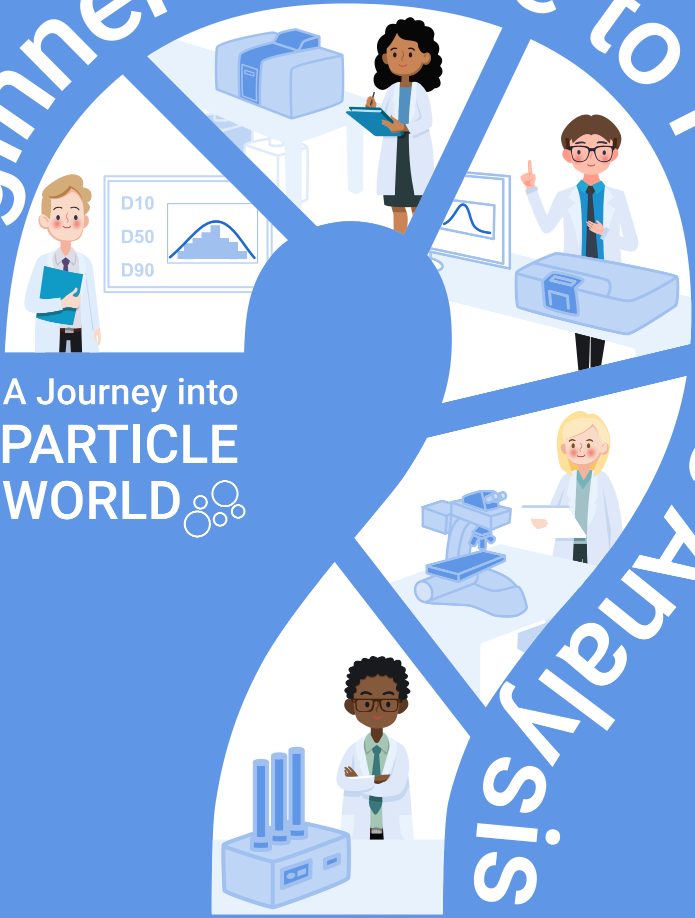
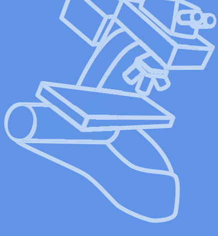
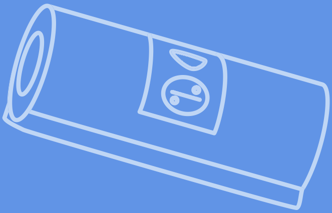
# Beginner's Guide to Particle Analysis

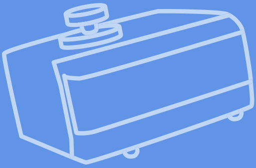


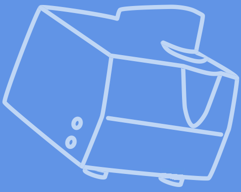
## A Journey into
## PARTICLE WORLD

Bettersize
BETTER PARTICLE SIZE SOLUTIONS









# PREFACE

Welcome to the fascinating world of particle analysis! In the realm of this field, a myriad of terms and concepts often perplex users, leading to confusion and frustration. Fear not! In this mini wiki handbook, we present a concise and enlightening guide to unravel the mysteries surrounding particle analysis.

Within these pages, we embark on a journey through six pivotal topics, spanning from the nanoscale to the microscale. Here, we dive deep into the most commonly used parameters, accompanied by clear and illustrative explanations. Our mission is to empower you with a fundamental understanding of particle analysis, making complex concepts effortlessly comprehensible.

From the ever-popular laser diffraction to the cutting-edge techniques of dynamic light scattering, dynamic and static image analysis, and light obscuration, we leave no stone unturned. Our goal is to equip you with the knowledge you need to decipher test reports and comprehend the intricacies of the measurement procedures.

This handbook is tailored for instrument users seeking to conquer the obstacles of particle analysis. Whether you're a seasoned veteran or an eager newcomer to the field, we hope this resource serves as your trusted companion on this enlightening journey.

Embrace the insights within these pages, and let the world of particle analysis unfold before you. Let's embark on this exciting adventure together!

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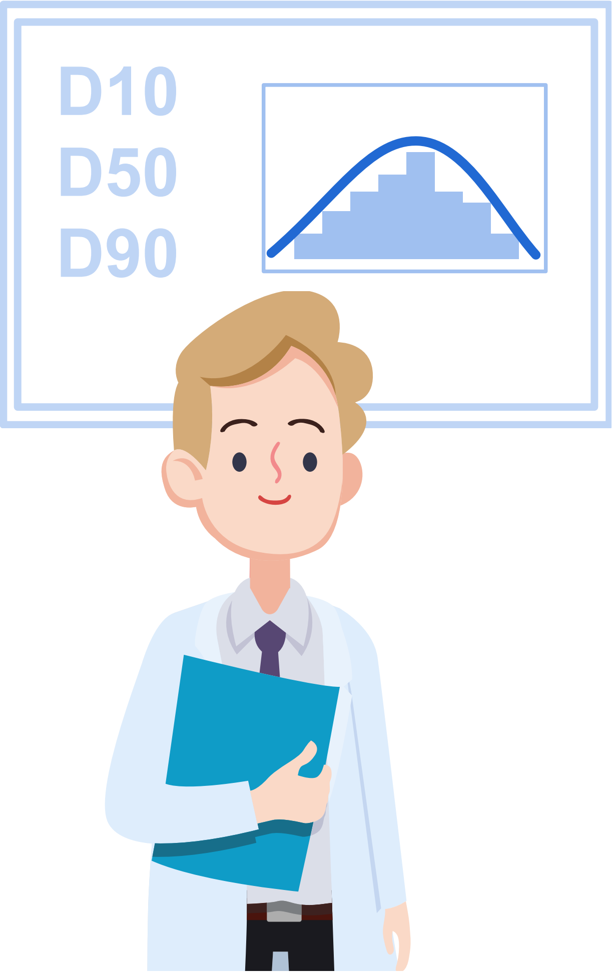
PART

A

# Basic Concepts of Particle Analysis



# 01

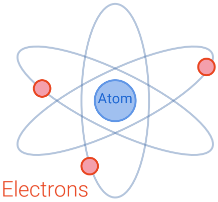
# WHAT IS A PARTICLE?

A particle or a corpuscule in older texts is considered a small discrete portion of something that could be large, small, microscopic, or even smaller. A particle physicist will consider a neutron, proton, or electron as a particle or even smaller when looking at quarks or leptons. In physical sciences, a particle is a small, localized object which can be described by several physical or chemical properties, such as volume, density, or mass. In physics, a particle refers to its geometry with a specific shape within a range of dimensions.

The difference between particles is enormous in size, from extremely small electrons to nanoparticles to microns and, ultimately, to coarse powders. Macroscopically speaking, not all particles are solids. Water droplets in the air, oil droplets, and gas bubbles in the liquid also qualify as particles as well.

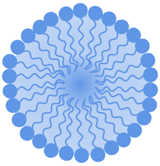
It is worth noting that the words “particulate” and “particulate matter (PM)” are terms used in environmental protection, which refer to particles suspended in the air.

Broadly speaking, when referring to size, particles can be described as macroscopic, microscopic, and subatomic. Macroscopic particles are the largest particles that can be seen by the naked eye. Microscopic particles are particles that are small enough to require at least a microscope to see them.



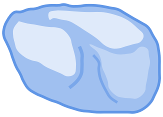
## 02

# HOW CAN PARTICLES BE DISTINGUISHED BY SIZE?



Micelle
(Nanoparticle)

Silica sphere
(Sub-micro particle)



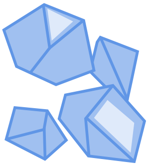
Carbide particle
(Microparticle)

A more accurate definition based on distinguishing between particles according to their sizes is as follows.

The first group contains particles whose sizes are smaller than 1nm, and an example of that category is water molecules whose size is from 0.3nm to 3Å.

The next grouping is nanoparticles, which generally fall into the size range between 1 and 100nm and can be such as naturally occurring iron oxide, iron sulfide, silver, and gold. Alternatively, there are those nanoparticles that are created and are being used in the manufacturing of scratchproof eyeglasses, concentrated pigments, crack-resistant paints, iron dextran injections, self-cleaning windows, thermosensitive polymers, ceramic coatings for solar cells, and nanoparticles coated with monoclonal antibody.

The size range of sub-micron particles is between 0.1 and 1μm. This grouping includes various types of particulate matter in seawater, ultrafine clays, pigments, and fine calcium carbonates.



Coal particle
(Microparticle)

*The figures do not show the true scale.

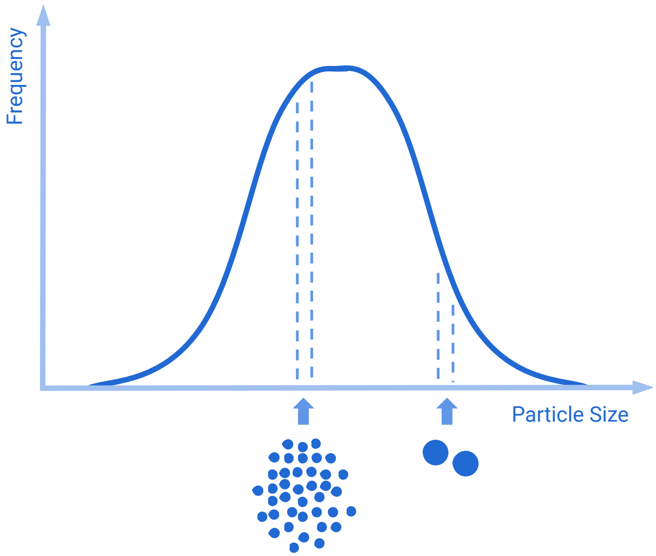
The largest group includes microparticles whose sizes range from 1 to 100µm, such as minerals, cement, pharmaceuticals, paints, toners, battery products, and 3D printing materials.

Coarse particles include plasters, fillers, sugar, coffee, and microcrystalline cellulose. Their sizes are between 100 and 1000µm.

Finally, there are granules that can be considered agglomeration of fine particles, which include pharmaceutical granules, extruded plastics, rice, and fertilizers. Commonly the sizes of granules are larger than 1000µm.

# 03

# WHAT IS PARTICLE SIZE DISTRIBUTION?

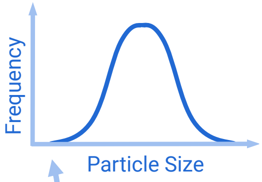


Particle size distribution describes the proportions of particles in different particle size intervals, which can be presented in various forms, such as a graph, a histogram, and a table. The particle size distribution can be displayed based on different distribution types, including number distribution, area distribution, and volume distribution. Particle size distribution is a statistical concept that affects the properties of products, including flowability, surface area, conveyance, extraction and dissolution behavior, reactivity, abrasiveness and even taste.

## 04

# HOW TO DESCRIBE THE PARTICLE SIZE DISTRIBUTION?

$$f(x) = \frac{1}{\sqrt{2\pi\sigma^2}} e^{\frac{-(x-\mu)^2}{2\sigma^2}}$$



| Interval | Percentage | Interval | Percentage |
|---|---|---|---|
| 0.100-0.111 | 0.00 | 0.138-0.155 | 0.34 |
| 0.111-0.124 | 0.00 | 0.155-0.173 | 0.29 |
| 0.124-0.138 | 0.24 | ...... | ...... |

Typically, particle size distribution can be displayed using the following three methods:

### Function

Particle size distribution is presented in the form of mathematical functions, such as normal distribution function, log-normal distribution function, and R-R distribution function.

### Graph

Cumulative distribution curve, frequency distribution curve, and histogram are commonly used to describe how the particle size of the sample distributes.

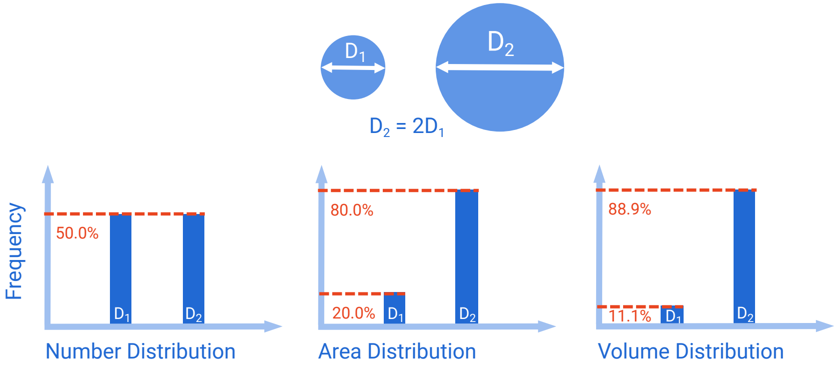
### Table

Percentages and their corresponding particle size intervals are listed in the table, providing quick and intuitive access to the particle size distribution. Cumulative distribution table and frequency distribution table are included. Table is the most commonly used form to show the particle size distribution.

## 05

# WHAT ARE THE DIFFERENCES BETWEEN DIFFERENT DISTRIBUTION TYPES?

The particle size distribution can be displayed in 3 different ways called distribution types. The most commonly used distribution type in laser diffraction particle size analysis is the volume distribution, as the volume is what is measured. The measurement technology restricts the distribution type used. For example, image analysis, in which the particle projections are identified, and the projected areas are calculated, resulting in area distribution, while the signal received by the laser diffraction particle size analyzer reflects the volume of the particle. When the distribution types are different, the results vary greatly. Suppose there are two particles, and these two particles are present in equal numbers. Their diameters are $D_1$ = 1μm and $D_2$ = 2μm, which is a 2 to 1 diameter ratio, then depending on which distribution type is used, the results will vary, as shown below.



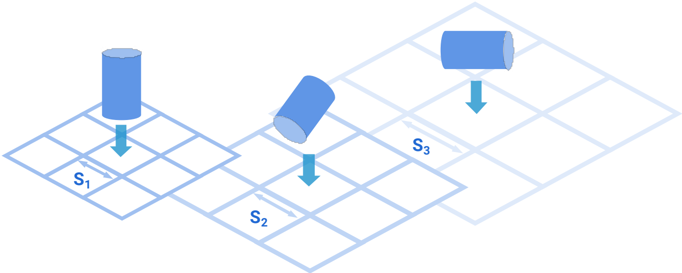
As the diameter ratio increases from 2 to 1, to 3 to 1, the number distribution will be constant at 50%/50%, whereas the percentage of $D_1$ will dramatically reduce for both the area and volume distributions.

# 06

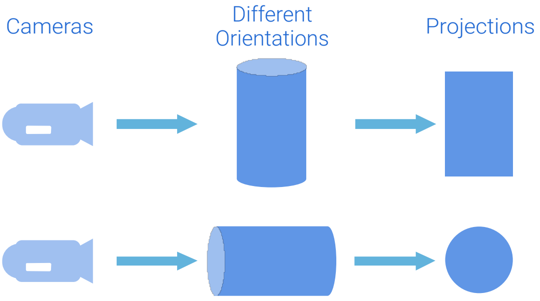
# WHAT IS EQUIVALENT SPHERICAL DIAMETER?

In most cases, the shape of the particle measured is irregular, which is not beneficial to communication on data, statistics, and analysis when using different measurement methods. Only perfectly spherical, non-porous, smooth, non-charged, mono-dispersed particles will give the same result on all techniques. Hence, the principle of an equivalent spherical diameter was introduced. The equivalent spherical diameter of an irregularly shaped particle is the diameter of a sphere whose physical, optical, or electrical property is the same as that of the particle. As examples, some typical equivalent spherical diameters are shown below.

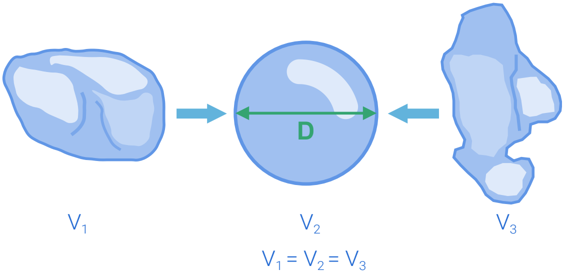
- **Sieve-equivalent diameter:** The sieve-equivalent diameter of a particle is the diameter of a sphere, which passes through a specific sieve aperture. For a sphere, the $S_1$ diameter would be the same diameter as the sieve-equivalent diameter. However, if the particles were not perfectly spherical, the orientation of the particle affects the sieve-equivalent diameter significantly. As shown below, the sieve-equivalent diameter varies due to different falling orientations.



- **Area-equivalent diameter:** The area-equivalent diameter of a particle refers to the diameter of a sphere whose projected area is the same as that of the particle. Area-equivalent diameter is commonly used in image analysis, in which the particle images are captured in random orientations, leading to different area-equivalent diameters of the same particle. However, results are obtained by averaging a large number of measurements, avoiding the impact of accidental errors.



● **Volume-equivalent diameter:** Particle size analysis based on laser diffraction technology commonly applies volume-equivalent diameter to present the particle size distribution. The laser diffraction equivalent diameter, which is a more accurate description, is calculated based on the scattering pattern of laser light as it is irradiated by the sample particles. However, the terms “volume equivalent diameter” and “laser diffraction equivalent diameter” are often used interchangeably, representing the equivalent diameter calculated based on the scattering pattern.



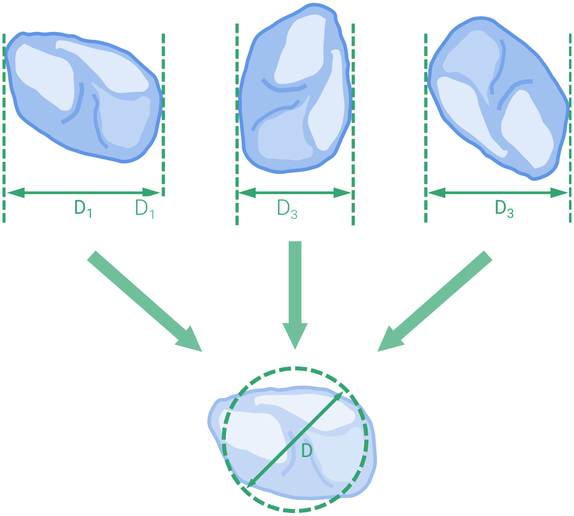
D: Volume-equivalent diameter of two diffrent particles

● **Stokes diameter:** Stokes diameter of a particle is the diameter of a sphere whose density and settling velocity are the same as those of the particle. Stokes diameter is also named equivalent spherical settling diameter. When particle size analysis based on gravitational and centrifugal sedimentation is carried out, measurement results using Stokes diameter are preferred.

## 07

# WHY DO WE USE EQUIVALENT SPHERICAL DIAMETER?

For perfect spheres, diameter can be used as a measure to describe their size. Real-world particles are characterized by their irregularities, resulting in several different size descriptions that could validly be used but with some difficulty. The equivalent spherical diameter was introduced as a valid base with which results from different techniques could be harmonized to facilitate and unify the illustration. The D shown below is the equivalent spherical diameter, which is not the largest or smallest diameter measured when interacting with the laser but can be the average of the sum of all that particle's orientations towards the laser.



## 08

# HOW TO DETERMINE MEAN, MEDIAN, AND MODE?

The median is the middle value in a list of values ordered from the smallest value to the largest value.

The mode is the value that most frequently occurs on the list of values.

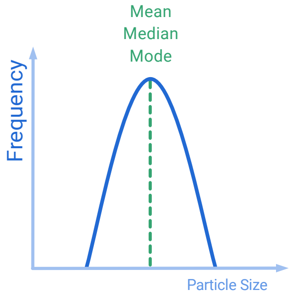
The mean is calculated by adding the values on the list and dividing the total sum of values by the number of values on the list. This is best described by the formula below.

The mean, denoted $\bar{x}$, of a set of values $x_1$, $x_2$,..., $x_n$, is defined as the sum of the numbers divided by $n$:

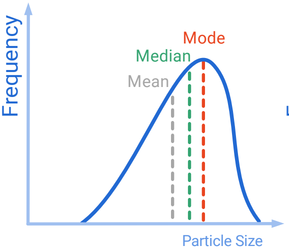
$$\bar{x} = \frac{x_1 + x_2 + x_3 + \cdots + x_n}{n}$$

From the normal/Gaussian distribution shown below, it can be seen that the mean, median, and mode have all got the same value. Also, a normal/Gaussian distribution has values that the nearer they are to the mean, they will occur more frequently than the values that are further away from the mean. So, the mean value = median value = mode value = D50 value = 50% value.

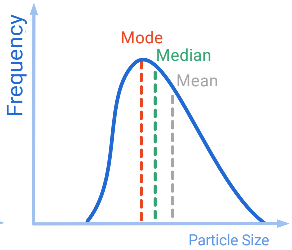
The values for mode and mean are different to the median for negatively and positively skewed distributions.



Normal/Gaussian Distribution

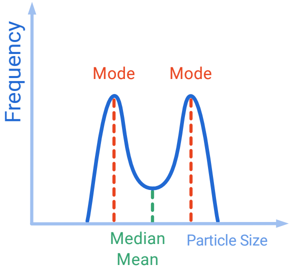


Negatively Skewed Distribution

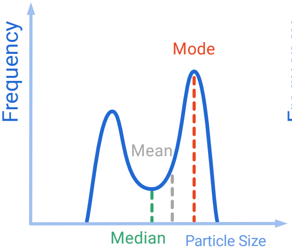


Positively Skewed Distribution

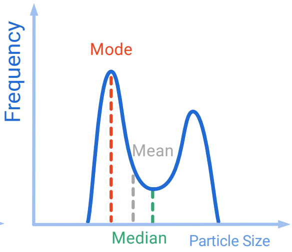
Normal/Gaussian distribution, negatively and positively skewed distributions for a bimodal distribution are shown below.



Normal/Gaussian Distribution



Negatively Skewed Distribution

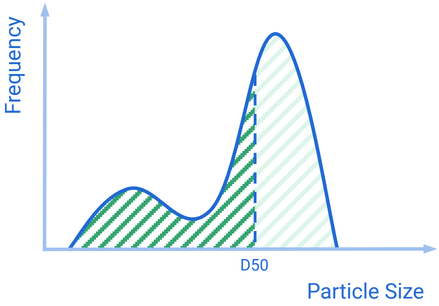


Positively Skewed Distribution

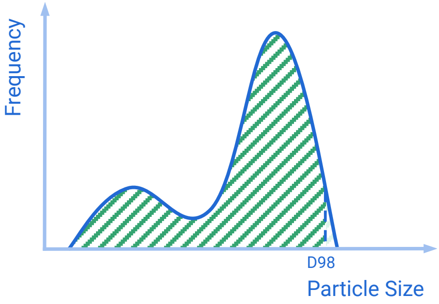
# 09

# WHAT IS D50?

The D50 of a particle size distribution refers to the particle size corresponding to the cumulative frequency of 50%. The D50 is a typical parameter to evaluate the quality of the powder. For example, if the D50 of a sample is 5μm by volume distribution, it means that the volume of particles less than 5μm accounts for 50% of the total sample volume, and likewise, the volume of particles greater than 5μm is also 50%. The D50 will be recognized in mathematical statistics as what we call the median.



Similarly, the definition of D10 and D90 can also be given, which are important parameters as well. Some manufacturing industries focus on specific typical values, such as D98 and D99, which are commonly used to indicate the coarse end of the sample.



# 10

# WHAT ARE D[4,3] AND D[3,2]?

D[4,3] and D[3,2] are the mean diameters of the particle size based on volume-weighted and area-weighted mean results, respectively. Different mean diameters are used to compare results with different techniques. For example, if results from the volume-based laser diffraction method are compared, then the D[4,3] volume-weighted mean result is preferred. If, however, the measurement results emanating from an image analysis system are compared, D[3,2] area-weighted mean result is prioritized.

The calculation of the mean diameter based on different weighting is shown below:

$$D[a,b] = \frac{\sum n \cdot d^a}{\sum n \cdot d^b}$$

**Number-weighted mean diameter: a=1, b=0**
**Area-weighted (Sauter) mean diameter: a=3, b=2**
**Volume-weighted (De Brouckere) mean diameter: a=4, b=3**

For example, two spheres are available, whose diameters are 1µm and 10µm, respectively. If the number-weighted mean diameter is required, then the number-weighted mean diameter is:

$$D[1,0] = \frac{1 \times 1^1 + 1 \times 10^1}{1 \times 1^0 + 1 \times 10^0} = 5.5$$

In this case, the mean diameter shows the quantitative relationship that corresponds to number-weighted.

Suppose the mean diameter of the system needs to be systematically reflected. In that case, the volume-weighted mean diameter shown below should be applied, which indicates that the volume of the large particle has occupied more than 99.9% of the system volume.

$$D[4,3] = \frac{1 \times 1^4 + 1 \times 10^4}{1 \times 1^3 + 1 \times 10^3} = 9.991$$

# 11

# WHICH METHODS CAN BE USED FOR PARTICLE SIZE ANALYSIS?

The most commonly used techniques for particle size determination are:

- **Laser Diffraction (LD)**
- **Static and Dynamic Image Analysis (SIA, DIA)**
- **Dynamic Light Scattering (DLS)**

Other approaches to particle size analysis are:

- **Gravity Sedimentation Particle Size Analysis**
- **Sieving**
- **Coulter Principle**
- **Scanning Electron Microscope (SEM)**
- **Light Obscuration (LO)**
- **Ultrasonic Extinction**
- **Time of Flight (TOF)**
- **Fisher Sub Sieve Sizer**

# 12

# WHAT ARE THE ADVANTAGES AND DISADVANTAGES OF DIFFERENT PARTICLE SIZING METHODS?

| | |
|---|---|
| **Laser diffraction** | **Advantages:** simple to use, fast analysis, wide measurement range, good repeatability and accuracy, optional sampling methods (wet/dry/online/small volume). |
| | **Disadvantages:** lower resolution for bimodal distribution whose peaks are close together, less suitable for nanoparticles. |
| **Static image analysis** | **Advantages:** morphology analysis, cost-effective, image is clear. |
| | **Disadvantages:** unsuitable for small particles (<2μm), more complex operation, slower analysis speed. |
| **Dynamic image analysis** | **Advantages:** morphology analysis, simple to use, fast analysis, good repeatability and accuracy, suitable for large particles. |
| | **Disadvantages:** unsuitable for small particles (<2μm), representativeness is affected by the sampling. |
| **Dynamic light scattering** | **Advantages:** wide measurement range, fast analysis, simple to use, excellent for nanoparticles. |
| | **Disadvantages:** measurement errors can occur for particles with wide particle size distribution, only suitable for transparent samples. |

| | |
|---|---|
| **Gravity sedimentation particle size analysis** | **Advantages:** continuous measurement, cost-effective, wide measurement range. |
| | **Disadvantages:** long measurement time, undersizes non-spherical particles, inaccurate for particles<1μm. |
| **Sieving** | **Advantages:** simple to use, cost-effective. |
| | **Disadvantages:** unsuitable for small particles (<38μm), measurement results are strongly dependent on operator methods, sieve apertures degrade with time, long measurement times for particles<100μm. |
| **Coulter principle** | **Advantages:** particle counting so higher resolution of peaks in a bimodal distribution, fast analysis, good repeatability, suitable for cellular analysis. |
| | **Disadvantages:** unsuitable for small particles and the particles with a wide particle size distribution, aperture needs to be changed for measurement of different sized particles, the maintenance is not simple, needs calibration regularly. |
| **Scanning electron microscope** | **Advantages:** accurate size analysis for ultrafine particles, clear image for particles with surface texture, high resolution, a standard technique to characterize nanoparticles. |
| | **Disadvantages:** low representativeness, apparatus is very expensive. |
| **Light obscuration** | **Advantages:** particle counting, fast analysis, measurement for sample with low concentration in liquid or gas is possible. |
| | **Disadvantages:** unsuitable for small particles, sample introduction is complicated, needs calibration regularly. |
| **Ultrasonic extinction** | **Advantages:** measurement for concentrated slurries without dilution, online measurement is also available. |
| | **Disadvantages:** measurement errors for particles with wide particle size distribution, apparatus is expensive. |

# Different Sizing

01

The laser diffraction technique is widely regarded as the most reliable technique for most industrial applications. The measurements are fast, repeatable, accurate, reproducible, and sensitive. It accurately measures the size of irregular as well as regular-shaped particles. It is not affected by the density of a particle or its porosity. It can measure wet, dry, or sprays either in the laboratory or online. The laser diffraction technique can also be combined with dynamic image analysis, providing more accurate measurement results when facing particles whose sizes are related to their orientation to the laser source, such as rod-like particles.

02

Static and dynamic image analysis is a measurement technique used for measuring particle size, which cover a wide size range with no need to change lenses or other components. In dynamic image analysis, wet and dry samples can be automatically measured with minimal human intervention, which is easy to be used, providing fast analysis and good repeatability, reproducibility, and accuracy.

03

Dynamic light scattering is primarily used for measuring sub-micron particles. However, for the particles whose sizes are greater than 3µm, dynamic light scattering is unsuitable because they have a failing in that the Brownian motion speed is very low, so low in fact that the sedimentation speed of the particles is greater than the Brownian motion speed.

04

Gravity sedimentation particle size analysis is a technique that relies on Stokes' law and has been a very popular method for those applications in which it is applicable. For the calculation of the particle size, the density of the material is necessary. Hence the method is not good for measuring low-density emulsions where the material does not settle or very dense materials which settle quickly. For the particles whose sizes are smaller than 2µm, this technique is limited due to the dominant Brownian motion.

# Particle Methods

**05**

Coulter principle was developed for sizing blood cells which are virtually mono-dispersed suspensions in a dilute electrolyte. Although particle number and volume-based particle size can be given, orifices have to be changed when measuring different samples. Therefore the operation is difficult. Besides, calibration should be carried out regularly.

**06**

Light obscuration is a particle counting method that is primarily used for measuring small amounts of contamination in clean room facilities, such as pharmaceutical labs and silicon chip manufacturing facilities. Detection of contamination level in aircraft fuels is an important application as well. So basically, it is a low-concentration detection technique that needs constant calibration and is unsuitable for most industrial applications.

**07**

Sieving is an old technique used to separate particles with different size ranges, which is easy to use and cheap. However, the measurement result is greatly affected by human errors.

**08**

Scanning electron microscope requires elaborate sample preparation and is slow. Though clear images of particles are provided, there can be large operator-to-operator variability on the same sample because the particles are analyzed manually, and the observation area greatly varies, leading to poor representativeness.

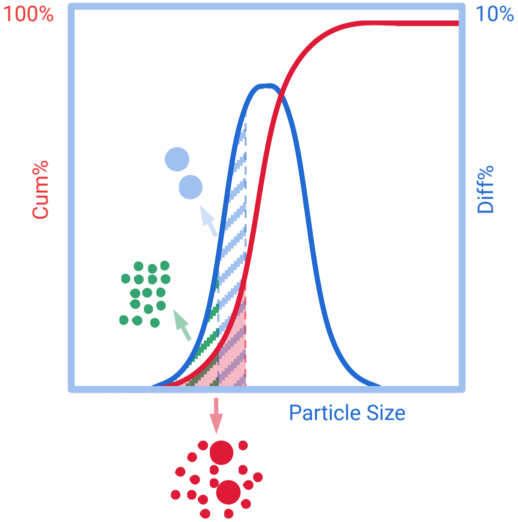
**09**

The ultrasonic extinction is primarily used in an online system for the detection of undiluted samples. In reality, it can work for some applications, but it is very expensive. To properly function, it needs to be set up with up to 13 different parameters, which are difficult to be found and sometimes non-existent.

# 13

# WHAT ARE FREQUENCY AND CUMULATIVE DISTRIBUTIONS?

Frequency distribution is also known as interval distribution or differential distribution. It describes the percentage of particles in a particle size band, e.g., between 1 and 2μm. Cumulative distribution is also known as integral distribution. It describes the percentage of particles that are larger or smaller than a certain particle size value, which can be calculated through the cumulative addition of interval distributions. The red curve below represents the cumulative distribution curve corresponding to the left ordinate. The particles included in the red area are equivalent to those in the green and blue areas under the frequency distribution curve.



# 14

# HOW DO WE CALCULATE REPEATABILITY?

In laser diffraction particle size analysis, repeatability refers to the measurement difference between multiple measurements of the same sample under the same measurement conditions in a short period of time. The repeatability is characterized by the following conditions:

● The measurements should be carried out following the same measurement procedure under the same measurement conditions and in the same location.

● The measurements should be implemented by the same operator.

● The sample should be the same.

● The particle size analyzer used for the measurements should be the same.

● The measurement should be repeated over a short period of time.

The calculation of the repeatability for each size channel, such as D50, is shown below:

$$\delta = \frac{\sigma}{x} = \frac{1}{x}\sqrt{\frac{\sum (x_i - x)^2}{n-1}}$$

where $n$ is the number of measurements (usually n ≥10), $x_i$ is the each individual measurement result, $x$ is the mean of the measurement results, $\delta$ is the standard deviation.

# 15

# WHY IS REPEATABILITY SO IMPORTANT?

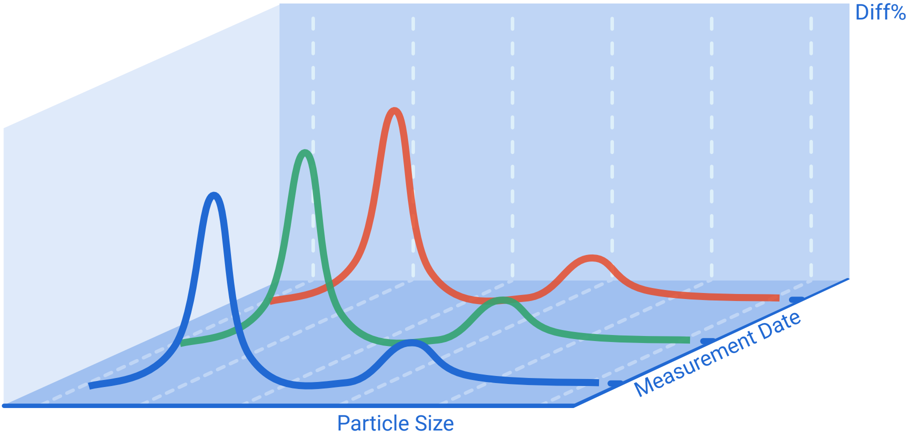
The repeatability of multiple measurements is a significant indicator used to describe either the working status of a particle size analyzer or the validity of the measurement procedure. Good repeatability shows a good working status of the whole measurement system, including laser source, detectors, signal transmission system, control system, circulation and dispersion system, and software. It can also confirm that a good measurement procedure has been utilized for the sample measurement.

In other words, the working status of the instrument is stable, the working environment is suitable for the operation of the instrument, and the instrument can be competent for measurement. Suppose that the repeatability is not ideal, a system inspection is necessary. In addition to the inspection of the above-mentioned components and systems, the choosing of the working environment is essential as well. Environmental factors, including input voltage, sunlight, external vibration, and electromagnetic interference, affect the working status of the instrument. For a smooth operation, the instrument needs to be isolated from the above-mentioned external factors that may cause bad repeatability.

Furthermore, a proper measurement procedure must be adopted that suits the sample to be measured in the correct solvent (wet measurement) or correct pressure (dry measurement). Other factors to be considered include the choice of admixture, choice of surfactant, measurement time, ultrasonication duration, and ultrasonication strength. For the measurement of nanoparticles, the zeta potentials in the chosen media need to be considered.

## 16

# WHAT IS REPRODUCIBILITY?



Reproducibility demonstrates the consistency of measurements, in which the sample will be measured after changing at least one measurement condition, such as measurement time, operator, measurement location, instrument used for measurement, and batch sampling. The calculation of reproducibility is the same as that used for repeatability.

## 17

# WHY IS REPRODUCIBILITY SO IMPORTANT?

For laser diffraction particle size analysis, the implementation of the reproducibility test can be used for:

**Evaluation of the stability of the instrument's long-term operation.**

**Evaluation of the stability of the sampling method.**

**Evaluation of the standardization of operation steps.**

**Evaluation of the measurement consistency.**

**Evaluation of the influence of the working environment on the measurement.**

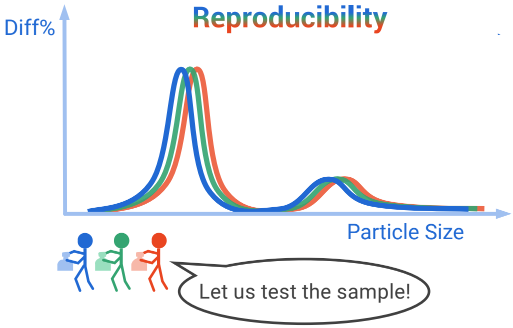
The reproducibility test requires qualified and stable reference materials to be used, which do not change during the measurement. These reference materials or standards, as they are normally known, should be stored under their prescribed conditions. They can be purchased from the analyzer supplier or ordered direct from a standard supplier of good reputation by the users.

The reference materials should be used before the expiry date. A reproducibility test should be carried out every three months. If the reproducibility lies within the acceptable error range, the working status of the instrument is confirmed; if not, a system inspection is necessary.

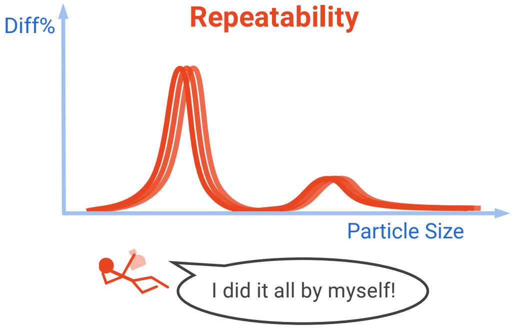
# 18

# WHAT ARE THE DIFFERENCES BETWEEN REPEATABILITY AND REPRODUCIBILITY?

Similarity, repeatability and reproducibility are obtained from multiple measurements made on the sample. However, repeatability is characterized by the same measurement conditions, reflecting the stability of the current working status of the instrument and the working environment.



In contrast, the measurement conditions are different in the reproducibility test, which is used to evaluate the long-term stability of the instrument operation, the stability of the sampling method, and the operator's influence on the measurement results. The repeatability and reproducibility tests must be carried out regularly, ensuring that the instrument works properly.



## 19

## WHAT ARE THE FACTORS THAT AFFECT REPEATABILITY AND REPRODUCIBILITY?

In case the repeatability or reproducibility is not ideal, the following factors should be considered:

**Working status of the instrument**

The working status of the whole system, including laser source, detectors, signal transmission system, control system, circulation and dispersion system, and software.

**Measurement procedure**

Background signal, obscuration level, measurement parameters, medium temperature, type and dosage of dispersing agent, solvent type, ultrasonication duration and strength, and cleanliness of the sample cell.

**Batch sampling**

Sampling method, including sampling from the process line and preparation in the laboratory, such as re-homogenization of the sample.

**Working environment**

Input voltage, temperature, external vibration source, and electromagnetic interference.

**Properties of the sample**

Fragility, the solubility of the sample, and the possibility of the expansion or agglomeration of the particles.

## 20

# WHAT ARE THE ACCEPTABLE ERROR RANGES OF REPEATABILITY?

According to ISO 13320: 2020, six consecutive measurements should be carried out to satisfy the repeatability test. The average values of D10, D50, and D90 should be calculated, and the acceptable error ranges are:

- Each measured value of

**D10**

should not be deviated from the average value by more than

**±2%**

- Each measured value of

**D50**

should not be deviated from the average value by more than

**±1.5%**.

- Each measured value of

**D90**

should not be deviated from the average value by more than

**±3%**.

## 21

# WHAT IS ACCURACY AND HOW DO WE CALCULATE IT?

In laser diffraction particle size analysis, an accuracy test describes consistency between the measurement result of the certified reference material and its nominal value. For the accuracy test, each certified reference material needs to be measured at least three times individually. Every test should be conducted in the presence of sufficient and proper sample concentration, enabling good representative results.

Based on ISO 13320: 2020, the acceptance limit $U_{lim}$ for the accuracy test can be calculated through the following formula:

$$U_{lim} = \pm CF\sqrt{u_{crm}^2 + u_p^2}$$

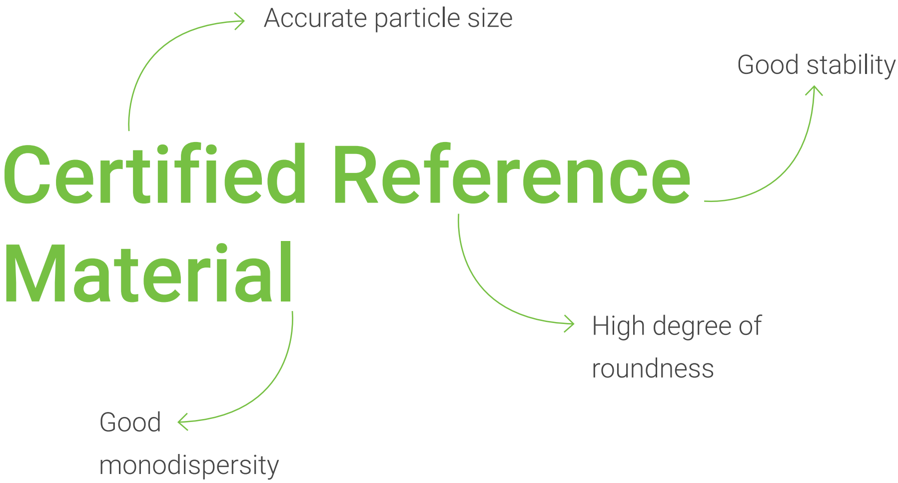
where $u_p$ is the maximum acceptable instrument uncertainty value for a laser diffraction system, $CF$ is the coverage factor. $u_p$ and $CF$ are shown in the following table. $u_{crm}$ can be found in the instructions provided for the certified reference material. The coverage factor describes the confidence of certain data within a defined standard deviation, which is dependent on the application area. For example, in the metrology industry, when uncertainty to a confidence interval of 95% is given, the coverage factor can be considered 2.

| Parameter | $u_p$ | $CF$* |
|---|---|---|
| D10 | 2% | 2-3 |
| D50 | 1.5% | 2-3 |
| D90 | 2.5% | 2-3 |

*The value of $CF$ is between 2 and 3, which is based on the desired level of confidence.

# 22

# WHAT IS A CERTIFIED REFERENCE MATERIAL (CRM)?



A certified reference material is a material used for instrument calibration or performance verification, whose typical values are determined by reference material manufacturers, sometimes by counting, laser diffraction, sieving, or sedimentation. For this reason, there are sometimes small differences between results from users and the specifications from the reference material providers.

In addition to accurate particle size, the certified reference material should also be characterized by its good stability within a period (For example, one year), good monodispersity with a low variation coefficient, and a high degree of roundness (95-98% roundness).

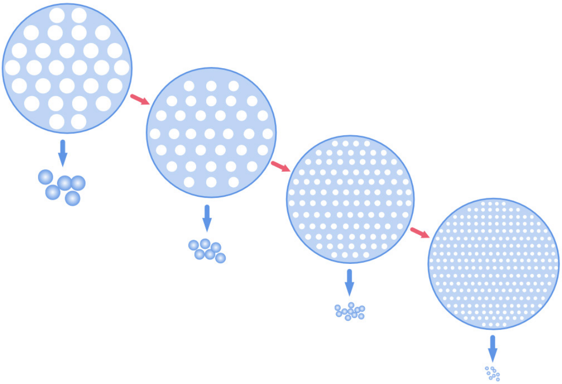
## 23

# WHAT IS A WORKING STANDARD?

A working standard, also known as an in-house standard or secondary standard, is an alternative to a certified reference material provided by reference material manufacturers. The working standard is used for instrument calibration, performance verification, repeatability and reproducibility test, accuracy test, and demonstration. Compared to certified reference materials, working standards are much more cost-effective but probably cannot provide the same level of accuracy. However, these working standards are also easier to produce and meet the ISO requirement.

# 24

# WHAT IS THE RELATIONSHIP BETWEEN MESH AND MICRON?



Mesh refers to the number of openings in a filter screen per linear inch. For example, if there are 50 openings in one inch on the filter screen, the mesh of this filter screen is 50. The larger the mesh is, the smaller the filtered particles will be. The size of the opening varies in the case of different mesh sieves. Various relationships between mesh and micron defined by different standards are available. The most commonly used standards are the ASTM standard, the ISO standard, and the Tyler standard.

The relationship between mesh and micron based on Tyler standard is listed below.

| MESH | MICRONS | MESH | MICRONS | MESH | MICRONS | MESH | MICRONS |
|---|---|---|---|---|---|---|---|
| 20 | 850 | 70 | 212 | 270 | 53 | 1000 | 13 |
| 25 | 710 | 80 | 180 | 325 | 45 | 1250 | 10 |
| 30 | 600 | 100 | 150 | 400 | 38 | 1670 | 8.5 |
| 35 | 500 | 120 | 125 | 450 | 32 | 2000 | 6.5 |
| 40 | 425 | 140 | 106 | 500 | 28 | 5000 | 2.5 |
| 45 | 355 | 170 | 90 | 600 | 23 | 8000 | 1.5 |
| 50 | 300 | 200 | 75 | 700 | 20 | 10000 | 1.3 |

PART

B

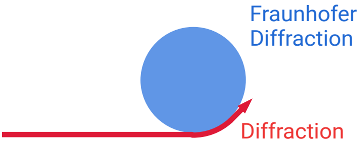
# Laser Diffraction Particle Size Analysis

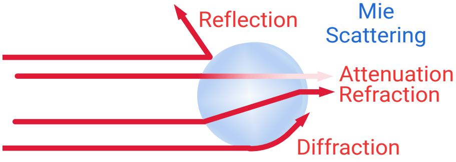
# 25

# WHAT ARE THE MIE SCATTERING THEORY AND THE FRAUNHOFER DIFFRACTION THEORY?

The Mie scattering theory is the Mie solution to Maxwell's electromagnetic equations that accurately describes the scattering of light by homogeneous spheres. In the Mie theory, parameters such as refractive indices and absorption coefficients of the particle and the medium are used to calculate the particle size. Computers in the 1970s were only powerful enough to compute diffraction data with the simpler Fraunhofer approximation. Mie has been widely used since the 1980s with the evolution of the computer, and the Mie theory is a rigorous solution for the light scattering pattern and particle size distribution. The results obtained by the Mie theory are accurate since all the optical properties are used and considered. Therefore, particle size measurement using the Mie theory delivers more reliable and robust results for particles whose sizes range from the sub-micron to the millimeter level. For this reason, the Mie scattering theory is widely used in modern laser diffraction particle size analyzers.

The Fraunhofer diffraction theory, on the other hand, is a simple approximate expression of the Mie scattering theory, which describes the maximal and minimum light scattering angles as a function of an object's size at any given wavelength. However, it does not require any knowledge of the optical properties and is preferred used for measurement when the particles are large and opaque with small light scattering angles.



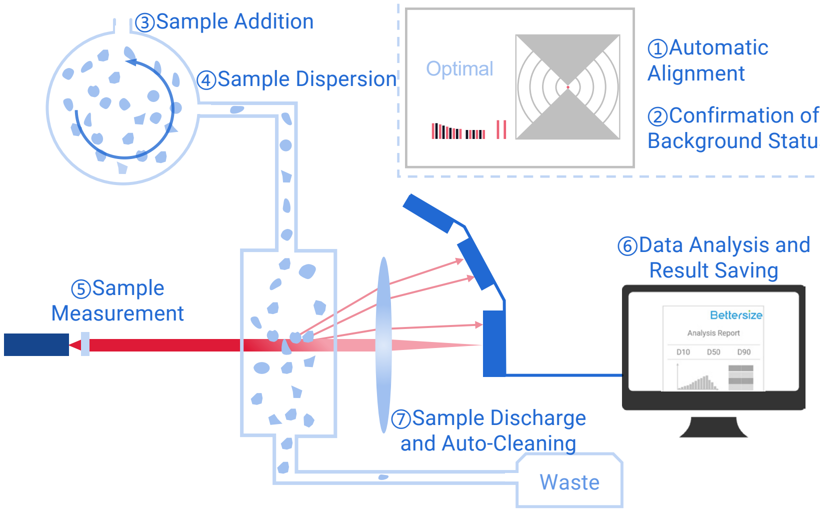


Laser diffraction particle size analyzers from Bettersize offer both optical theories for particle size analysis, while Mie is the default, and Fraunhofer is used for measuring particles accurately if their sizes are greater than 25 microns.

## 26

# HOW IS A MEASUREMENT MADE WITH A LASER DIFFRACTION PARTICLE SIZE ANALYZER?

In laser diffraction particle size analysis, the measurement is carried out using the following sequence:



### ① Automatic Alignment

This is an adjustment function that enables the focused laser to pass through the Fourier lens and coincides with the center of the forward detector. The alignment will be carried out automatically until the laser light impinges perfectly on the center of the forward detector. Once aligned, the detectors will receive the correct scattering light signal for the baseline measurement. Before alignment, the sample cell should be completely filled with measurement medium, and bubbles and dirt need to be eliminated when using the wet method. Gas needs to be introduced to the measurement zone when using the dry method.

### ② Confirmation of Background Status

A high background signal may be caused by optical or electrical noises, e.g., optic noise that is caused by the dirty sample cell or even by pure media. To zero out the influence of those noises, the background signal should be measured prior to the measurement, such that it can be subtracted from the total light scattering signal on completion of the measurement.

### ③ Sample Introduction

After confirming a good background, the sample can be introduced (or pre-dispersed sample) to the dispersion module.

### ④ Sample Dispersion

Once inside the dispersion module, the sample will be dispersed using stirring and ultrasonication in the wet method. The pressure will be adjusted, and collisions and shear force will be ensured in the dry method.

### ⑤ Sample Measurement

A fully dispersed sample will be transported to the sample cell, where the laser irradiation occurs. A laser source emits a beam of monochromatic and coherent laser light. This laser beam is then filtered, expanded, and collimated to ensure parallel rays. If particles are in the beam's optical path, they will interact with the light in the form of scattering. A portion of light rays is scattered outward at certain angles with respect to the principal axis. Larger particles scatter greater amounts of light at smaller scattering angles, while smaller particles scatter greater amounts of light at larger scattering angles.

### ⑥ Data Analysis and Result Saving

Signals caused by scattered lights can be received by the detectors and transmitted to the computer to be analyzed so the system can generate a particle size distribution.

### ⑦ Sample Drainage and Auto-Cleaning

After the measurement, the sample will be drained, and the auto-cleaning will be activated.

## 27

# WHICH MATERIALS CAN BE ANALYZED BY A LASER DIFFRACTION PARTICLE SIZE ANALYZER?

Dry powders, liquid suspensions, and liquid/particle sprays can be analyzed by a laser diffraction particle size analyzer. The dry powder can be introduced using a dry dispersion module, in which the dry powder is sucked into the measurement zone due to the presence of a Venturi device. Alternatively, the dry powder can be added directly to the wet dispersion module or prepared as a liquid suspension. However, the prerequisite is that the dry powder is insoluble in the medium and does not react with the medium. Some materials can be introduced through a wet dispersion module or a dry disperser. The choice of which one to use depends on the repeatability and accuracy of both sets of measurements.

# 28

# HOW MANY METHODS ARE THERE FOR SAMPLING?

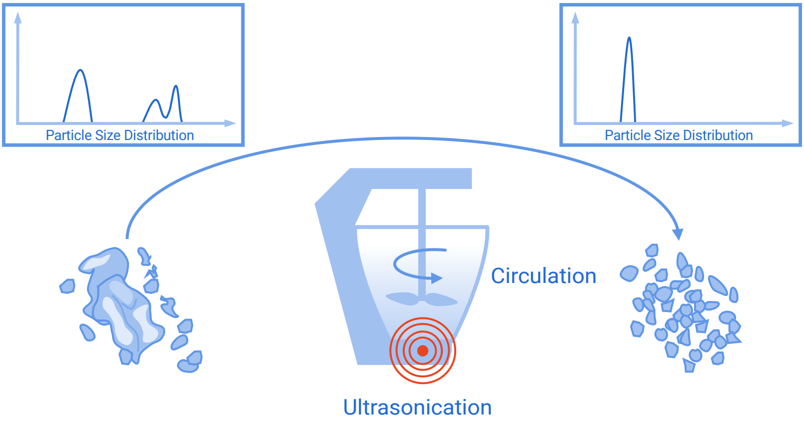
Laser diffraction particle size analysis is a process that characterizes the particle size distribution of a large number of substances by testing and analyzing a small number of samples. Representative and convincing measurement results are inseparable from a reasonable sampling process. Accordingly, sampling is the first and most important part. The sampling is a process of obtaining a “laboratory sample ($10^n$ g)” from a “large amount of sample ($10^n$ kg)”, and then obtaining a “measurement sample ($10^n$ mg)” from the “laboratory sample”.

**Acquisition of Laboratory Sample from the Process Stream:**
The best method of sampling from the process is to remove the sample from a flowing powder stream rather than from a static pile like a silo or a drum. If the only possibility is a static powder pile, samples should be removed from different positions and different depths in the pile or drum, which will be mixed to obtain a representative laboratory sample.

**Acquisition of Test Sample:**
Users can use a spinning riffler to acquire a representative test sample from the laboratory sample. A spinning riffler is a device that divides the laboratory sample into many fractions whose characteristic composition corresponds exactly to that of the original laboratory sample. Each fraction can be used as a test sample. If the fraction is excessive, it needs to be further split by the spinning riffler again to make sure that the entire subsample will be used for a single measurement. A spatula or spoon can be used to sample from the laboratory sample if the sample is held in a solid glass jar with a screw cap. However, the jar must be at least 30% empty to allow the rest of the powder in the jar (70% or less) to be tumbled for re-homogenization. The tumbling should be done seven times in succession before taking the sample from the jar. The jar must never be shaken as vibration creates stratification causing the user to sample coarse particles from the top of the jar. This will not be representative and will lead to a test producing a larger particle size analysis.

## 29

# HOW ARE PARTICLES DISPERSED WHEN USING THE WET METHOD?

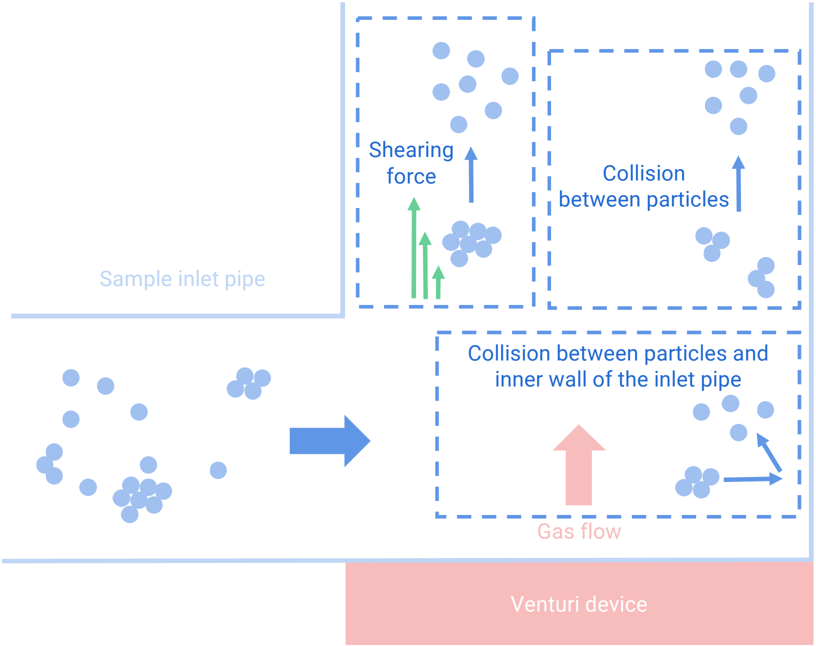


In laser diffraction particle size analysis, inaccurate results can be caused by particle agglomerating in the suspension, especially when they are fine. Therefore, a full dispersion of the sample prior to measurement is essential.

Three effective methods are available for the sample dispersion when using the wet method. Primary dispersion of aggregates can be achieved by circulating the suspension through a wet flow path containing a stirring blade. In addition, variable levels of ultrasonication can be helpful in deagglomeration. Thirdly in some systems, the downward slope of the circulation tank and a higher pump speed can effectively prevent the precipitation of particles with a higher specific gravity or larger particle size in the wet flow path, leading to a complete dispersion and hence a statistically good representation of the particles. For particles that float on the meniscus of the water, an appropriately diluted surfactant may be necessary to lower the surface tension. Hence the particles can be absorbed into the water.

## 30

# HOW ARE PARTICLES DISPERSED WHEN USING THE DRY METHOD?

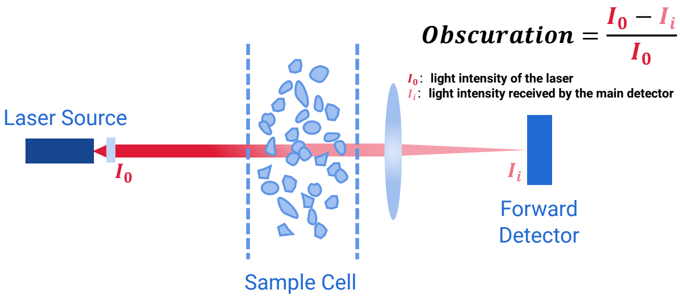


Samples that either dissolve or agglomerate in a wet medium or react with the medium are usually analyzed using the dry method. Samples are dispersed and transported into the measurement zone by pressurized air with the help of a Venturi device. Dispersion of the powdered sample can be achieved by the collision between the sample particles, the collision between the sample particles and the inner wall of the sample inlet tube, and the shearing force that separates sample aggregates. For the samples that are produced and applied in dry conditions, a measurement using the dry method avoids potential factors associated with measurement errors. The dry method is also applicable to small volume samples and valuable samples in the presence of a specific sampling system, allowing the measurement with samples of dozen of milligrams.

# 31

# WHAT IS OBSCURATION?

Obscuration refers to the proportion of light scattered and absorbed by the particles in the measurement zone, whereas transmission is the total of light minus the obscuration. The obscuration indicates the concentration of the suspension. The calculation of the obscuration is shown below. It also influences the calculation of the particle size distribution.



$$Obscuration = \frac{I_0 - I_i}{I_0}$$

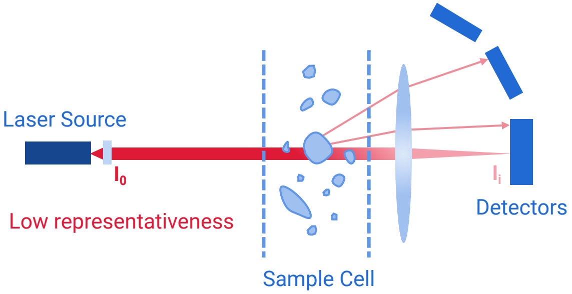
$I_0$: light intensity of the laser

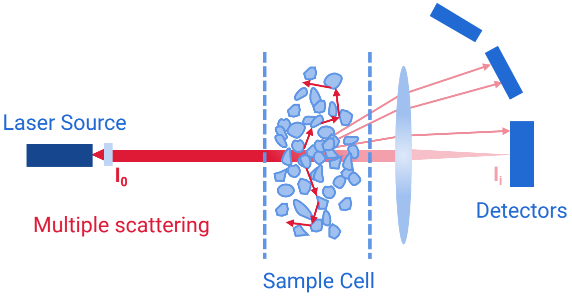
$I_i$: light intensity received by the main detector

## 32

# HOW TO CHOOSE A PROPER OBSCURATION RANGE?

In general, laser diffraction particle size analysis is carried out when the obscuration of the sample in the flow stream is between 10 and 15%. A proper working obscuration lies between having too few particles thus affecting statistical representativeness, and too many particles leading to multiple scattering and hence undersizing of the particle results. In other words, if the obscuration is set between 10 and 15%, multiple scattering can be avoided, and sampling representativeness can be guaranteed. Small particles have a more significant impact on the obscuration. For samples of the same volume, samples with smaller particle size distributions have more particles, leading to higher obscurations and, therefore, the potential for multiple scattering if the obscuration is higher than the upper recommended obscuration limit. According to experience, the proper obscurations for large particles are higher than those for smaller particles during measurement.

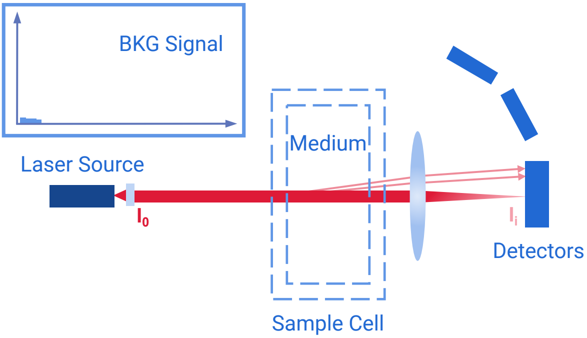




## 33

# WHAT IS BACKGROUND SIGNAL?

The background signal is made up of both optical and electrical signals. The optical noise is composed of an extraneous signal caused by unrelated substances during the measurement. In a laser diffraction particle size analyzer, the background signal is made up of optical interference from the medium, which can be liquid or air, and the cleanliness of the sample cell, e.g., a small number of impurities that may exist on the cell window, creating signals with low intensities on the left side of the signal diagram. These background signals need to be measured prior to the particle size analysis and later removed from the measurement signal, which improves the measurement accuracy.



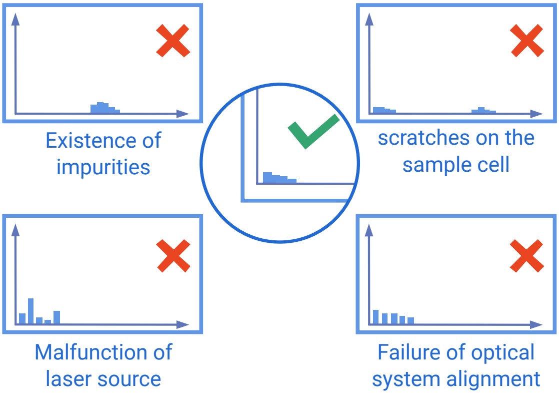
# 34

# WHAT ARE THE FACTORS THAT AFFECT BACKGROUND?

The normal background signal is characterized by the following conditions:

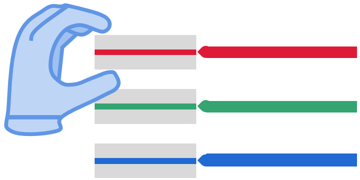
- **The normal background should occupy less than 20 units of channel.**
- **The higher signals should appear on the far left of the signal diagram.**
- **The intensity of the background signal should be quite low.**
- **The signal intensity decreases from left to right sequentially.**
- **There should be no castellation in the signals as this could indicate bad alignment.**

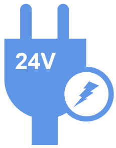
The reasons for abnormal background signals vary. For example, the optical system alignment cannot be carried out; there may be impurities attached to the inner surface of the sample cell or the surface of the lens; the sample cell is foggy due to the temperature difference between the medium and the working environment, causing condensation on the sample cell; the medium is not pure; the working status of the laser source is not ideal; the lens or the sample cell are scratched. Troubleshooting should begin with inspecting the sample cell, then the laser source and lens, and finally, the alignment system.



## 35

# WHAT ARE THE ADVANTAGES AND DISADVANTAGES OF SEMICONDUCTOR LASER?



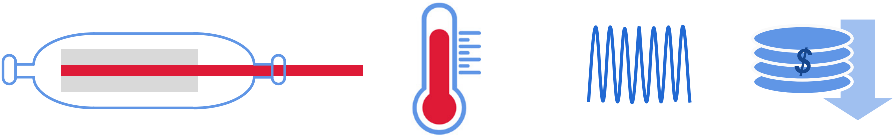


A semiconductor laser, also known as a laser diode (LD), uses electricity as the pump source to emit a monochromatic and coherent laser beam. Laser diodes are usually small in size and weight, making them easy to handle. Powered by a constant current at low voltage, laser diodes are safe to operate and have a low failure rate and energy consumption. Because of these advantages, the laser diode is known as a reliable laser source that is widely applied in various industries. To expand the measurement range of the particle size analyzer and optimize measurement accuracy, different types of laser diodes are available: infrared or red light with a longer wavelength and green or blue light with a shorter wavelength.

In the early days, the performances of laser diodes were limited and impacted by operating temperatures, and the beam divergence was larger. Therefore, the properties of the laser beam emitted, such as directionality, coherence, and monochromaticity, were limited. Laser diode performance significantly improved as the semiconductor industry rapidly developed over the years. Nowadays, the laser diode is widely applied as a laser source in analyzer manufacturing. When using a laser diode as the laser source, a constant current should be provided, and a constant working temperature should be guaranteed, which ensures a stable output power.

# 36

# WHAT ARE THE ADVANTAGES AND DISADVANTAGES OF HELIUM-NEON LASER?



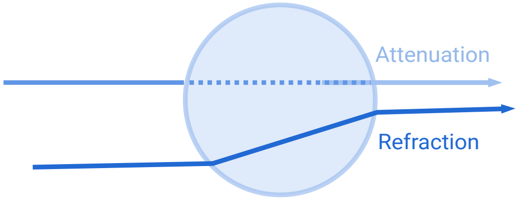
- High Collimation
- Independent of Thermal Fluctuations
- High Stability
- Low Cost

The He-Ne laser is one of the earliest, most mature, and most widely used laser devices. Its gain medium is a gaseous mixture of approximately a 10:1 ratio of helium and neon. In a gaseous mixture, helium atoms are excited from the ground state to higher energy levels, triggering stimulated emission of photons that allows photon emissions in the gain medium. A He-Ne laser device emits a monochromatic laser with a long coherence length, and the wavelength deviation can be controlled to several nanometers. The performance of a He-Ne laser device is independent of thermal fluctuation, and the presence of an optical cavity ensures collimation of the emitted laser beam (divergence angle is only a few milliradians). He-Ne laser is widely applied in industries where good monochromaticity, coherence, and collimation are required, especially in the manufacturing industry for precision measuring devices.

However, a direct high-voltage current is required for the power supply, increasing the device’s failure rate. Because of the consistency of the wavelength, the application area of the He-Ne laser is restricted. The wavelength of the He-Ne laser is larger than those of green and blue lasers, resulting in a low capability of measuring sub-micron particles. The service life of the He-Ne laser is much shorter than that of the semiconductor laser, and its usage is also limited by the large volume of the device.

# 37

# WHAT ARE REFRACTIVE INDEX AND ABSORPTION COEFFICIENT?



$$N = n + ik$$

$N$ : complex refractive index
$n$ : refractive index
$k$ : absorption coefficient

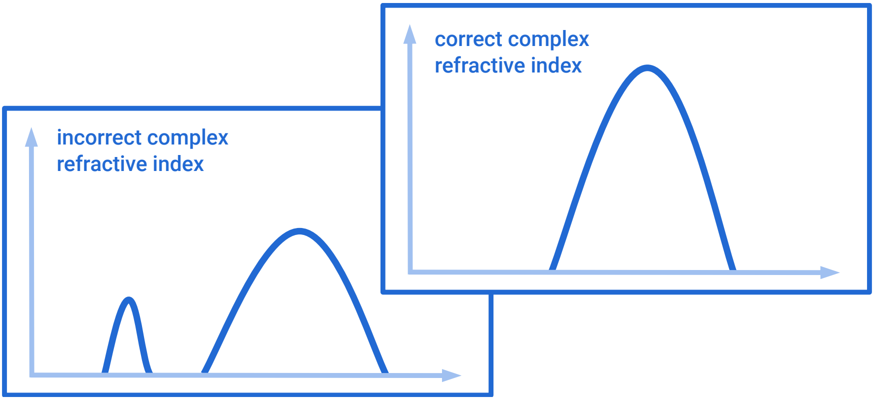
The refractive index describes the degree to which light rays are bent as they pass from one medium to another, such as water into glass. The refractive index can be calculated through the quotient of the light velocity in the two media. When the light travels in a vacuum and then propagates through a material, the refractive index of the material can be calculated by the ratio of the light velocity in a vacuum and in the material. In this situation, the refractive index of the material is also called the absolute refractive index.

The absorption coefficient is a measure of the light beam penetration through a material. In laser diffraction particle size analysis, when a narrow laser beam irradiates the particles, light can be absorbed when it propagates through the particles, resulting in attenuation. The attenuation degree depends on the particle material. Generally speaking, the darker the material is, the larger the absorption coefficient will be. For example, coal would have an absorption coefficient of 1.0, and glass would have a value of 0.0.

For the calculation of particle size distribution, the refractive indices of both the medium and the sample are required in addition to the absorption coefficient of the sample material. These two parameters are used in the complex refractive index, which combines a real part (the refractive index) and an imaginary part (the absorption coefficient), respectively.

# 38

# WHAT IS THE EFFECT OF REFRACTIVE INDEX ON LASER DIFFRACTION PARTICLE SIZE ANALYSIS?



The laser diffraction particle size analysis based on the Mie theory guarantees the accuracy of the measurement result, in which the complex refractive index plays a major role. For small particles whose size approaches the wavelength of the light source interacting with them, complex refractive indices with slight differences can produce completely different measurement results. With the development of new materials, composite materials, and mixed materials, the determination of the complex refractive index is difficult. Therefore, measurement of the complex refractive index prior to particle size analysis ensures the reliability and accuracy of the measurement result.

# 39

# WHAT ARE THE DIFFERENCES BETWEEN DIFFERENT ANALYSIS MODES?

Three analysis modes, namely general mode, multi-peak mode, and R-R mode, are commonly written in the analysis software for the calculation of the particle size distribution.

## GENERAL MODE

The general mode is suitable for the analysis of most of the particles, including mono-dispersed and multi-dispersed samples. The general mode is usually set as default in the analysis software.

## MULTI-PEAK MODE

Multi-peak mode is used when high resolution for analysis is required. The multi-peak mode is more sensitive to the samples whose particle size distributions are similar.

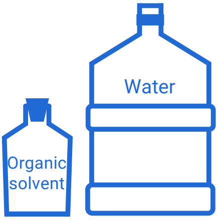
## R-R MODE

The R-R mode is a fixed analysis mode that is only used in some domains, for example, cement manufacturing.

# 40

# WHAT ARE THE COMMONLY USED MEDIA AND REQUIREMENTS?

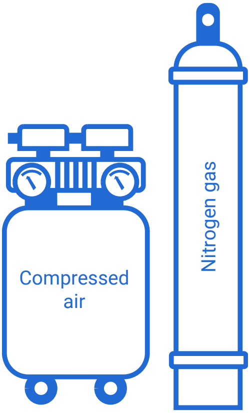
For particle size analysis using the wet method, the most commonly used medium is water, followed by organic solvents, such as absolute ethanol. Compressed nitrogen and air are usually applied as the media for the dry method. The role of the medium is to allow the particles to be distributed evenly during measurement.



For liquid medium, the following conditions should be satisfied:

- The medium should be pure without impurities.
- The sample dispersed in the medium should not be swollen or shrunk; the medium should be inert to the sample.
- The medium should have good miscibility with the particles; in other words, the surface-wetting effect should be obvious.
- In the presence of the medium, the particles are well dispersed.
- The temperature difference between the medium and the working environment should be as small as possible in order to reduce the possibility of condensation on the sample cell.

The requirements for the gaseous medium are that the medium should be pure, dry, non-toxic, odorless, and cost-effective. The gas pressure should be adjustable up to 4bar normally but occasionally up to 6bar. For friable materials, the pressure may need to be controlled down to 0.5bar.



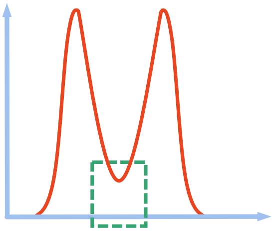
# 41

# WHAT ARE THE REQUIREMENTS OF THE WATER USED FOR LASER DIFFRACTION PARTICLE SIZE ANALYSIS?

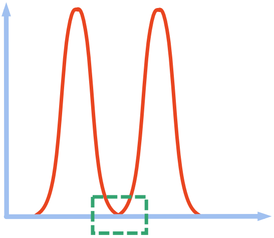
According to the demand of the application, distilled water, purified water, deionized water, clean tap water, and well water can be used as media for particle size measurement. No matter which kind of water is used for measurement, it should be pure without impurities. The water temperature should be as close to room temperature as possible, especially higher than the dew point temperature, in case the sample cell becomes foggy. When using tap water or well water as the medium, it should be placed in a container inside the laboratory for at least 24 hours before use. The reasons are that the impurities will precipitate, and the water temperature will be closer to the laboratory room temperature, especially after a long period of storage.

# 42

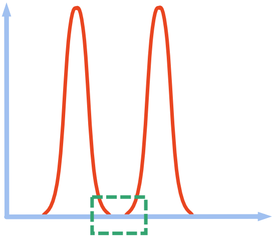
# WHAT IS RESOLUTION AND HOW TO VERIFY IT?



Low Resolution



High Resolution



Higher Resolution

In laser diffraction particle size analysis, resolution is used to evaluate the degree of distinguishing two closely adjacent peaks. Just as repeatability and reproducibility are important factors, resolution is also a significant indicator related to the performance of an instrument. Currently, no certified reference standards are utilized to verify resolution. However, the following method can be implemented for resolution verification: two reference standards whose ratio of nominal values is greater than five are mixed at equal proportions and measured with the particle size analyzer. If two separated peaks are shown in the particle size distribution graph, high resolution can be confirmed, and the instrument is qualified; If the two peaks are separated, and the baseline is clear (the coarse end of the fine sample can be separated from the fine end of the coarse sample), it indicates that the instrument exhibits a higher resolution. Otherwise, the resolution is relatively low.

# 43

# WHAT ARE THE FACTORS THAT AFFECT RESOLUTION?

There are many factors affecting the resolution of a laser diffraction particle size analyzer:

| | |
|---|---|
| **The number of photodetectors** | In the appropriate range, the more the number of photodetectors spaced logarithmically, the higher the resolution. |
| **Inverse algorithm** | A good inverse algorithm leads to a higher resolution. |
| **Distribution mode** | Measurement result based on the multipeak mode has a higher resolution than that based on the single peak model. |
| **Fourier lens** | Fourier lens with less aberration leads to a higher resolution. |

# 44

# WHAT SHOULD WE BE AWARE OF WHEN MEASURING NEODYMIUM MAGNET POWDER?

Aneodymium magnet, also known as NdFeB, NIB, or Neo magnet, is a rare-earth magnet widely used in motor, sensor, computer, and microwave component manufacturing. When measuring the neodymium magnet powder, it has to be taken into account that the sample is magnetic, and the particles are very easy to agglomerate. Neodymium magnet powder should therefore be measured using the dry method, which overcomes the agglomeration phenomenon caused by magnetism through instantaneous dispersion of the agglomerates in the presence of a high-speed airflow. The degree of de-agglomeration is dependent on the air pressure used. Some materials might only need 2bar pressure, whilst others require higher pressure for full dispersion. Thus, the dispersed neodymium magnet sample can be measured accurately.

Since neodymium magnet powder is flammable when exposed to air, some precautions can be taken during the measurement:

**Inert gas or nitrogen gas should be used as the medium.**

**To prevent the neodymium magnet particles from attaching to the inner wall of the tubes, smooth polyethylene tubes need to be applied.**

**A water filtration vacuum cleaner is necessary for collecting measured particles.**

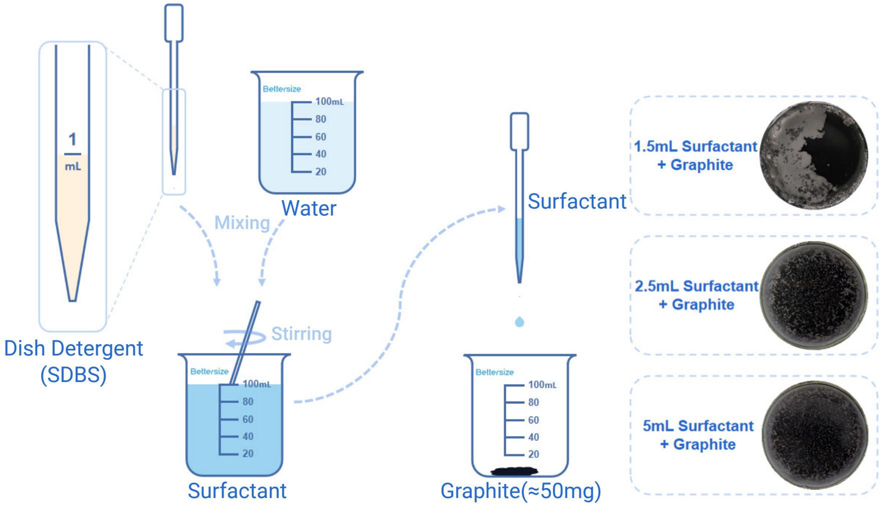
# 45

# WHAT IS A SURFACTANT?

A surfactant is a substance that reduces the surface tension of the medium significantly and hence improves the wetting behaviors or charged states of the particles. A surfactant is commonly used as a dispersing agent to prevent particles from agglomerating. Sodium hexametaphosphate and sodium pyrophosphate are commonly used in laser diffraction particle size analysis for the separation of particles that are easy to agglomerate in water.

The application of a surfactant made of alkylbenzene sulfonate allows a full dispersion of fine graphite particles in water, which facilitate the measurement procedure. Alkylbenzene sulfonate is readily available that can be obtained from soap or detergent.

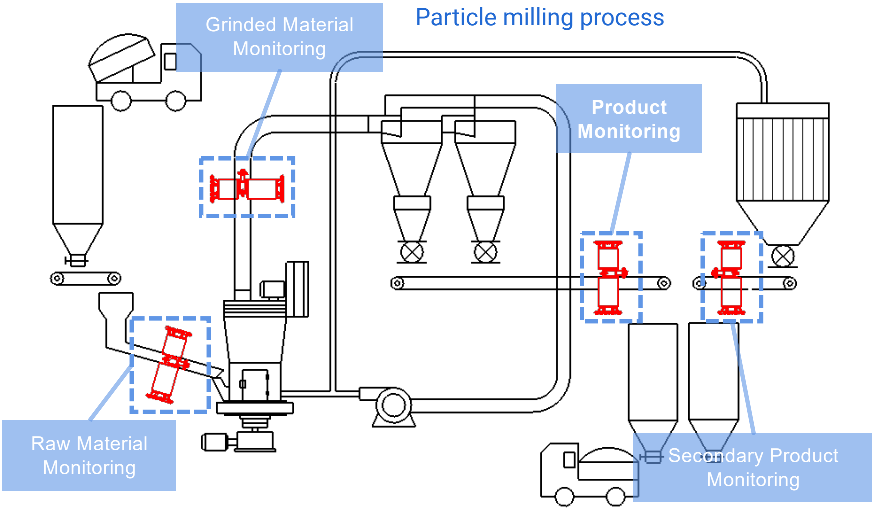
The schematic below shows the preparation of the surfactant with sodium dodecyl benzene sulfonate (SDBS) from dish detergent and the dispersion effect of graphite after introducing different amounts of surfactant.



Sample preparation guide for wet measurement using the laser diffraction particle size analyzer

## 46

# WHAT IS AN ONLINE PARTICLE SIZE ANALYZER?

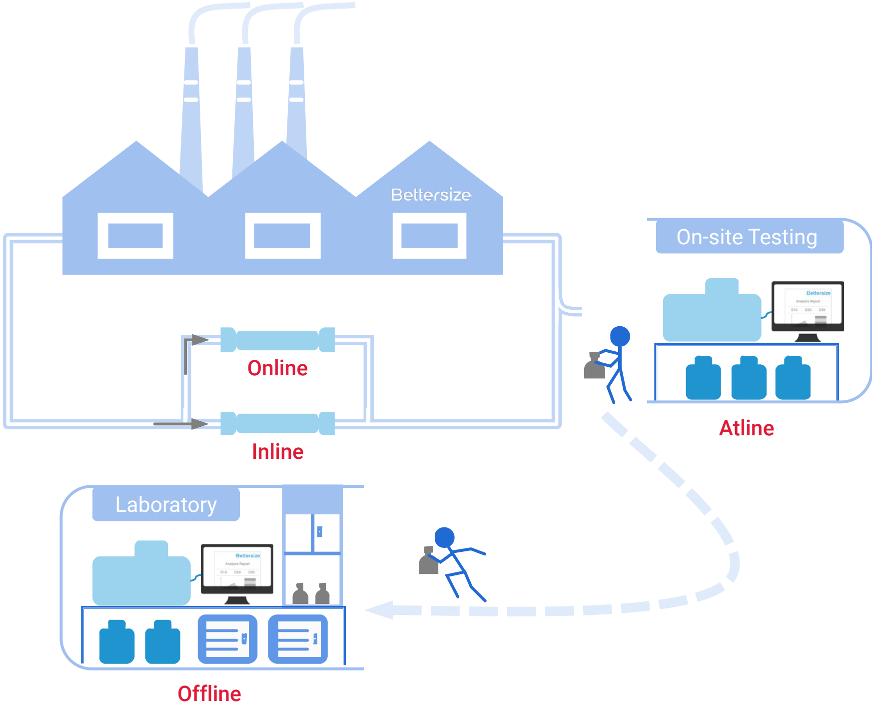


An online particle size analyzer is commonly used as an online detection system integrated into a process loop, providing real-time measurement results that assist in the quality inspection or data monitoring of a process. Combined with the online particle size analyzer, the process control system enables feedback control by abnormal process status, reducing the standard deviation of the product and producing less waste or fewer recycled products. Online particle size analyzer provides continuous real-time measurement, whose measurement interval is customized to optimize process control.

## 47

# WHAT ARE THE DIFFERENCES BETWEEN ONLINE, INLINE, ATLINE, AND OFFLINE ANALYSES?

Laser diffraction particle size analysis can be carried out in different processing locations, which can be classified as online, inline, atline, and offline measurements.



| | |
|---|---|
| **Online** | The sample is extracted from the process line in a statistically representative way and introduced to the measurement zone via a sample inlet tube. The measurement conditions are similar to those of the process line. After the measurement, the sample can be drained as waste or introduced back to the process line. This method is primarily used for dense phase flow where there is a high concentration of particles in air or water. |
| **Inline** | The particle size analyzer is integrated into the process line, where the particle size analysis is implemented under the process conditions. After the measurement, the sample moves forward continuously along the flow. This was the original method for real-time particle size analysis and was only performed on processes or applications with a very low concentration of particles in air or water, called lean phase flow. |
| **Atline** | The sample is extracted from the process line and introduced manually or automatically to a laser diffraction particle size analyzer in its vicinity. The measurement is carried out by process engineers. The sample can be prepared again before the measurement. This can be an ideal method for applications where there are multiple process lines that all need to be monitored in sequence but without feedback control for optimization. |
| **Offline** | The sample is extracted from the process line and measured with a laser diffraction particle size analyzer in the laboratory a distance away from the process line. The sample can be prepared again before the measurement. |

## 48

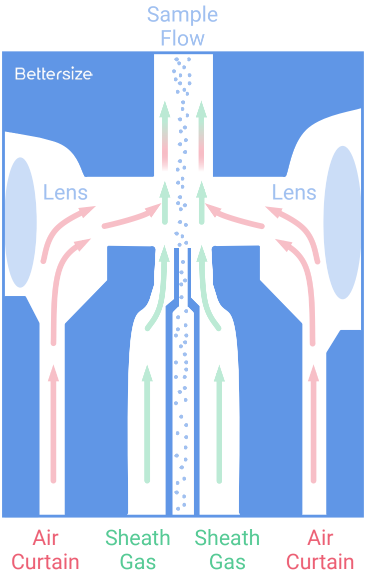
# WHAT ARE THE DIFFERENCES BETWEEN ONLINE PARTICLE SIZE ANALYZERS AND PARTICLE SIZE ANALYZERS IN THE LABORATORY?

An online particle size analyzer is an apparatus integrated into a process loop, while a particle size analyzer in the laboratory is a separate unit with a specific sample circulation and dispersion system. The differences between them are listed below.

| Instruments / Properties | Online particle size analyzer | Laboratory particle size analyzer |
|---|---|---|
| Timeliness | Real-time monitoring and control to ensure the product quality. | Delayed measurement and delayed feedback to operators. |
| Sample Preparation | Automatic sampling, sample dispersion, and sample return without waste. | Manual sampling, sample preparation, and sample dispersion with waste. |
| Automation | Fully automatic unattended measurement. | Manual sample preparation and sample introduction. |
| Measurement Precision | Measurement precision is affected by a bad working environment, so the location of the analyzer is critical to the performance when assessing the optimum installation point. The optical structure also influences measurement precision. | Measurement precision can be higher due to its well-designed optical and analysis system. |

## 49

# HOW TO PROTECT THE LENS IN THE ONLINE PARTICLE SIZE ANALYZER FROM CONTAMINATION WHEN USING THE DRY METHOD?



When using the dry method for introducing samples in online particle size analysis, the protection of the lenses from contamination caused by sample particles or dust is essential. The protection is achieved by two different methods, namely, the utilization of a sheath gas and a dual air curtain.

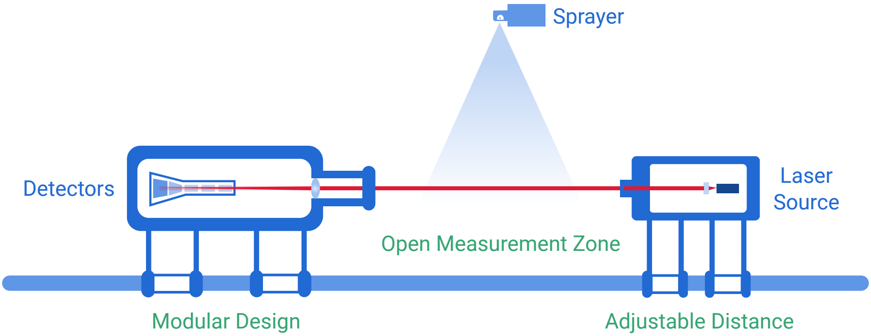
The sheath gas is emitted through the peripheral channel of the sample nozzle, which provides stretching and focusing force on the sample jet, restricting the movement of particles to the optical lenses.

The effect of the dual air curtain system is to generate positive stress zones. The application of the air curtain ensures that the lenses are fully protected and that the sample accurately passes the measurement zone. The result is, therefore, more precise, with little to no contamination of the lenses by sample particles or dust.

# 50

# WHAT IS A SPRAY PARTICLE SIZE ANALYZER?

A spray particle size analyzer is a laser diffraction particle size analyzer used for the measurement of the suspended particles, for example, spray droplets. The spray particle size analyzer is characterized by its open measurement zone, modular design that increases the flexibility of the measurement, corrosion-resistant surface that is suitable for various applications, and durable structure that allows usage in a harsh working environment.



# 51

# WHAT IS THE SPRAY PARTICLE SIZE ANALYZER TYPICALLY USED FOR?

The particle size distribution of a spray is an important parameter in the research of pharmaceutical spray, pesticide spray, and engine combustion efficiency, which is helpful in optimizing the atomizer design, spray pressure control, and evaluation of the spray effect. Here are some examples to demonstrate the importance of the particle size distribution of the spray droplet. The size of the pesticide droplets should be between 150 and 300μm for optimal bioavailability. The droplet size of the inhaled drug should be less than 10um to be effectively inhaled into the lungs and exerts its medicinal effect. The diesel droplets in diesel engines should be less than 100um to improve combustion efficiency and reduce carbon emissions.

The spray particle size analyzer is typically used in water sprays, fuel sprays, beauty sprays, atmospheric monitoring, inhaled drugs, agrochemicals, routine atomizers, fuel injection systems, and spray granulation.

PART

C

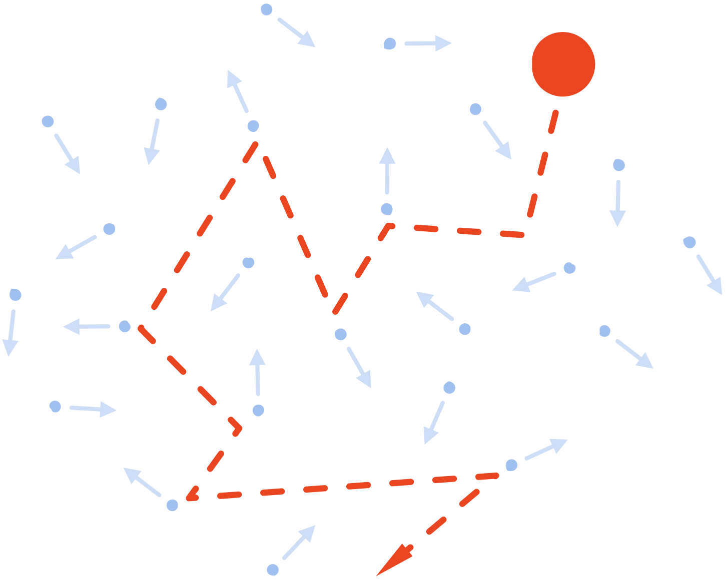
# Nanoparticle Size and Zeta Potential Analysis

## 52

## WHAT IS BROWNIAN MOTION?

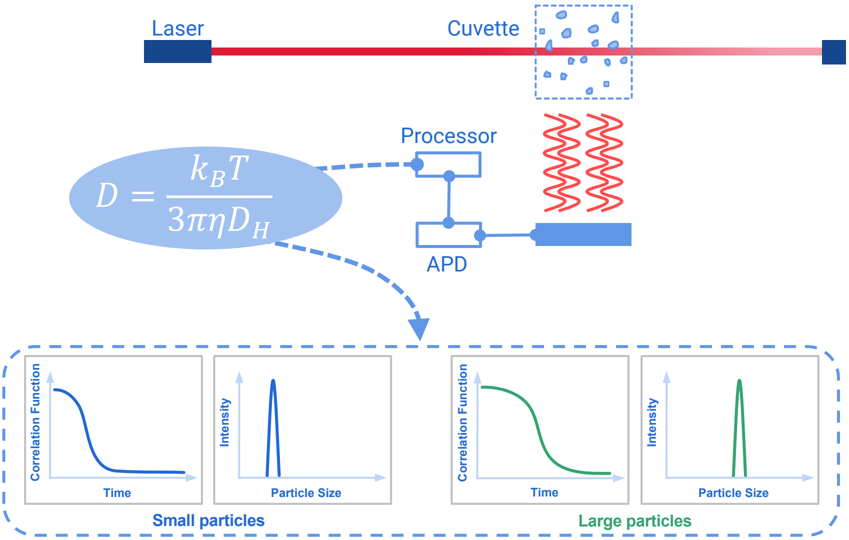
Brownian motion refers to the random motion of suspended particles in a liquid or gaseous fluid due to the unpredictable collision between particles and solvent molecules caused by thermal fluctuations.

Brownian motion is often demonstrated through the movements of small particles such as pollen that can be observed in the liquid under the sunlight. An important feature of Brownian motion is that small particles move fast while large particles move slowly. Therefore, the diffusion behaviors of particles can be used to calculate the particle size.



# 53

# WHAT IS DYNAMIC LIGHT SCATTERING (DLS)?

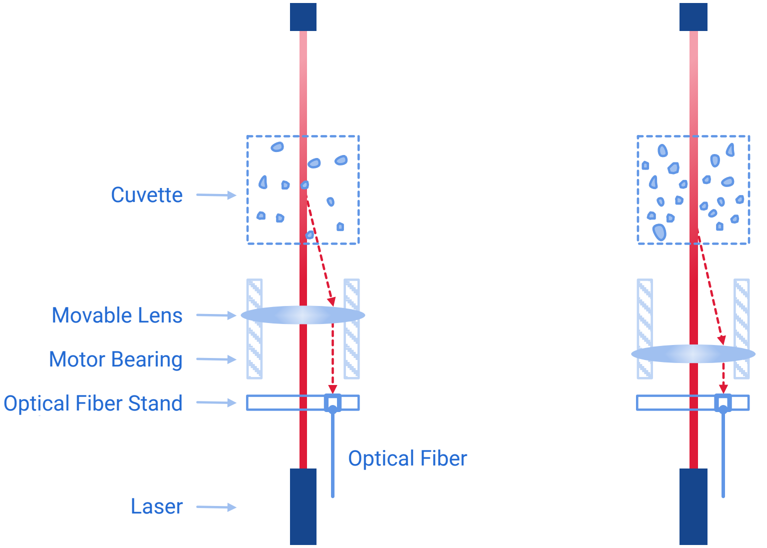


Dynamic light scattering (DLS), also known as photon correlation spectroscopy (PCS) or quasi-elastic light scattering (QELS), is a technique utilized to characterize the particle size of samples on the nanoscale.

In a DLS setup, uniformly dispersed nanoparticles in a dedicated cuvette are illuminated by a coherent laser beam. The scattering intensity fluctuates over time due to the continuous random walk of particles undergoing Brownian motion. The photon detector determines the number of photons in scattered light, and the correlator computes the auto-correlation function. By applying a mathematical algorithm to the correlation function, the particle size distribution of the sample is thereby obtained.

# 54

# WHAT IS DLS BACKSCATTERING TECHNIQUE?



Diluted or weakly scattering samples

Concentrated or strongly scattering samples

The traditional dynamic light scattering analysis used a 90° detector which received scattered light signals emanating from diluted samples. The emphasis was on dilution as more concentrated samples would have strong particle interactions as well as create multiple scattering rendering the technique unusable. Therefore, in most applications, laborious sample preparation before analysis was necessary, resulting in inconvenient operation and potential destruction of colloidal stability.

A DLS backscattering technique was made available in recent years that can detect scattered signals at a large angle, usually between 160° and 175°.

The DLS backscattering technology enables intelligent adjustment for the optimal detection position. By moving the lens, the detection point can be set from the center to the cuvette wall, allowing the detection of different types and concentrations of samples to a great extent.

The advantages of DLS backscattering technology over the traditional 90° setup include higher detection sensitivity for samples with very low concentrations. Besides, it is now possible to make intelligent adjustments of optical elements to avoid multiple scattering and, therefore, the capability of analyzing concentrated samples, in addition to limiting the interference caused by dust.

## 55

# WHAT ARE Z-AVERAGE AND POLYDISPERSITY INDEX (PDI)?

Z-average is the intensity-weighted mean hydrodynamic size of the ensemble collection of particles measured by DLS.

The Z-average is a hydrodynamic size that is determined by an ISO method, i.e., the cumulants method. For the cumulants method, the correlation function is solved by the following expression:

$$\ln G_1(\tau) = \ln A - \bar{\Gamma}\cdot\tau + \left(\frac{\mu_2}{2!}\right)\cdot\tau^2 - \left(\frac{\mu_3}{3!}\right)\cdot\tau^3$$

where A is the intercept of the correlation function, which represents the signal-noise ratio and $\bar{\Gamma}$ is the average decay rate, which can be substituted into the equation $\bar{\Gamma}=q^2\bar{D}$ to calculate the average diffusion coefficient $\bar{D}$ of all particles. $\bar{D}$ is then put into the Stokes-Einstein equation to produce the average particle size $D_H$, namely the Z-average mean hydrodynamic diameter.

$$\bar{D} = \frac{k_B T}{3\pi\eta\bar{D}_H}$$

For dynamic light scattering, the Z-average is used to present the average particle size, which is the intensity-weighted average size. PDI (Polydispersity Index), defined by PDI=$\mu_2/\bar{\Gamma}^2$ , which indicates the width of the distribution.

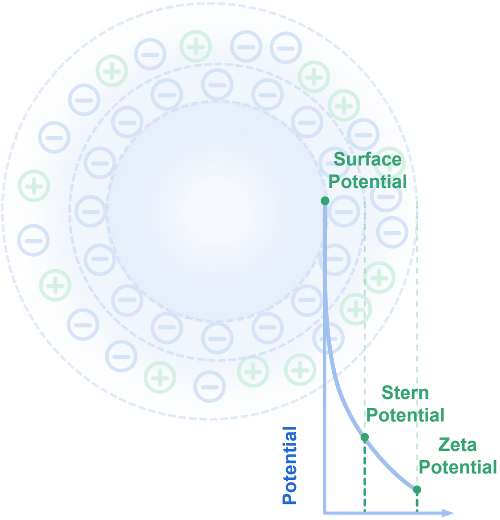
As a rule of thumb, the following PDIs are shown for different samples.

| Sample Type | PDI |
|---|---|
| Mono-dispersed Sample | 0 - 0.05 |
| Narrow-dispersed Sample | 0.05 – 0.08 |
| Moderate-dispersed Sample | 0.08 – 0.7 |
| Wide-dispersed Sample | > 0.7 |

DLS is a technique suitable for samples whose particle size distributions are relatively narrow. For samples with PDI greater than 0.7, DLS measurement might not be applicable.

# 56

# WHAT IS ZETA POTENTIAL?



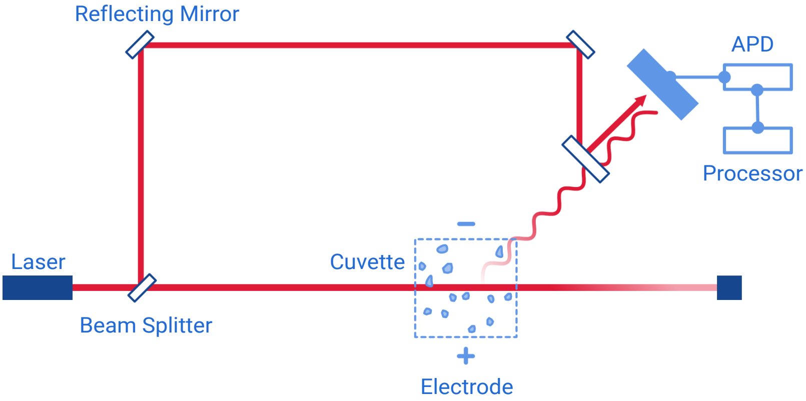
Particles carry charges on their surfaces in aqueous systems, surrounded by counter-ions that form a firm inner layer called the Stern plane and an outer shear plane. The electrostatic potential reaches the maximum at the particle surface, which is called the surface potential. The Stern plane is located at the electrical double layer's inner region, where ions are strongly bonded. The electrostatic potential at the Stern plane is called the Stern potential. The shear plane describes the circle where the number of ions, both positive and negative, are equal, and thus an electrochemical equilibrium is established at the particle-liquid interface. The electrostatic potential in this region is called zeta potential. The zeta potential can be regarded as the potential difference between the dispersion medium and the stationary layer of the fluid attached to the particle layer.

A colloidal system with a higher zeta potential tends to be more stable and less likely to form aggregates, while lower zeta potentials lead to flocculation and coagulation of the particles due to Van der Waals forces. Measurement of zeta potential is an advantageous method to evaluate the stability of the colloidal system, which is widely applied in many fields, including ceramics, paints, inks, pharmaceuticals, water treatment, food, and beverage emulsions.

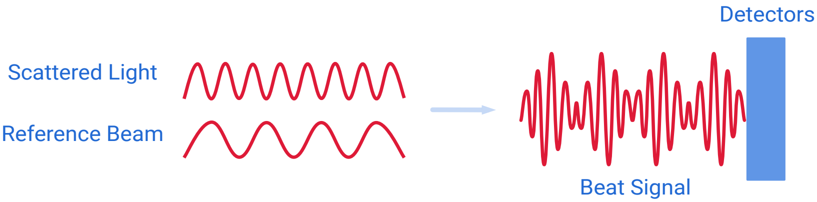
## 57

# WHAT IS ELECTROPHORETIC LIGHT SCATTERING (ELS)?

Electrophoretic light scattering (ELS) is a light scattering technique used to measure the zeta potential of suspended particles in solution based on Doppler shifts in scattered light.



The laser beam is split through the beam splitter into an incident beam and a reference beam. The sample particles are illuminated by the incident beam, while the reference beam passes through the PZT, which provides the piezoelectric effect, and reflecting mirror and combines with the scattered light detected at the forward angle of 12°. Charged particles experience electrophoresis when subjected to an electric field. As a consequence, the frequency of the scattered light shifts compared to that of the incident light due to the Doppler effect. Moreover, the magnitude of the frequency shift correlates to the electrophoretic velocity.

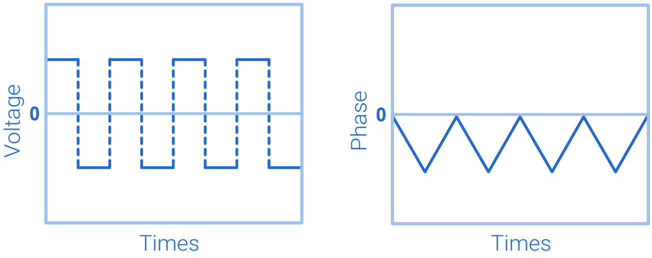


A detector is set forward to detect the scattering signals, with which the frequency shift and the electrophoretic mobility are obtained. By using Henry's equation, the zeta potential and its distribution can be obtained based on electrophoretic mobility.

## 58

# WHAT IS PHASE ANALYSIS LIGHT SCATTERING (PALS)?

Laser Doppler electrophoresis (LDE) is commonly used in electrophoretic light scattering (ELS) to measure the electrophoretic mobility and zeta potential. Recently, a more sensitive technique, phase analysis light scattering (PALS), has been developed to analyze the beat signals by measuring the phase shift instead of the frequency shift. PALS can provide more accurate results, especially for low electrophoretic mobility samples. An alternating voltage is applied to the sample to change the electrophoresis directions of charged particles in an ELS measurement. PALS is utilized to detect the change in phases of scattered light with respect to time. The slope of the phase plot is proportional to the frequency shift $\Delta f$.



The relationship between electrophoretic mobility μ and frequency shift $\Delta f$ is described as follows:

$$\mu = \frac{\lambda}{n \sin\theta} \cdot \frac{1}{E} \cdot \Delta f$$

Henry's equation relates to the electrophoretic mobility μ and zeta potential ζ :

$$\mu = \frac{2\varepsilon_r \varepsilon_0 \zeta}{3\eta} f(\kappa\alpha)$$

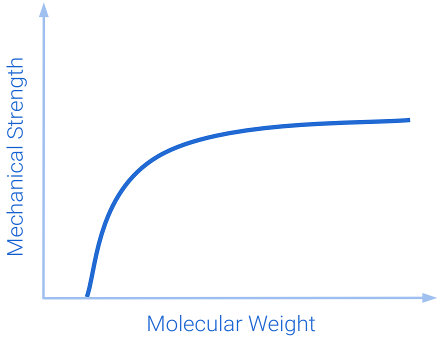
where $\varepsilon_0$ is the solvent dielectric constant in a vacuum, $\varepsilon_r$ is the relative dielectric constant, $\eta$ is the solvent viscosity, $f(\kappa\alpha)$ is the Henry function, $\kappa$ is the reciprocal Debye length, $\alpha$ is the particle radius, and $\kappa\alpha$ refers to the ratio between the thickness of the electrical double layer and the particle radius.

## 59

# WHAT IS MOLECULAR WEIGHT AND WHY IS IT SO IMPORTANT?

Molecular weight (Mw) is the weight of a molecule of a substance. A macromolecule is a very large molecule with a high molecular weight ranging from the thousands to millions. Most common macromolecules include natural polymers, proteins, and polypeptides.

Researchers give close and thoughtful attention to the measurement of molecular weight, which significantly impacts the product performances of polymers and other macromolecules. The following figure shows an example of the dependence of mechanical strength on the molecular weight of polymers.



Another example of molecular weight dependence is polymeric biomaterials. When preparing drug release systems, one must choose polymers with a low molecular weight to obtain microspheres with fast drug release. For microspheres with delayed release, polymers with a larger molecular weight are required.

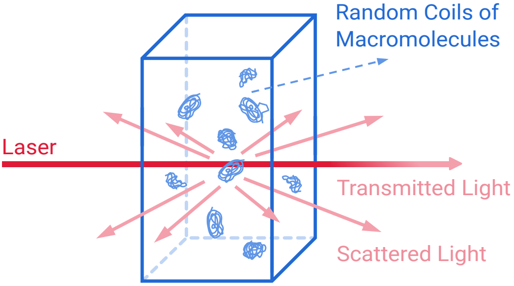
## 60

# WHAT IS STATIC LIGHT SCATTERING (SLS)?

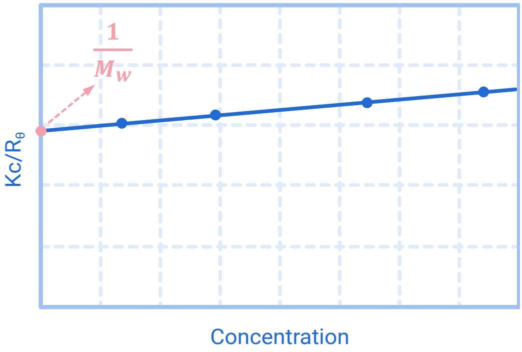
Static light scattering (SLS) is a technique that measures the average scattering intensities to calculate the weight-average molecular weight of particles in a liquid solution. When a laser beam irradiates macromolecules in solution, the macromolecules scatter light in all directions.

The relationship between the scattering intensity and the molecular weight of a macromolecule is described by the simplified Rayleigh equation:

$$\frac{Kc}{R_\theta} = \frac{1}{M_w} + 2A_2c$$



The instrument collects scattering intensities of macromolecules in solutions at different sample concentrations. The Debye plot is then constructed by plotting and linearly fitting the $Kc/R_\theta$ values versus the concentrations. The slope of the linear fit is used to calculate the second virial coefficient $A_2$. And the Y-axis intercept of the linear fitting yields the reciprocal of molecular weight.



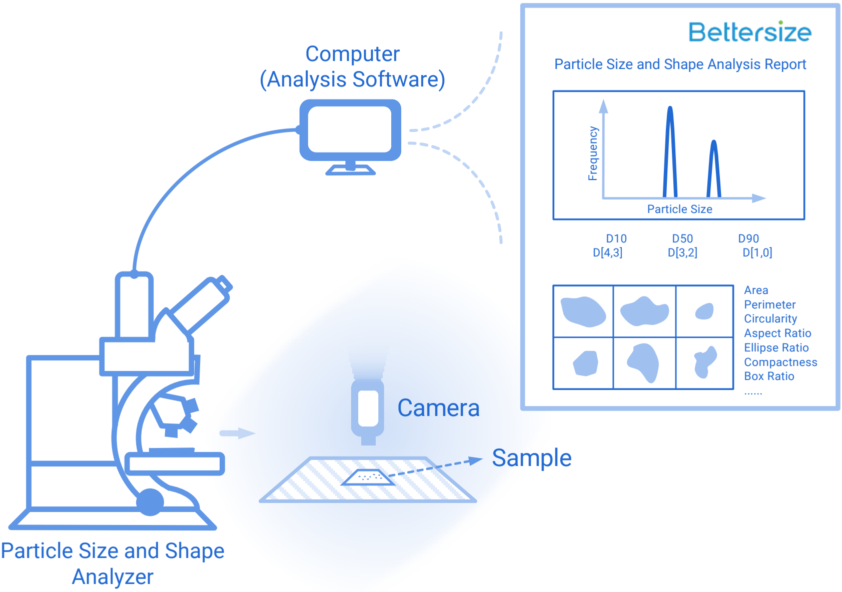
PART

D

# Static and Dynamic Image Analysis

## 61

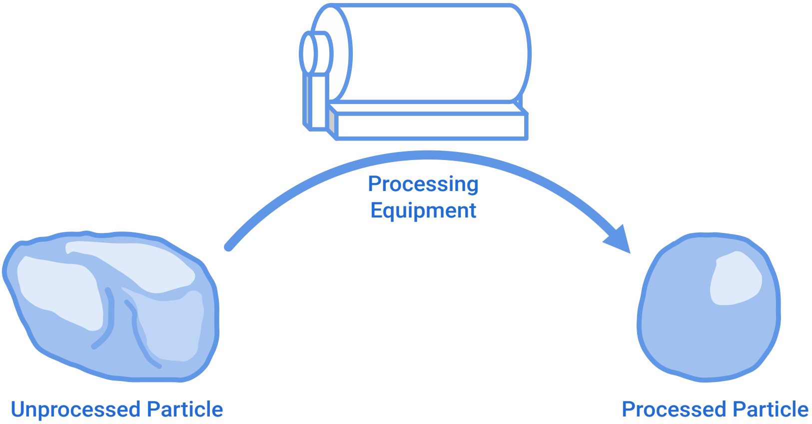
# WHAT IS PARTICLE IMAGE ANALYSIS?



Particle image analysis is a technique for determining particle size and particle shape parameters based on the captured images obtained by using specific cameras. The critical step in particle image analysis is processing the captured images, which provides two-dimensional pictures from the pixels counted. The data acquired is used to provide image binarization, enhancement of picture quality, and particle identification. The processed picture is then analyzed automatically by a computer with the help of algorithms to yield the particle size distribution and related particle shape parameters of the observed sample.

# 62

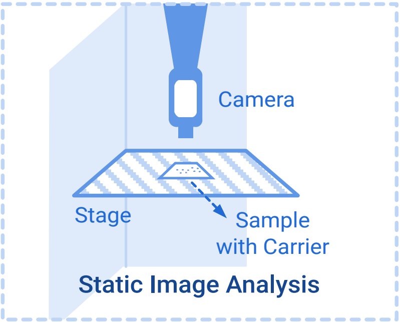
# WHY IS PARTICLE SHAPE SO IMPORTANT?



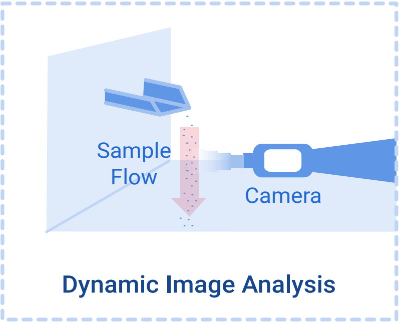
Particle shape is a factor that significantly affects the processing and performance of products. Therefore, it is critical to measure this parameter. Particle shape can be modified based on the desired performance in scientific and industrial research, improving product quality and performance. Here are some examples. In the manufacturing of inks and cosmetics, plate-shaped particles are prioritized for better reflection and adsorption; Granular particles are desired in rubber fillers, which are beneficial to abrasion resistance and strength; Long particles are required in plastic filters to improve the impact strength; Smooth spherical particles are commonly used to produce explosive, thus improving the stability; Polygonal shaped particles are necessary for abrasive to improve grinding efficiency.

## 63

# WHAT IS STATIC AND DYNAMIC IMAGE ANALYSIS?



**Static Image Analysis**



**Dynamic Image Analysis**

Static and dynamic image analysis is a method utilized to determine particle size distribution and particle shape parameters according to their particle projections onto the acquired picture processed by the analysis software. The difference between static and dynamic image analyses is the relative motion between the sample and the lens of the camera. In static image analysis, the sample is statically located on the carrier, above which a camera is installed for image capturing. For dynamic image analysis, particles pass through the focal plane of the camera to be captured clearly. For both image analyses, cameras with different magnifications are available for image acquisition of particles with different particle size ranges.

## 64

# WHAT ARE THE ADVANTAGES OF STATIC IMAGE ANALYSIS?

In static image analysis, particles are fixed using a microscape slide, and the stage that holds the slide, which can be modified manually or programmed automatically to search for an optimal location so that the particles can be clearly captured. Static image analysis is characterized by its cost-efficiency and clear image. Besides, more particles can be clearly captured at high magnification compared to other techniques. The reason is that the particles are all fixed on the focal plane, which avoids the consideration of the depth of field.

# 65

# WHAT ARE THE ADVANTAGES OF DYNAMIC IMAGE ANALYSIS?

Dynamic image analysis is an efficient method to analyze particle size and shape, which provides rapid results due to the particles' short exposure time coupled with a high-speed data acquisition system. Making accurate measurements is easy because of its simple operation using an automatic analysis system that provides excellent repeatability based on the analysis of many particles in a very short time. In dynamic image analysis, samples are automatically transported to the measurement zone with the help of an automatic feeding system which is achieved by a vibrating feeder with air dispersal for the dry method. When using the wet system, an automatic liquid circulation system is in operation to disperse and transport the particles in liquid to the measuring zone, where they are subsequently analyzed.

## 66

# WHAT IS IMAGE BINARIZATION?

Image binarization is a process that converts the color or grayscale original captured images into digital binary images consisting of small black and white pixels. Image binarization is the basis of image analysis and is the key step in image processing used for particle identification.

The measurement system provides two image binarization methods called automatic image binarization and double-threshold image binarization. Automatic image binarization is a binarization threshold determination method with which the binarization threshold is formulated automatically based on the original image. In addition to the automatic image binarization, users are allowed to use double-threshold image binarization to highlight the pixels within a specific range of pixel values. In double-threshold image binarization, only pixels whose pixel values are larger than or equal to the minimum threshold and lower than the maximal threshold will be identified as bright pixels. Other pixels will be recognized as dark pixels. An example is shown below. The binarization process varies with modified thresholds.

Pixel value of “Bettersize” = 195
Pixel value of “Instruments” = 125
Pixel value of black = 0
Pixel value of white = 225

Maximal threshold = 200
Minimum threshold = 190

Maximal threshold = 130
Minimum threshold = 120

# 67

# WHAT IS CALIBRATION COEFFICIENT?

The calibration coefficient refers to the actual length of one of the sides of a single pixel in the image. The area of the pixel can be calculated easily because the pixels are all squares. Particle size and various particle shape parameters are related to length measurement and area calculation. Therefore, the calibration coefficient is of vital importance to particle size and shape analysis.

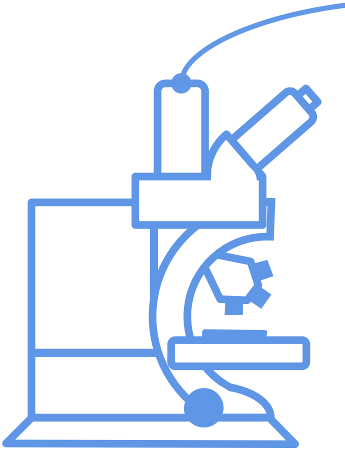
The general calibration procedure is described below:

- Users need to photograph an object of known length (For example, a ruler) to obtain an image containing the complete object of known length.

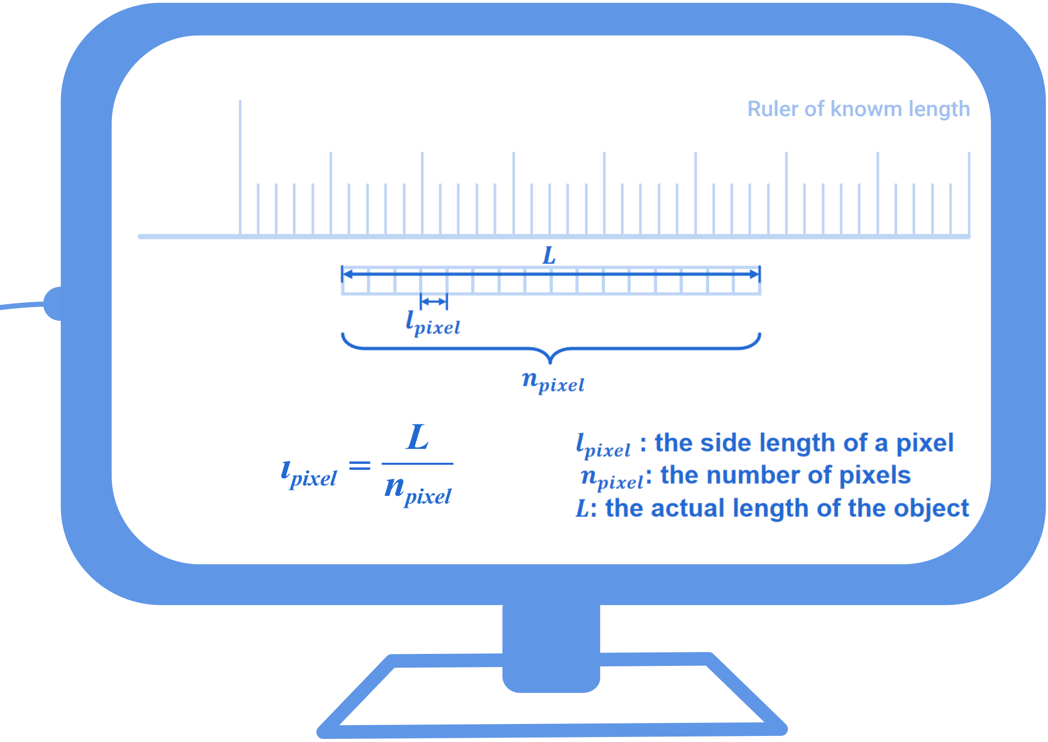
- Users need to draw a line segment for the known length and highlight it. The width of the line segment is one pixel.

- The number of pixels on the line segment will be calculated automatically by the software.

- Users need to input the actual length of the line segment to the software. The actual length of one side of a pixel can therefore be obtained; that is, the calibration coefficient.



**Particle Size and Shape Analyzer**



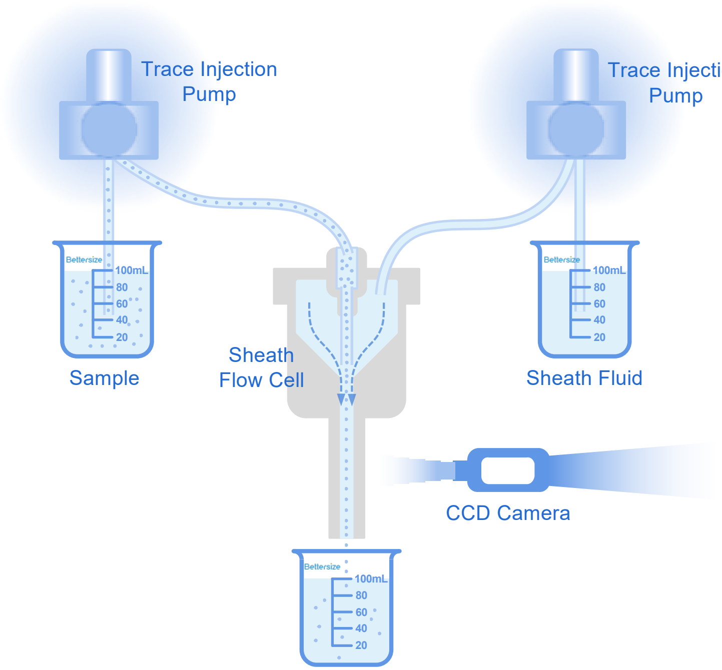
$$l_{pixel} = \frac{L}{n_{pixel}}$$

where $l_{pixel}$ is the actual length of a pixel, $L$ is the actual length of the object and $n_{pixel}$ is the number of pixels.

From the calibration procedure, it is known that the obtained calibration coefficient is only applicable to the photos taken by the same camera under the same magnification. Any operations such as photo distortion, image quality compression, and camera replacement will change the calibration coefficient.

## 68

# WHY IS SHEATH FLUID APPLIED IN DYNAMIC IMAGE ANALYSIS WHEN USING THE WET METHOD?

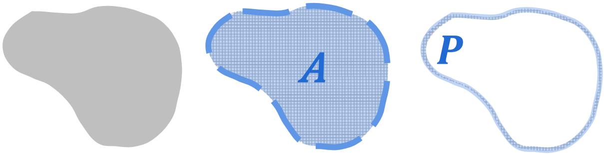


In dynamic image analysis, when using the wet method, sheath fluid is used to provide a stretching and focusing force on the sample flow. In the presence of the sheath fluid, the sample flow is “locked” on the focal plane of the camera, with which the sample particles can be clearly captured, ensuring the accuracy of particle size and shape analysis. Suppose that the sheath fluid is not utilized, blurred particle images will be caused due to the defocussing of the sample flow, leading to an incorrect particle identification and analysis result.

## 69

# HOW TO CALCULATE THE PROJECTED AREA AND THE CONTOUR OF A PARTICLE?

Area, in this instance, refers to the projected area of a particle. The projected area of a particle can be calculated by multiplying the number of pixels by the area of a pixel. The area of a pixel can be calculated with the help of the calibration coefficient. The perimeter of a particle projection refers to the total length of its projected contour. The perimeter can be calculated through contour representation using curve fitting with the help of an algorithm.

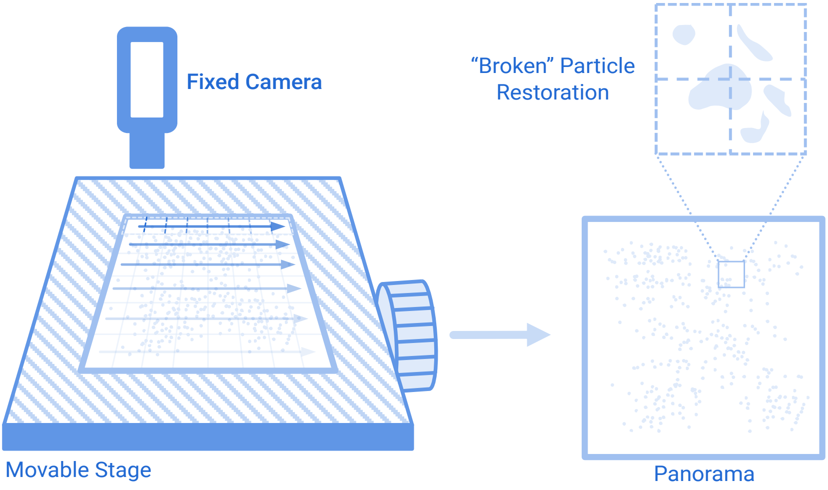


$A$: projected area of the particle

$P$: the length of the projected contour

# 70

# WHAT IS PANORAMIC MODE?



Panoramic mode is an analysis mode used to stitch individual images into a panorama, in which the movement of the stage is controlled by software. Before analysis, users are allowed to set the scanning area using the analysis software. Compared to the common static image analysis, the stage moves step by step, and individual images are taken by the CCD camera every single step until the whole chosen area is scanned. The acquired individual images are then stitched into a panorama with the help of algorithms. The main advantage of the panoramic mode is that the accurate counting and analysis of the whole sample on the carrier, including the edge particles, can be carried out. The accuracy of the counting of particle numbers is significant in the automotive and pharmaceutical industries. Besides, the measurement for oversized particles is also accessible by this technique.

## 71

# HOW TO CHARACTERIZE PARTICLES WITH IMAGE ANALYSIS?

Particles can be characterized using various related particle shape parameters, such as:

| | | |
|---|---|---|
| Area-equivalent diameter | Perimeter-equivalent diameter | Circularity |
| Ellipse ratio | Aspect ratio | Compactness |
| Box ratio | Straightness | Martin diameter |
| Convexity | Concavity | Irregularity |

They are vital parameters that are beneficial to quality inspection and scientific research.

A guide to particle size and shape parameters by image analysis ☝

## 72

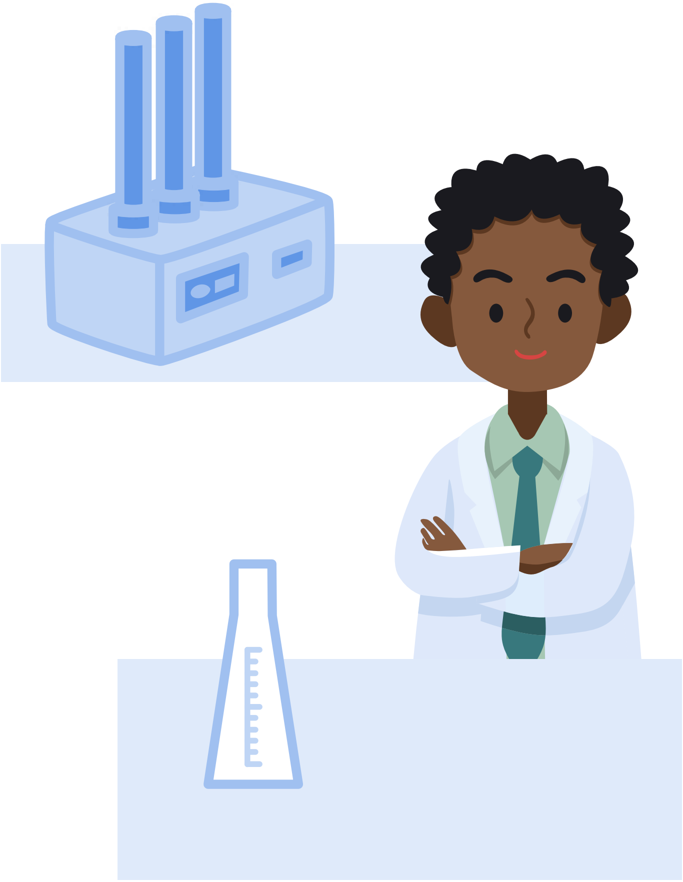
## WHAT ARE THE DIFFERENCES BETWEEN MEASUREMENT RESULTS OBTAINED BY IMAGE ANALYSIS AND LASER DIFFRACTION PARTICLE SIZE ANALYSIS?

The measurement result obtained by the laser diffraction particle size analysis is based on the scattered signals of particles captured onto detector arrays. An inverse algorithm is carried out to calculate a theoretical signal intensity distribution, which is compared to the measured one. An index called residual is used to describe the difference between the theoretical and measured signal intensity distributions. The theoretical signal intensity distribution with the smallest residual value is considered the measurement result. In contrast, the measurement result obtained by image analysis is calculated based on the captured images of real particle projections instead of algorithms. The laser diffraction particle size analysis yields signal-equivalent diameters, which is suitable for the analysis of small micro-level particles, while image analysis provides area-equivalent diameters, measurement of oversized particles is therefore possible.

PART

E

# *Powder Characterization*



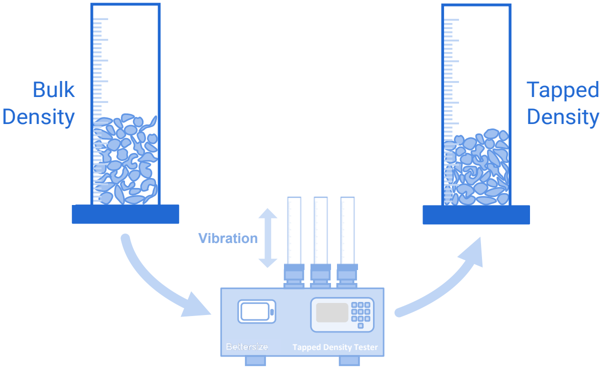
# 73

# WHAT IS POWDER CHARACTERIZATION?

Powder characterization is the term that covers the measurement of powder characteristics using various techniques or apparatuses. Powders are composed of solid particles, which can be defined through various parameters, including particle size and shape. However, the powder characteristics are not simply defined by the attributes of the characteristics of particles because there are not only individual particles to be concerned with, but also noting that powders also contain air between the particles, and those particles may also contain pores that contribute to the powder characteristics, including flowability, compressibility, and density. Powder characterization is significant in many industries, of which the most important and relevant are the manufacturing of pharmaceuticals, additive materials, and paints since the performance of their final products is greatly impacted by the particle properties.

# 74

# WHAT ARE BULK AND TAPPED DENSITIES AND HOW TO MEASURE THEM?



Scan to learn more

Bulk density refers to the mass of the bulk powder divided by the bulk volume. Bulk density can be calculated through the formula below:

$$\rho_B = \frac{M}{V_B}$$

where $\rho_B$ is the bulk density, $M$ is mass in grams, and $V_B$ is the bulk volume in milliliters. The bulk volume includes the volumes of the particle solid, interparticle voids, and pores in the particles.

The bulk density is greatly influenced by the spatial arrangement of powder particles. The tapped density value can be determined by mechanically tapping a container producing a regular vibration that rearranges the powder particles, thus reducing the volume of the interparticle voids. Hence with fewer voids, the tapped density is greater than the bulk density.

The calculation of the tapped density can be achieved using a tapped density tester after a regular vibration within a particular time frame.

## 75

# WHAT ARE THE COMPRESSIBILITY AND THE HAUSNER RATIO?

The compressibility and the Hausner ratio can be calculated using the bulk density and the tapped density, which are utilized to indicate the flowability of powder material.

The calculation of the compressibility:

$$Compressibility = \frac{\rho_T - \rho_B}{\rho_T} \times 100$$

The calculation of the Hausner ratio:

$$Hausner\ Ratio = \frac{\rho_T}{\rho_B}$$

where $\rho_T$ is the tapped density, and $\rho_B$ is the bulk density.

The flowability of a powder can be characterized based on the compressibility or the Hausner ratio. A lower compressibility or a lower Hausner ratio of a material indicates better flow properties. A compressibility of <10 or a Hausner ratio of <1.11 is considered an "excellent" flow, whereas a compressibility of >38 or a Hausner ratio of >1.60 is considered a "very poor" flow.

| Flowability | Compressibility | Hausner Ratio |
|---|---|---|
| Excellent | <10 | 1.00-1.11 |
| Good | 11-15 | 1.12-1.18 |
| Fair | 16-20 | 1.19-1.25 |
| Passable | 21-25 | 1.26-1.34 |
| Poor | 26-31 | 1.35-1.45 |
| Very Poor | 32-37 | 1.46-1.59 |
| Very Very Poor | >38 | >1.60 |

The flowability of a powder material can be evaluated by integrating the compressibility, the Hausner ratio, and the angle of repose. It is also possible to predict the flowability of the material through individual parameters.

# 76

# WHAT ARE TRUE AND SKELETAL DENSITIES OF A POWDER AND HOW TO MEASURE THEM?

Skeletal density and true density are parameters related to the surface and interior structures of porous particles. Skeletal density is the ratio of the mass of the porous particles to the volume of the particles that excludes open pores. The true density is also known as absolute density, in which open and closed pores are not considered in the calculation of the volume of the porous particles. The measurement of skeletal density can be implemented using a gas pycnometer or a liquid pycnometer based on the volume-pressure relationship of Boyle's Law. Therefore, the density obtained with the help of a pycnometer is also called pycnometric density. The skeletal density equals the true density only in the case that the porous particles do not contain closed pores or sealed pores.

Boyle’s law demonstrates the relation concerning the compression and expansion of an ideal gas at a constant temperature, which states that the pressure of a given quantity of gas varies inversely with its volume, as shown below:

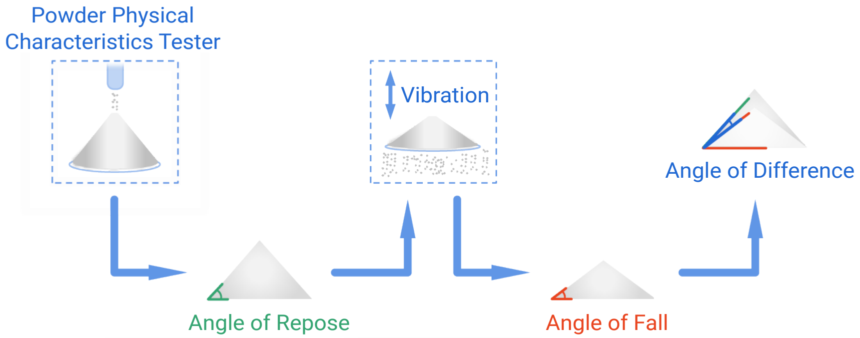
$$P = \frac{1}{V}$$

where $P$ is the gas pressure and $V$ is the gas volume.

Real gases obey Boyle’s law at sufficiently low pressures, although the product $PV$ decreases slightly at higher pressures, where the gas begins to depart from ideal behavior.

## 77

# WHAT ARE THE ANGLE OF REPOSE, THE ANGLE OF FALL, AND THE ANGLE OF DIFFERENCE?



The angle of repose refers to the angle between the sloping side of a powder cone and the horizontal plane after the powder particles freely fall onto a horizontal plane under specific conditions. The angle of repose is a significant indicator of powder flowability. The smaller the angle of repose is, the better the flowability will be. It is generally assumed that the angle of friction and the angle of repose are approximately equal. For some materials, such as sorghum, the magnitudes of the two angles can be different.

Scan to learn more

The angle of fall is the decreased angle obtained after the angle of repose has been measured, and the powder pile is subjected to regular mechanical vibrations. During the vibration, the particles slide down the powder pile along the sloping surface, resulting in a decreased tilt angle which is called the angle of fall.

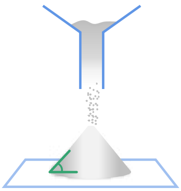
The angle of difference is calculated by subtracting the angle of fall from the angle of repose. The angle of difference is one of several parameters that can be used to evaluate powder floodability. The larger the angle of difference is, the better the floodability will be.

# 78

# HOW TO MEASURE THE ANGLE OF REPOSE?

The angle of repose of a material can be obtained through various methods depending on the ultimate application of the material. The value of the angle of repose is affected by different factors, including particle size distribution, particle shape, and particle surface friction. Fixed funnel, circular platform, tilting box, and revolving cylinder methods are four commonly applied methods to determine the angle of repose.

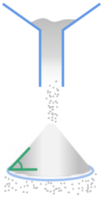
In addition to the above methods, the angle of repose is also accessible through alternative measurement techniques, for example, the internal funnel method. The measurement results for the same powder sample vary after applying different measurement methods. Therefore, it is critical to choose an appropriate measurement method for the determination of the angle of repose, depending on the application.



**Fixed Funnel**

## Fixed funnel method

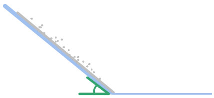
The powder is poured through a funnel and placed onto a platform, forming a cone. When the predetermined height or the base predetermined width is reached, the powder pouring is stopped. In that case, the angle between the sloping side of the powder cone and the horizontal plane is the angle of repose. During the measurement, the funnel height increases slowly and stably, gradually reducing the impact of the particle falling on the measurement results.



**Circular Platform**

## Circular platform method

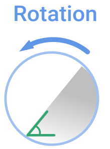
The powder is poured through a funnel and placed onto a circular platform whose diameter is specific. A cone is formed by falling particles. When the powder particles start to fall towards the edge of the circular platform, the angle between the sloping side of the powder cone and the horizontal plane is the angle of repose.



**Tilting Plate**

## Tilting plate method

Powder particles are flattened on a flat plate, then one side of the flat plate is lifted upwards. When the powder particles start to slide down along the tilting plate, the angle between the tilting plate and the horizontal plane is the angle of repose.



**Revolving Cylinder**

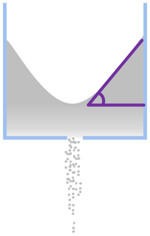
## Revolving cylinder method

The powder is placed into a transparent cylinder. As the cylinder rotates at a specific speed, the sloping side of the powder appears. The angle between the sloping side and the horizontal line is the angle of repose.

# 79

# HOW TO MEASURE THE ANGLE OF INTERNAL FRICTION?

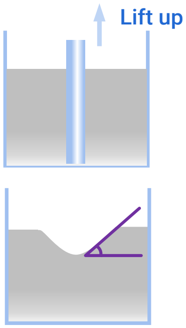
The angle of internal friction indicates the friction property between the internal powder layers. Any point in the powder interior is suffered from the interaction caused by the particles nearby, resulting in friction between the sliding particles. The measurement of the angle of internal friction can be achieved through the following methods.



Flowing

## Flowing test

Powder particles are poured into a cylinder with a hole at the bottom through which the powder escapes. The angle between the sloping side and the horizontal line is defined as the angle of internal friction.



Rod Tension

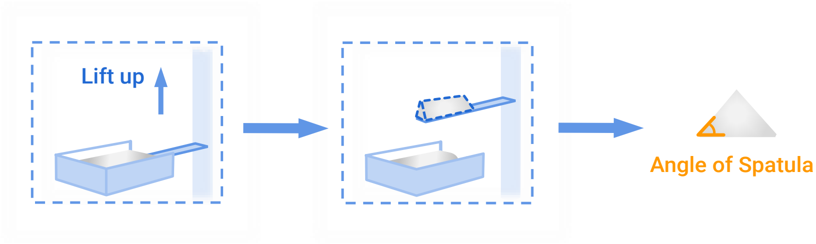
## Rod tension test

A rod is placed vertically at the center of a cylindrical container, which is surrounded by powder particles. The powder surface is flat. Then the rod is lifted upwards, creating an angle between the sloping side and the horizontal line, which is defined as the angle of internal friction.

# 80

# WHAT IS THE ANGLE OF SPATULA?

Suppose a spatula is covered by powder, and then the spatula is lifted upwards vertically. At this point, the angle between the sloping side of the powder on the spatula and the horizontal plane is called angle 1. Then an external force is applied to cause a vibration on the spatula to obtain angle 2. The average of these two angles is called the angle of spatula. The smaller the angle of spatula is, the better the flowability will be. In general, the angle of spatula is larger than the angle of repose.



# 81

# WHAT IS FLOWABILITY?

Flowability is used to describe the flow properties of the powder under gravity. The flowability can be simply evaluated using only one parameter, such as the angle of repose, the compressibility, or the angle of spatula. However, flowability is a property related to various indices, which can be expressed more rigorously as the weighted sum of many parameters associated with powder characterization.

Carr indices method based on the angle of repose, the compressibility, the angle of spatula, uniformity, and cohesion is a more comprehensive method to evaluate the powder flowability. Uniformity can be calculated by dividing D60 by D10. Cohesion is determined by measuring the mass of powder on each sieve after the vibration of the sample for a specific period of time.

## Carr Indices Chart of Flowability

| Angle of Repose | | Compressibility | | Angle of Spatula | | Uniformity* | | Cohesion* | |
|---|---|---|---|---|---|---|---|---|---|
| Degree | Index | % | Index | Degree | Index | No. | Index | % | Index |
| *≤25* | **25** | *≤5* | **25** | *≤25* | **25** | *1* | **25** | - | - |
| *26-29* | **24** | *6-9* | **24** | *26-30* | **24** | *2-4* | **24** | - | - |
| *30* | **22.5** | *10* | **22.5** | *31* | **22.5** | *5* | **22.5** | - | - |
| *31* | **22** | *11* | **22** | *32* | **22** | *6* | **22** | - | - |
| *32-34* | **21** | *12-14* | **21** | *33-37* | **21** | *7* | **21** | - | - |
| *35* | **20** | *15* | **20** | *38* | **20** | *8* | **20** | - | - |
| *36* | **19.5** | *16* | **19.5** | *39* | **19.5** | *9* | **19.5** | - | - |
| *37-39* | **18** | *17-19* | **18** | *40-44* | **18** | *10-11* | **18** | - | - |
| *40* | **17.5** | *20* | **17.5** | *45* | **17.5** | *12* | **17.5** | - | - |
| *41* | **17** | *21* | **17** | *46* | **17** | *13* | **17** | - | - |
| *42-44* | **16** | *22-24* | **16** | *47-59* | **16** | *14-16* | **16** | - | - |
| *45* | **15** | *25* | **15** | *60* | **15** | *17* | **15** | *≤6* | **15** |
| *46* | **14.5** | *26* | **14.5** | *61* | **14.5** | *18* | **14.5** | *6-9* | **14.5** |
| *47-54* | **12** | *27-30* | **12** | *62-74* | **12** | *19-21* | **12** | *10-29* | **12** |
| *55* | **10** | *31* | **10** | *75* | **10** | *22* | **10** | *30* | **10** |
| *56* | **9.5** | *32* | **9.5** | *76* | **9.5** | *23* | **9.5** | *31* | **9.5** |
| *57-64* | **7** | *33-36* | **7** | *77-89* | **7** | *24-26* | **7** | *32-54* | **7** |
| *65* | **5** | *37* | **5** | *90* | **5** | *27* | **5** | *55* | **5** |
| *66* | **4.5** | *38* | **4.5** | *91* | **4.5** | *28* | **4.5** | *56* | **4.5** |
| *67-89* | **2** | *39-45* | **2** | *92-99* | **2** | *29-35* | **2** | *57-79* | **2** |
| *90* | **0** | *≥45* | **0** | *≥99* | **0** | *≥35* | **0** | *≥79* | **0** |

| Evaluation of Flowability | | | | | | | |
|---|---|---|---|---|---|---|---|
| Degree | Very Good | Fairly Good | Good | Normal | Not Good | Bad | Very Bad |
| *Sum of Indices* | **90-100** | **80-89** | **70-79** | **60-69** | **40-59** | **20-39** | **0-19** |

* Determination of using uniformity or cohesion is based on mean bulk density and sieve diameter of the powder sample.

## 82

# WHICH FEATURES OF POWDER AFFECT FLOWABILITY?

Flowability is influenced by many factors, such as particle size distribution, particle shape, density, and electrostatic state, among which the particle size distribution and particle shape are the most important elements. For example, according to research, the decrease in the proportion of the particles whose sizes are smaller than 100μm is conducive to increasing flowability.

# 83

# WHAT IS FLOODABILITY?

Floodability is an indicator used to describe the splashing properties of the powder after overcoming gravity. Similar to flowability, according to the Carr indices chart, floodability can be evaluated through the sum of four indices, representing four parameters of the powder sample, namely flowability, the angle of fall, the angle of difference, and dispersibility. The following chart shows how the parameter values are converted to their corresponding indices.

### Carr Indices Chart of Floodability

| Flowability | | Angle of Fall | | Angle of Difference | | Dispersibility | |
|---|---|---|---|---|---|---|---|
| Value | Index | Degree | Index | Degree | Index | % | Index |
| *≥60* | **25** | *≤10* | **25** | *≥30* | **25** | *≥50* | **25** |
| *59-56* | **24** | *11-19* | **24** | *29-28* | **24** | *49-44* | **24** |
| *55* | **22.5** | *20* | **22.5** | *27* | **22.5** | *43* | **22.5** |
| *54* | **22** | *21* | **22** | *26* | **22** | *42* | **22** |
| *53-50* | **21** | *22-24* | **21** | *25* | **21** | *41-36* | **21** |
| *49* | **20** | *25* | **20** | *24* | **20** | *35* | **20** |
| *48* | **19.5** | *26* | **19.5** | *23* | **19.5** | *34* | **19.5** |
| *47-45* | **18** | *27-29* | **18** | *22-20* | **18** | *33-29* | **18** |
| *44* | **17.5** | *30* | **17.5** | *19* | **17.5** | *28* | **17.5** |
| *43* | **17** | *31* | **17** | *18* | **17** | *27* | **17** |
| *42-40* | **16** | *32-39* | **16** | *17-16* | **16** | *26-21* | **16** |
| *39* | **15** | *40* | **15** | *15* | **15** | *20* | **15** |
| *38* | **14.5** | *41* | **14.5** | *14* | **14.5** | *19* | **14.5** |
| *37-34* | **12** | *42-49* | **12** | *13-11* | **12** | *18-11* | **12** |
| *33* | **10** | *50* | **10** | *10* | **10** | *10* | **10** |
| *32* | **9.5** | *51* | **9.5** | *9* | **9.5** | *9* | **9.5** |
| *31-29* | **8** | *52-56* | **8** | *8* | **8** | *8* | **8** |
| *28* | **6.25** | *57* | **6.25** | *7* | **6.25** | *7* | **6.25** |
| *27* | **6** | *58* | **6** | *6* | **6** | *6* | **6** |
| *26-23* | **3** | *59-64* | **3** | *5-1* | **3** | *5-1* | **3** |
| *≤23* | **0** | *≥64* | **0** | *0* | **0** | *0* | **0** |

| Evaluation of Floodability | | | | | |
|---|---|---|---|---|---|
| Degree | Very High | Fairly High | Tends to Flush | May Flush | Won't Flush |
| Sum of Indices | **80-100** | **60-79** | **40-59** | **25-39** | **0-24** |

PART

F

# *Optical Particle Counting and Sizing*

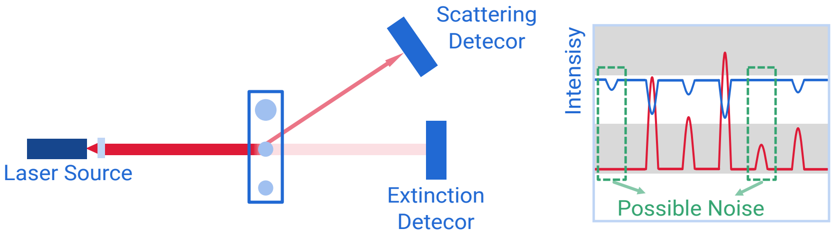
# 84

# WHAT IS AN OPTICAL PARTICLE COUNTER?

An optical particle counter is an apparatus used for particle counting and sizing by applying light extinction or light scattering technology. When a particle passes through the illuminated zone, the light will be partially blocked or scattered, causing a decrease in light intensity or an increase in the scattered light signal, which will be captured by the detectors. Particle counting can be achieved through the calculation of the amount of positive or negative pulses caused by the scattering or blockage of light. A standard calibration curve obtained from a set of reference materials enables particle sizing by substituting the detected pulse heights of particles into the calibration curve.

## 85

# WHAT ARE THE ADVANTAGES OF THE COMBINATION OF LIGHT EXTINCTION AND LIGHT SCATTERING TECHNIQUES?



The combination of light extinction and light scattering technologies increases measurement accuracy. When a particle passes through the illuminated zone, the laser will be blocked, resulting in attenuation in the direction of light propagation, which will be identified by the extinction detector, and a negative pulse will be formed subsequently on the signal diagram. Simultaneously, the light will be scattered at a certain angle. Therefore, the scattered light signal will be received by the scattering detector, yielding a positive pulse, which corresponds to the negative pulse caused by the same particle on the extinction detector. For the same particle, its positive and negative pulse signals correspond to the same x-coordinate in the signal diagram. Therefore, these two coordinated pulse signals can verify each other, and any other pulses not verified will point to the possibility that the signal is treated as noise. Light extinction technology enables particle counting and sizing with high resolution down to 1μm. For fine particles smaller than 1μm, laser scattering is more sensitive, so the application of light scattering expands the measurement range.

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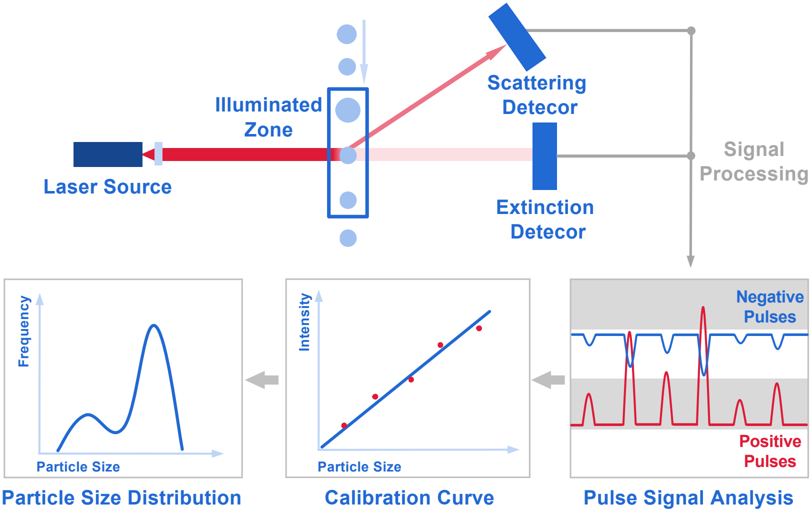
# HOW TO CALCULATE THE NUMBER OF PARTICLES AND MEASURE THE PARTICLE SIZE WITH AN OPTICAL PARTICLE COUNTER?

Optical particle counting uses both light extinction and laser scattering techniques. The particles in the liquid will periodically interrupt the laser light as they pass through the measurement zone, which causes a shadow on the extinction detector when interupted, resulting in a change in current, and, therefore, a negative pulse. The number and strength of the negative pulses indicate the number of particles and their particle size distribution. For small particles, lower light extinction signals are created. When the particle size is smaller than 1μm, the extinction signals are too low to detect. In contrast, the laser light scattering method uses a detector to capture the scattered light. Laser light scattering works by monitoring the change in current caused by the particle interactions with the laser.

Particle counting starts with calculating the amount of the position where the positive and negative pulses simultaneously appear and overlap, which are caused by light scattering and light blockage. Suppose that only a positive or negative pulse signal is generated at a certain position in the signal diagram. Based on the threshold, the algorithm works here to determine whether the signal is treated as a noise or a valid signal. At the same time as particle counting, the pulse height of a single particle will be compared with the calibration curve to obtain the particle size, which is illustrated based on the pulse heights of a set of reference materials. In contrast to the laser diffraction particle size analysis, the particle size distribution yielded from the optical particle counter depends on the calibration curve instead of the inverse algorithm.


Illuminated Zone
Scattering Detecor
Laser Source
Signal Processing
Extinction Detecor
Frequency
Particle Size
Particle Size Distribution
Intensity
Particle Size
Calibration Curve
Negative Pulses
Positive Pulses
Pulse Signal Analysis

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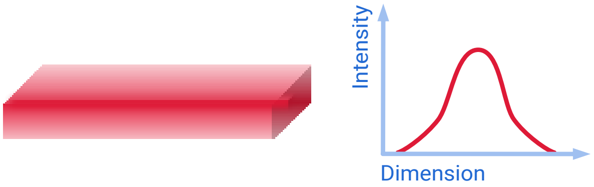
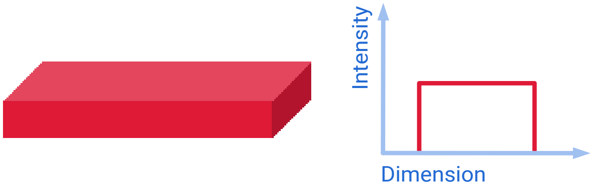
# WHAT IS A FLAT TOP LASER BEAM?



**Gaussian Profile**



**Flat Top Profile**

A flat top laser beam is a laser beam whose light intensity distribution is uniform on the cross section of the laser and with a sharp drop-off at the edges. According to the laser application, the light intensity profile can be modified. An example would be a Gaussian laser beam, whose light intensity reaches its maximum at the central axis and smoothly reduces until it reaches zero at the edge of the laser beam. This type of laser is mainly used for laser cutting. For optical particle counting, counting accuracy increases in the presence of flat top laser beam. The generation of a flat top laser beam can be achieved using an optical beam shaper, with which the initial Gaussian laser beam is capable of being converted to flat top laser beam.

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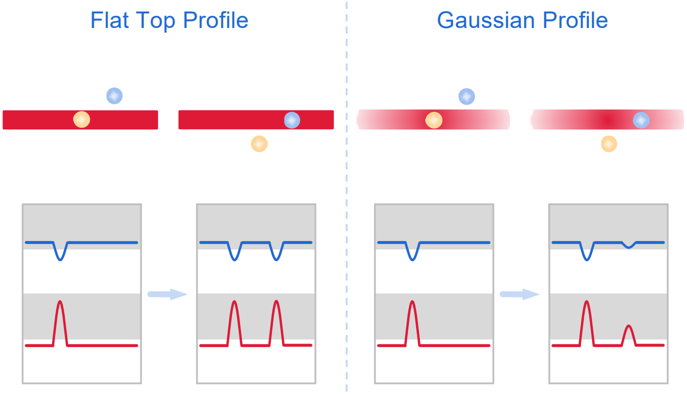
# WHAT ARE THE ADVANTAGES OF A FLAT TOP LASER BEAM?



The flat top laser beam utilized in the optical particle counter provides a laser beam with uniformly distributed light intensity, ensuring a homogeneous illumination of the particles passing through the laser beam. The light extinction method is suitable for particles whose particle size is large than 1μm; for smaller particles smaller than 1μm, laser scattering is prioritized. The light intensity of the “wings” of the Gaussian laser beam is relatively low. Therefore, the corresponding scattered light signal probably cannot be identified when a small particle is illuminated in this position due to insufficient light intensity. The problem can be solved by using a flat top laser beam, in which the particles with the same particle size on the cross section of the laser beam are identically irradiated, providing constant pulse signals, improving counting accuracy, and expanding measurement range. Besides, pulse signals are different when two identical particles are illuminated at different positions on the cross section of a pure Gaussian laser beam, leading to errors in particle size analysis. Compared to Gaussian beams, the application of flat top beams offers more accurate and predictable results.

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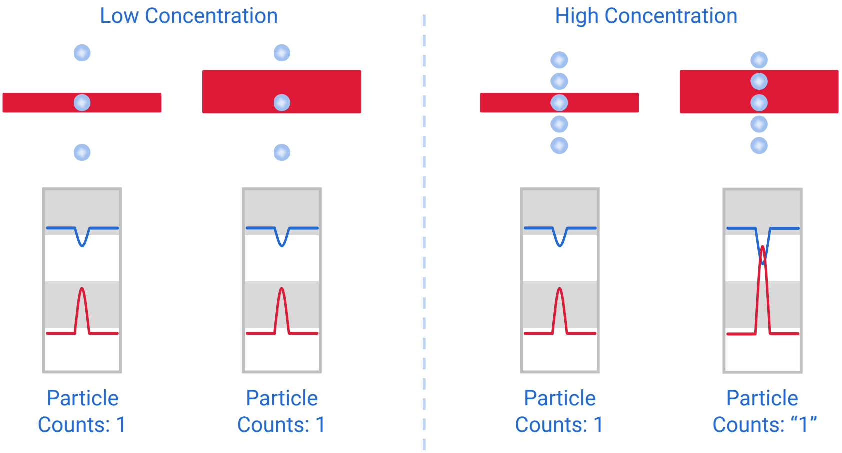
# IS LASER BEAM SHAPING NECESSARY FOR OPTICAL PARTICLE COUNTING AND SIZING?

The shaped beam cross section plays an important role in particle counting and sizing. The rectangular-shaped cross section, whose height is much smaller than its width, is preferred. Below, two laser beams with different cross section shapes are illustrated.



In the case of low concentration, when the particles pass through the measurement zone, the impact of different cross section shapes on counting accuracy is trivial because the interval between the particles along the sample flow is relatively large. However, if the sample concentration is high, the particles in the sample flow are closely arranged. Suppose that the height of the rectangular-shaped cross section equals or is slightly smaller than the particle size, one pulse signal will be generated when the particle is illuminated by the laser, and the particle counting and sizing are accurate. In contrast, if the height of the rectangular-shaped cross section equals, for example, two times of particle size, a counting error probably occurs when two closely arranged particles are illuminated simultaneously, resulting in one pulse signal with higher intensity. In other words, the two pulse signals that should be generated are now overlapped. The number of particles will be therefore recognized as 1. The particle size will also be calculated by mistake due to the high intensity. It is worth noting that the width of the rectangular-shaped cross section is equal to or greater than the upper measurement limit of the particle size, which makes light extinction effective for the detection of large particles.

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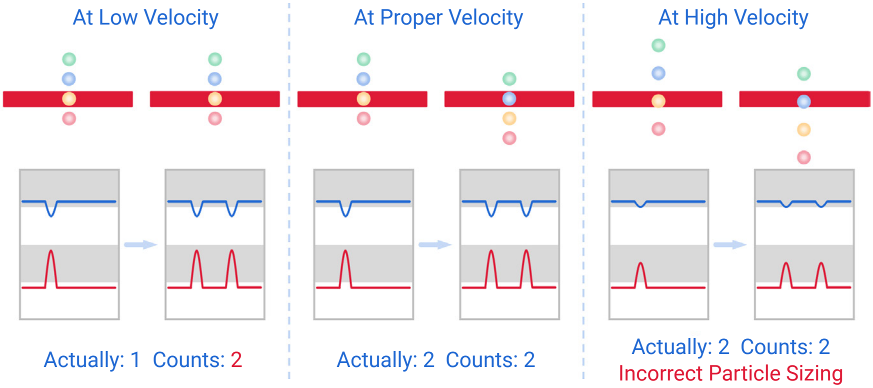
# WHAT IS THE EFFECT OF THE SAMPLE FLOW VELOCITY ON THE MEASUREMENT ACCURACY?

An incorrect sample flow velocity will affect the particle counting and sizing results. If the flow velocity is too low, particles may remain in the measurement zone, leading to repeated identification of the same particle, and hence a counting error will ensue. At high flow velocity, there will be issues with the frequency response of the detector. When the particle leaves the illuminated area, the corresponding response signal has not reached its maximum value due to the short response time within the cell. In this situation, the pulse signal used for the particle size calculation cannot reflect the real particle size because of the low signal height. The flow velocity limit of an optical particle counter can be determined when the measurement error between the results of particle counting at normal velocity and at varied velocity equals ±5%. The larger the error is allowed, the better the performance of the instrument will be.



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# WHAT ARE THE DIFFERENCES BETWEEN LIGHT EXTINCTION AND LASER DIFFRACTION TECHNIQUE?

In an optical particle counter, particles are closely arranged one by one in a straight line in one dimension in the sample flow. In an ideal situation, particles will enter the illuminated area individually, and one pulse signal corresponds to one particle whose signal height will be applied for particle size calculation. In a laser diffraction particle size analyzer, particles are circulated in the dispersion system and irradiated by the laser beam, leading to a signal diagram that is generated through the overlapping of numerous single signals. In the optical particle counter, only the scattered signal at a certain angle will be received. In contrast, photodetectors are arranged in large quantities in specific locations in the laser diffraction particle size analyzer.

For a single particle, most of the scattered signals generated can be received, theoretically, resulting in a signal diagram of a single particle. When numerous particles are illuminated by a laser beam simultaneously, the signal diagram displayed is integrated by innumerable signals of the single particles. Under these circumstances, the particle size calculation cannot be achieved using a calibration curve. Instead, the inverse algorithm is a solution, in which the particle size distribution can be acquired by comparing the theoretical light intensity distribution based on assumption with the measured light intensity distribution. When the residual is within the acceptable range, the assumption can be considered the measurement result.

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# IN WHICH FIELD IS THE OPTICAL PARTICLE COUNTER APPLIED MOST?

For water quality monitoring, an optical particle counter is used for detecting the number of insoluble particles dispersed in water. In the pharmaceutical industry, an optical particle counter is also widely used, especially in vials. The detection of the fat emulsion is based on PFAT5 because excess large particles can lead to capillary clogging. Insoluble particles in a sodium chloride infusion can cause pyrogenic reactions or phlebitis. Determination of PFAT5 using an optical particle counter is necessary. Another important application of the counter is the quantitative analysis of contaminants in lubricating and hydraulic oils. The existence of the particles caused by the contaminants leads to a series of failures. Contamination level evaluation based on various standards using an optical particle counter is of vital significance to the stable long-term operation of the equipment.

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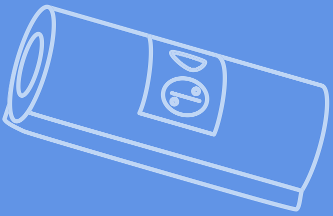
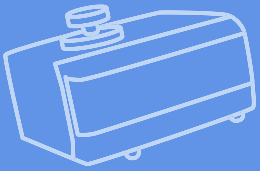
# HOW TO MEASURE HIGH-VISCOSITY SAMPLES WITH AN OPTICAL PARTICLE COUNTER?

For low-viscosity samples such as water which contain insoluble particles, they can be introduced directly to an optical particle counter without an auxiliary feeding system. For high-viscosity samples, a positive and negative pressure auxiliary feeding system is beneficial to sample introduction. For removing bubbles in high-viscosity samples, a negative pressure mode is utilized. A positive pressure is required to introduce the samples to the measurement zone through a narrow path, resulting in stable and continuous sample flow despite the high viscosity.





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Bettersize
BETTER PARTICLE SIZE SOLUTIONS

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