Measurement of Particle Size Distributions

Static Laser Scattering – perfect for Particle Size Measurement in the Range of 0.01 - 2000 μm

Particle size measurement with state of the art laser technology: simple operation, short analysis times, secure, reproducible and dependable, comparable results, cleverly designed dispersion units, fully automatic analysis. And all this in one single instrument from 10 nm into the millimetre range - in production and quality control just as well as in research and development. Fast, simple, flexible.

The Principle
“Static Light Scattering”, “Laser Scattering”, “Laser Diffraction” yes even still “Laser-Granulometry” are frequently used terms for the same technology of particle size determination: The sample material is irradiated with a beam of light and the intensity of the scattered light is measured in as many directions as possible. From this anisotropic intensity distribution and with the aid of a suitable scattering theory, the particle size can be calculated.

The Limits of the Technology
Since large particles lead to small diffraction angles, is due to the sensible upper measuring limit, the possibility given, to even measure the smallest diffraction angles reliably. With a particle size for example of 5 mm, the intensity distribution of the diffracted light in the angle areas of below 0.01° has to be detected. Separating alone the diffracted light of these small angles from the undiffracted laser beam, puts high demands on the optical set-up, its stability and its adjustability. As a logical compromise, which covers the majority of applications and yet still can be implemented with a sensible effort, for numerous instruments the particle size of approximately 2 mm has been established as the upper measuring range. The lower obtainable measuring range of the static light scattering – and here the term is suitable as a distinct differentiation of the relying on a total different principle “Dynamic Light Scattering” – is defined on the basis of the scattering processes. If the scattering particles are getting smaller, a point will reached, where the intensity of the scattered light is the same in all directions. Therefore no information is obtained from the angle dependence, since it is simply non-existent any more. Here we refer to a transition from the (angle dependent) Mie Scattering to the (isotropic) Rayleigh Scattering. Where exactly this transition takes place, mainly depends on the wavelength of the used light and the optical properties of the sample material. Generally it is in a range of about 10 - 30 nm.
While with not too small particles only the intensity distribution in forward motion (i.e. for scattering angles smaller 90°) for particle size determination is sufficient, has the scattered light in the retral-area to be analysed with diameters smaller than approximately 100 nm.

The Instrument Design
In the majority of the cases, as a light source, a laser is utilized, but several manufacturers use LED’s or conventional light sources. The central advantage of lasers is the high light intensity and the excellent beam quality, which is of central significance for the exact measurement of the scattered light.
With an optical set-up we differentiate between the so called “Conventional Design” and “Inverse Fourier Design”, which is also used with the FRITSCH Laser Particle Sizers ANALYSETTE 22.
Conventional Design
With the conventional design the measuring cell is moved in a wide, parallel laser beam and the scattered light is directly depicted behind the measuring cell with a lens on an angle resolving semiconductor detector. The advantage of his set-up lies in the possibility to even use thick measuring layers, which can be advantageous especially with aerosols. Disadvantages of the design are the measurement of large scattering angles and also very small particles. The coverage of a wide measurement range is also coupled here with instrumental intricacy.

Inverse Fourier-Design
With the Inverse Fourier Design, as an inherent difference, the laser beam is moved through a focussing lens (which by the way is called “Fourier-Lens”) and the now convergent laser beam moves through the measuring cell. Now with the ANALYSETTE 22, the distance of the measuring cell from the detector can be varied, where the by the detector detected angle range can be adapted to the particular requirements. The movability of the measurement cell has even especially for the detection of the backward scattered light – and therefore the measurement of very small particles – major advantages. Is the measurement cell positioned just in front of the detector, so through a central micro-drilling in the detector, an additional laser beam can irradiate the sample from the opposite direction. The backward scattered light is then very efficiently captured from the detector, which is also used for the normal forward scattering. Due to the small distance of measurement cell to detector a high sensitivity is obtained.
The detector is one of the decisive factors for the quality of the measurement. Usually either semicircular shaped detectors are used, which are divided in fine rings with gaining width towards the outside or the geometry shows wedge-shaped sectors, which cover specifically defined sections of circles. Are these sectors arranged in a suitable manner, so additional information can be obtained when using polarized laser light, which further increases the accurateness of the measurement.
But as a decisive quality characteristic for a sensor used for particle size determination, the number of the detector elements must be considered. In order to obtain an exact size determination and also a good separation of various particle sizes, thus perhaps achieving a high resolution, must basically, at first the measurement of the scattering intensity with a high angle resolution follow.
And this requires the highest possible number of detector elements. Decisive is not the by every software given number of particle size classes (then this is granted through the analysis routine and not through the exactness of underlying scattering data), but rather more important is initially the effective amount of the detector elements, i.e. how exact can the intensity distribution be measured.
The Dispersion
Laser, optical-set up, detector. These are components, which strongly influence the quality of the instrument, but normally the user will be more or less occupied with them. The main challenge for the user is the sample treatment. In order to guarantee a dependable measurement, the sample material must mostly be fragmented in its single primary particles – i.e. possible present agglomerates have to be fragmented – and then these in sufficient, but not in a too high concentration transported through the laser beam. So called dispersion units assume this part. Here basically it is differentiated between wet and dry dispersion units.

Wet Dispersion
The wet dispersion unit is a closed circulatory system in where in a suitable liquid – mostly water – the sample material is continuously recirculated and dispersed. For the support of the dispersion process, an integrated ultrasonic generator can be used and its intensity is adjustable via the operation software. Unproblematic samples which without great effort submerge directly into the waters surface and have no high share of fines, will be added directly with an applicator into the dispersion unit. The system provides continuous feedback about the already added sample amount and signals when a sufficient amount of material for a dependable measurement is available. Then after a brief dispersion, already a first measurement can start, mostly followed by a second measurement in order to obtain possible changes of the dispersion condition.

The advantage of the wet dispersion is its flexibility and easy handling. Due to the adjustability of ultrasound, variable dispersion duration and if necessary the addition of dispersion aids, even difficult samples can be measured dependably and reproducibly. After a completed measurement the entire reservoir can be automatically emptied, rinsed and filled with new liquid.

Dry Dispersion
In contrast to the wet dispersion, the dry dispersion is not a closed circulatory system. Here each sample portion is accelerated only once with compressed air through a so called annular gap Venturi nozzle system and for the most part broken up into primary particles. The dispersion effect is based on multiple, consecutively occurring strong pressure fluctuations, which leads to highly turbulent flow ratios. Here, strong shearing forces emerge which break the agglomerates apart. Compared with the wet dispersion less energy is introduced into the sample material, so that the dispersion efficiency does not attain the level of the wet measurement.

Therefore, the application range is limited to not too small particle sizes, since the relative adhesive forces with declining particle size clearly rise. Depending of course on
the individual sample material a complete dispersion becomes problematic from a few micrometer particle size. Although the efficiency of the dispersion process of the dry dispersion could be increased by accelerating the sample material on an impact plate positioned right in front of the measurement cell, but with insufficiently hard materials the threat, that via the impact not only the agglomerates are broken apart, but also already a comminution process of the primary particles assumes.

**The Evaluation and Software**

The operation and evaluation software of the ANALYSETTE 22, stores all measurements in a SQL database, whereas the requirements of 21 CFR part 11 can be met. In order to guarantee an optimal reproducibility of the measurement results, the operation of the measuring process is by so called SOP’s (Standard Operating Procedures), which can be flexibly programmed, so that they can be matched to the requirements of each sample. For the evaluation of the measuring data is of course the Fraunhofer theory, as well as for particle sizes in the lower micrometre range and smaller required Mie theory possible.

**Examples from Practical Experience**

In closing, two examples of applications, which were measured with the Laser Particle Sizer ANALYSETTE 22. In the first example Al₂O₃ was ground for four hours in the Planetary Micro Mill PULVERISETTE 7 premium line – black graph in the left area of the distribution. The blue graph on the right shows the distribution of the original material. Clearly recognizable is a particle size distribution which begins at approximately 30 – 40 nm and down to approximately 200 nm shows a continuous course. Above this, in the range between approximately 200 and 500 nm a second peak occurs, which is caused by the abrasion of the ZrO₂ used during the comminution.

![Graph](image)

*Fig. 6: Al₂O₃ comminuted with the Planetary Micro Mill PULVERISETTE 7 premium line - measured with the ANALYSETTE 22 NanoTec plus*

The advantage of the Static Light Scattering compared to the Dynamic Light Scattering (like for example used with the Fritsch Nano Particle Sizer ANALYSETTE 12) becomes immediately clear: with only one method, particle distributions from very large particles down to below clearly under 100 nm can continuously be captured. For example, grindings starting with the original material can be consistently analyzed up to the final fineness.
Also in the second example, the wide measuring range which can be covered with only one measurement plays a decisive part: motor oil with different specifically added aggregates. Initially, during the treatment of this sample pure oil is used in order to perform the so called background measurement. This is performed prior to each measurement in order for example to be able to separate the possible contamination of the measuring cell from the actual measurement data. Afterwards the motor oil with the aggregates was added into the circulatory circuit and the actual measurement was performed. A multiple modal distribution is obtained where to each mode a material can be allocated to.

Fig. 7: Motor oil with different distinct added aggregates-measured with the ANALYSETTE 22 NanoTec plus

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