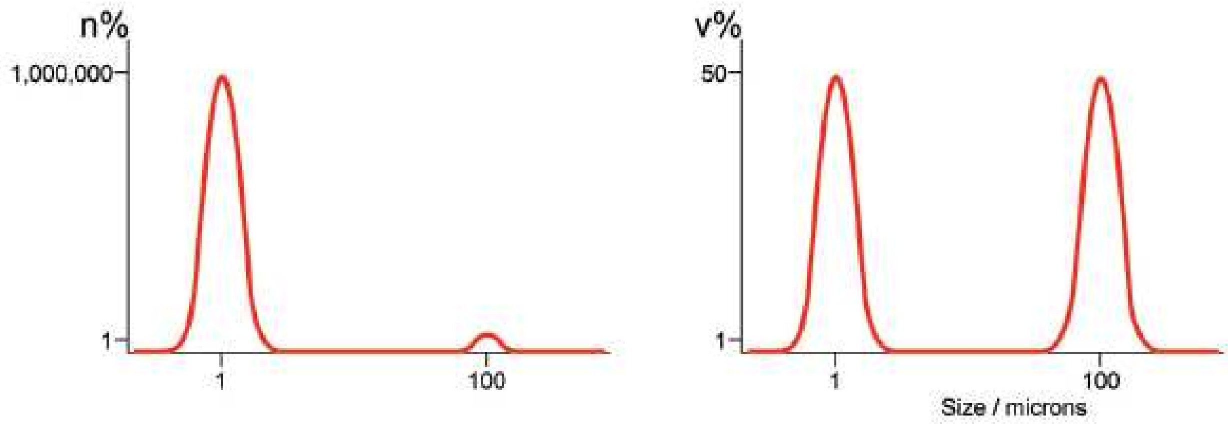
Understanding the size distribution



Volume distributions, as the name suggests, are based on the volume occupied by their constituent particles.

One million 1µm spherical particles will occupy the same volume as one 100µm particle.

The Number/Volume relationship



This should be remembered if you ever compare volume distribution results with a number-based distribution.

Conversion from one to another is often error prone!

Size distribution statistics

Size distributions are commonly described by their main statistical parameters,

such as the

- **mean (average)**
- **Median**
- **mode**

Most people are familiar with the arithmetic mean:

$$X_{nl} = D[1,0] = \frac{1+2+3}{3} = \underline{2.00}$$

Laser diffraction **does not use the arithmetic mean**

- **The volume of the particles is measured – not the number**

Consequently laser diffraction uses a **volume weighted mean**

Size distribution statistics

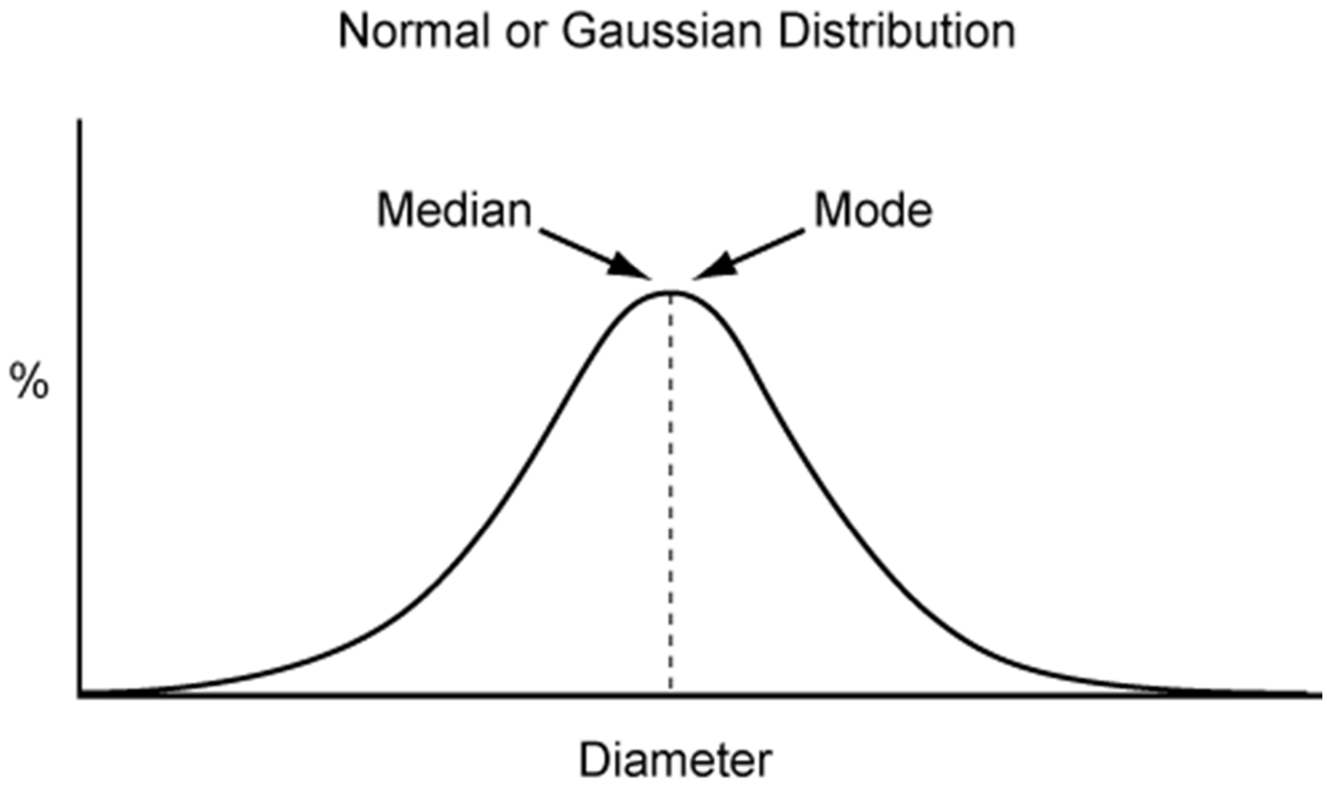
The mean is an “average” particle size.

There is no **unique** mean. We can average all particle diameters, weighting them according to:

- **volume**
- surface area
- Number

Each mean will show different sensitivities to **changes in the particle size distribution**

The median is the exact **midpoint** of the distribution
The mode is the most commonly occurring size class

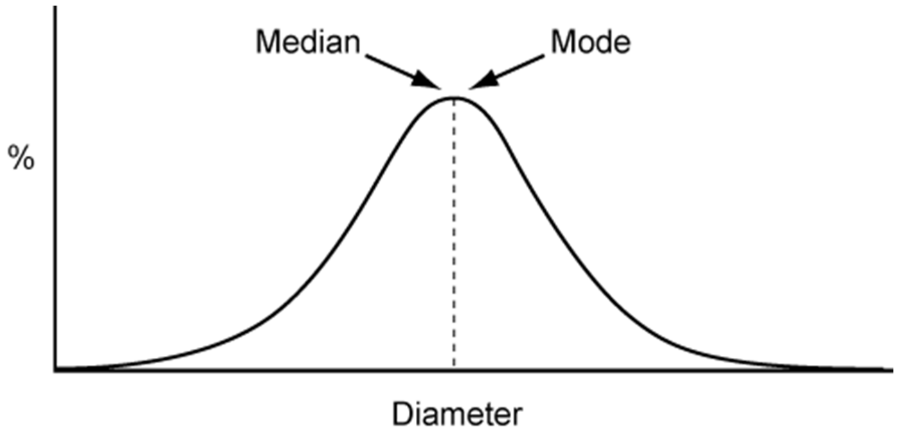


Other size distribution parameters

The span of a distribution provides a measure of the distribution width:

Span = (Dv90-Dv10)/Dv50

The span has a value of 1 for a symmetrical distribution



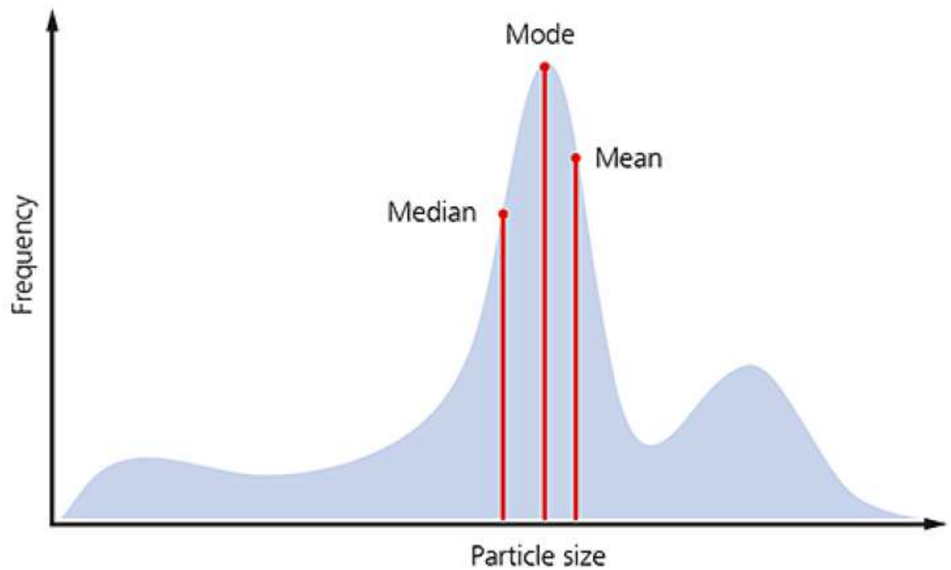
The uniformity is another parameter describing distribution spread.

- For narrow distributions this will be a small number

Particle size distribution statistics

Mean Mode Median

Mean = Average size of population
Median = Size in the middle of frequency distribution (Mid point)
Mode = Size with highest frequency



Size distribution statistics

The median of a particle size distribution is the size above [and below] **which we can find 50% of all particles**

It is commonly referred to as:

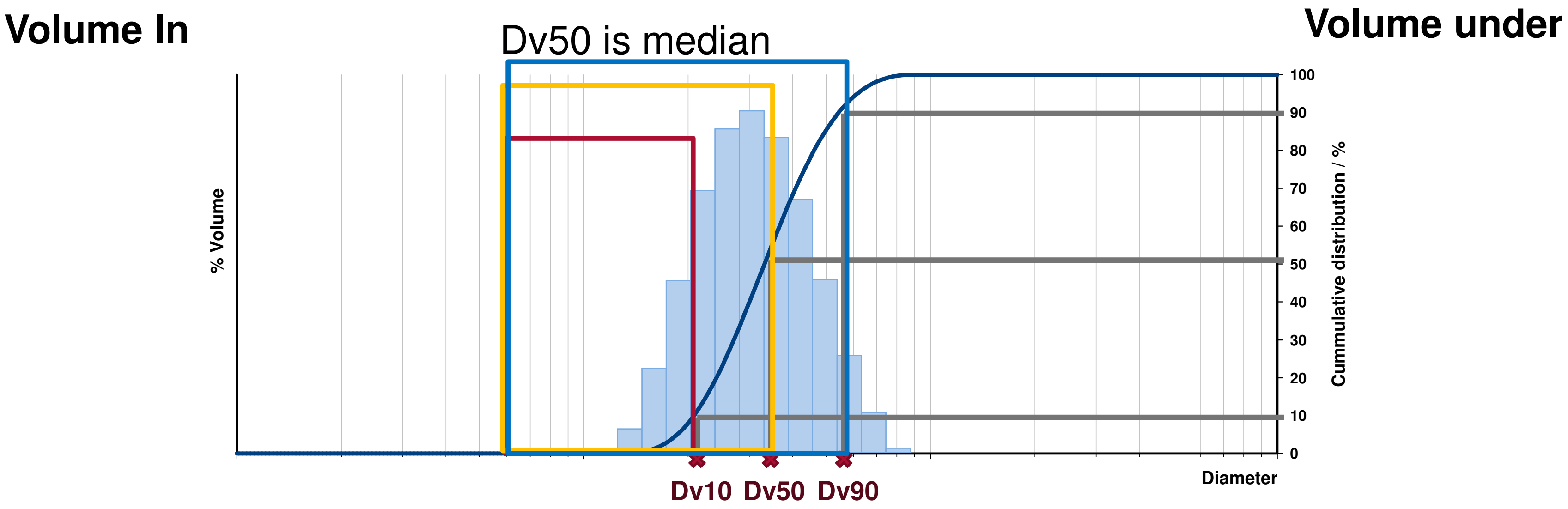
- The 50th percentile or the
- Dv50 or the D[v,0.5]

The “**v**” emphasises that the median was obtained from a volume distribution

Other percentiles such as the D**v**10 and D**v**90 can also be specified

PSD Statistics: Percentiles

Percentiles are the size below which there is a certain volume of the sample
Taken from the cumulative distribution



Record Number	Dx10 (um)	Dx50 (um)	Dx90 (um)
1	1.00	5.00	9.00

Percentiles

- The **Dv10, Dv50 and Dv90** are the default percentiles shown in the analysis report.
- These percentiles are commonly used to describe a particle size distribution but are by no means the only ones that can be used.
- Percentiles allows us to detect changes in our distributions that might have occurred due to changes in sampling, dispersion or measurement conditions.
- Report designer allows the user to build a report page detailing other measurement parameters.

Method Validation: what precision values are considered reasonable?

ISO13320-1: Section 6.4

- Dv50 - 5 different readings: COV < 3%
- Dv10 and Dv90: COV < 5%
- "Below 10µm, these maximum values should be doubled."

USP <429>

- Provides reproducibility ranges
- Dv50 or any central value: <10%
- Dv10, Dv90 or any non-central value: <15%
- "Below 10µm, these maximum values should be doubled."

EP 2.9.31 provides similar advice to USP<429>

THANK YOU

Think Asia. Think DKSH.