# Possibilities of High Resolution Intensity Sampling for Laser Diffraction Instruments

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## ABSTRACT

*Laser diffraction (LD) is one of the leading measurement techniques for particle size analysis, covering applications in laboratory and process environments. Basically, laser diffraction is based on first principles not requiring calibration in a strict sense. Particle size is only dependent on instrument parameters as the wavelength of incident light, the geometry of the detector elements and the focal length of the system. All of these instrument parameters are usually precisely known. However, the number of detector elements is limited, especially for laser diffraction restricted in forward direction.*

*Optical models like Fraunhofer or Mie theory are used to resolve particle size distributions out of intensity patterns by inversion techniques. These intensity patterns show a huge variation in intensity spanning several decades in scattering angles. However, resolution and particle size distribution width are limited by the number of detector elements for highly structured intensity profiles (e.g. for monodisperse particles or especially for broad particle size distributions with multiple single peak features as for picket fence distributions, PFDs) even for sophisticated detector geometries.*

*By combining several different focal lengths of single measuring ranges it is possible to increase the effective number of detector elements significantly even for forward scattering regimes spanning a much broader range of scattering angles on a logarithmic scale and therefore increase the resolution and bandwidth of the resulting particle size distribution. With the HELOS/R LD system and the Range Combination option it is very easy and fast to combine several measuring ranges for that purpose. By using all eight measuring ranges of the HELOS/R LD system it is in principle possible to combine up to 248 separate effective detector elements for evaluation.*

*In this paper the possibilities of high-resolution intensity sampling together with future extensions of this method towards resolution enhancement in particle size analysis will be shown.*

**Keywords:** Laser Diffraction, Intensity Pattern, Resolution, Range Combination

## 1 HIGH-RESOLUTION INTENSITY SAMPLING

### 1.1 Range Combination

With the help of Range Combination (RC) of different measuring ranges with the HELOS/R laser diffraction (LD) system it is possible to combine 31 different intensity signals from each of up to eight different measuring ranges to obtain at maximum 248 different intensity signals – and therefore 248 different scattering angles - for one HELOS evaluation. By this increased number of intensity signals even for forward scattering measurements it is obvious that the resolution and sensitivity will be increased by each measuring range added. In Figure 1 all of the 248 separate scattering angles are shown for the used standard HELOS detector. The scattering angle range covers more than four decades in scattering angle for enhanced intensity profile sampling.

In the following examples the possibilities of this high-resolution intensity sampling will be shown exemplarily.

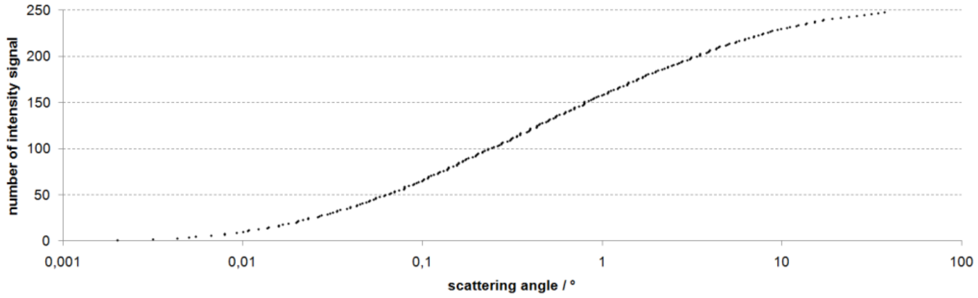


**Figure 1. Range combination of eight different measuring ranges R1 to R8 of the HELOS/R laser diffraction system leads to 248 separate scattering angles for particle size analysis for the standard HELOS detector. The scattering angle range covers more than four decades in scattering angle for high-resolution intensity profile sampling.**

## 1.2 Picket Fence Distribution (PFD)

With the help of Picket Fence Distributions (PFDs), which have been introduced at PSA2008 by Witt et al. (Witt 2008) several equally spaced (on the logarithmic scale) fractions of quasi monodisperse material with equal density are mixed together by equal mass quantities leading to a staircase-like cumulative distribution Q3(x) as it could be seen in Figure 2 (right) for a PFD with seven peaks per decade, indicating six intervals between smallest and coarsest fraction of the mixture shown in the distribution density q3*(x) (Figure 2 left). With simulations based on the precise Mie calculation also presented at PSA2008 (Stübinger 2008) intensity profiles have been performed for various different PFD scenarios.

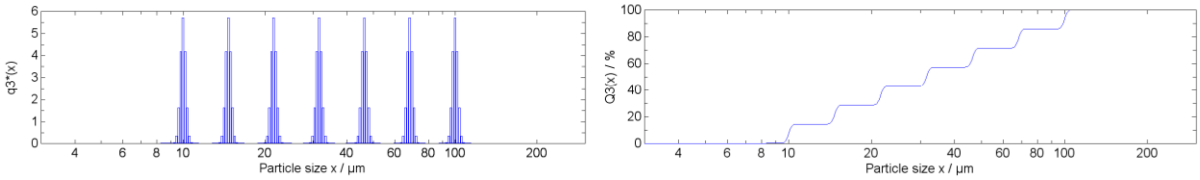


**Figure 2. Simulated Picket Fence Distribution (PFD) of seven equally spaced fractions on logarithmic scale. Distribution density q3*(x) with seven peaks (left); cumulative distribution Q3(x) with a staircase-like characteristic (right).**

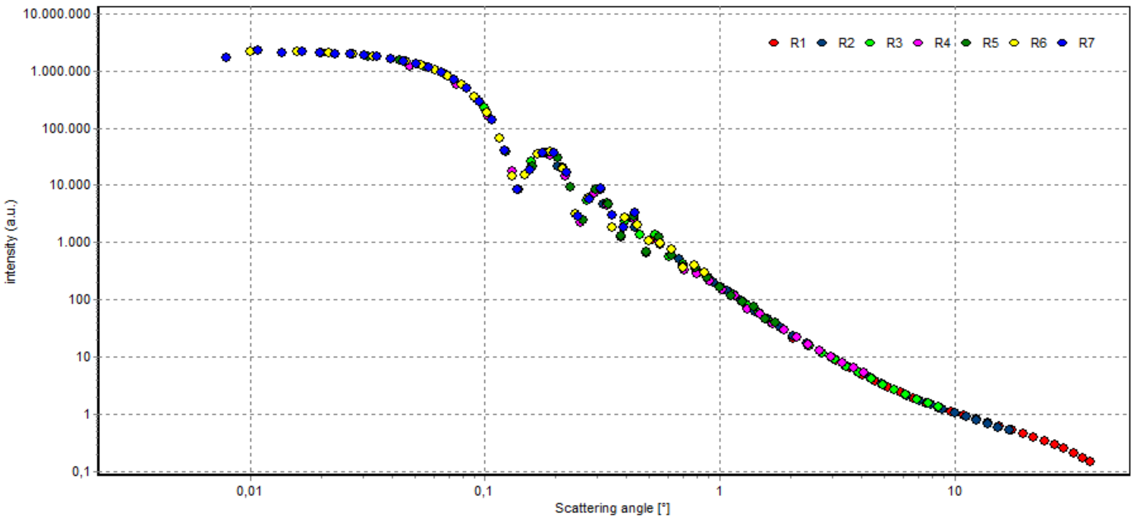


**Figure 3. Angular dependent light scattering intensity pattern of a typical single picket of a PFD resolved by combination of seven different measuring ranges (R1 – R7) resulting in 217 independent sampling points spanning up to four decades in scattering angles and more than seven decades in light scattering intensity.**

In Figure 3 the high-resolution intensity profile is shown for the RC measurement from R1-R7 for one specific picket out of a PFD. With the intensity profile covering more than seven decades in light scattering intensity and more than three decades in scattering angles it is possible to characterize the position and width of the picket peak not only by inversion technique of the HELOS evaluation but also with direct comparison to light scattering profiles using precise Mie calculation.

### 1.3 Mixing ratio

Another possibility for high-resolution intensity sampling is to quantify mixing experiments of different particle fractions with different mixing ratios.

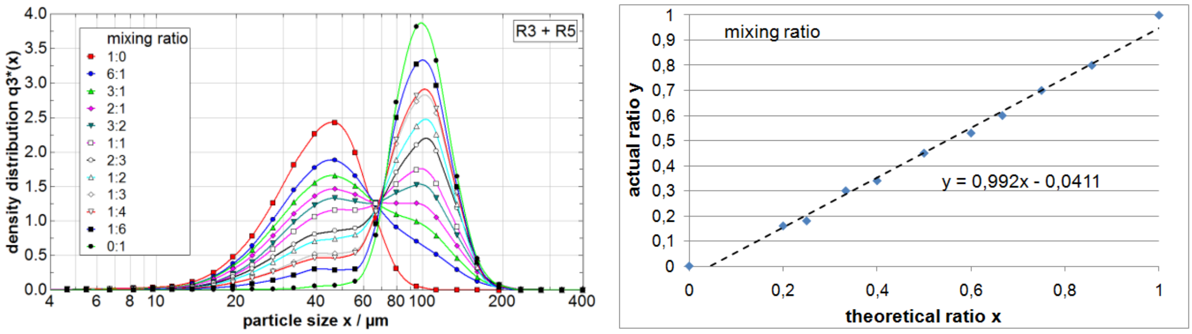


**Figure 4. Mixing ratio experiments of two different glass beads with different mixing ratios ranging from 6:1 to 1:6 together with the pure fraction of the smaller and coarser particle size distribution (left), respectively. The correlation of the actual detected mixing ratio vs. the initial theoretic mixing ratio shows an almost perfect slope ~ 1 (right).**

As an example, mixing experiments with two different fractions of transparent glass beads have been investigated. The mixing ratio achieved by weighing of mass contents of each fraction has been varied from 6:1 to 1:6 in several steps together with the pure fractions (Figure 4 left). The correlation between measured and theoretical mixing ratio should be a straight line with slope = 1 in the perfect case. In Figure 4 (right) a very good agreement between measured and theoretical mixing ratio with slope = 0.992 has been achieved using RC measurements with R3+R5. MIEE evaluation mode applying Mie scattering model has been used because of the transparent glass beads.

### 1.4 Soil samples

Also very broad PSDs of soil samples could be easily measured using RC with combination of measuring ranges from R1 – R7 and therefore use the possibility of high-resolution intensity sampling. In Figure 5 a broad PSD of soil sample is shown containing contributions from very fine material (silt, clay), but also containing contributions from coarser material (sand) covering more than four decades in particle size.

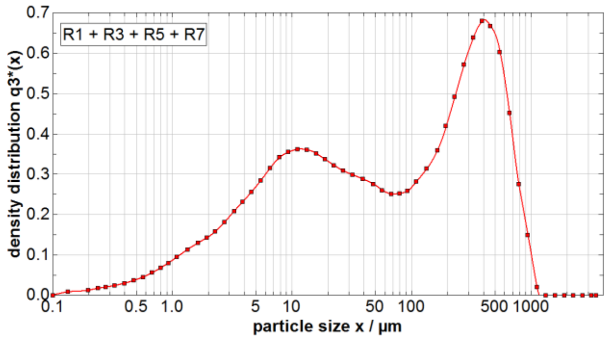


**Figure 5. Very broad particle size distributions from soil samples covering more than four decades in particle size including very fine particles (clay, silt) up to very coarse particles (sand).**

## 2 RESOLUTION

### 2.1 Introduction

For laser diffraction systems three important parameters are relevant: repeatability, accuracy and resolution.

Repeatability and accuracy are well known and already manifested in ISO 13320:2009. Resolution in particle size analysis, however, is still an open field. There are some definitions for resolution of distributions (see section 2.3), however for broad PSDs or highly structured PSDs like PFDs resolution has not yet been defined adequately. In section 2.3 a resolution definition for particle size analysis will be given which is correlated to the well known Rayleigh criterion for spatial resolution in optics using Airy disk formalism described in the next subsection 2.2.

## 2.2 Theoretical background – Rayleigh criterion

Spatial resolution in optics is derived from the Airy disk pattern and the question of how two point spots can be seen as separate spots. For the Airy disk the following scheme is given:

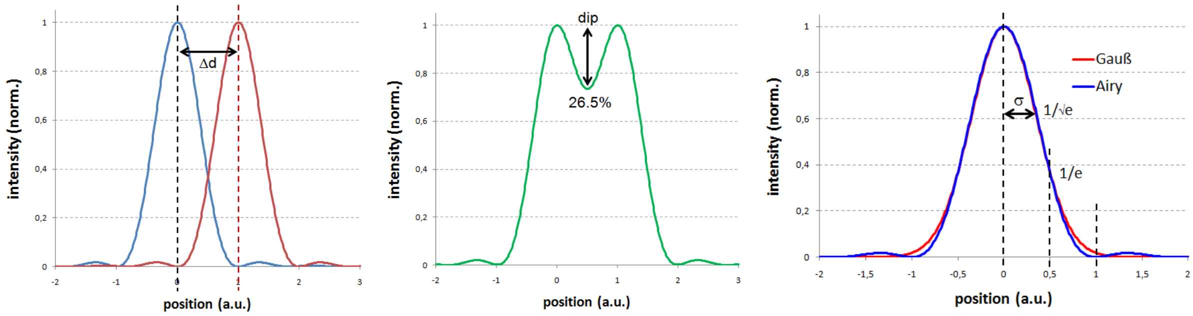


**Figure 6. Resolution defined by Airy disks leading to the well known Rayleigh criteria in optical imaging (left). Combining both functions results in a dip of about 26.5% (middle). Gauß function approximating Airy pattern (right).**

The Rayleigh criterion for barely resolving two objects that are point sources of light is that the centre of the Airy disk for the first object occurs at the first minimum of the Airy disk of the second as can be seen in Figure 6 (left). If this is the case or the distance of both peaks is much higher than the gap Δd than both spots could be seen separated. With the help of the first zero points of the Bessel functions $J_1(y)$ the Rayleigh criterion could be simply obtained from the argument. The zeros of the Bessel function $J_1(y)$ are at y=0, 3.8317, ... leading to the first maximum of the Airy pattern at y=0 and the first minimum at y=3.8317, so that

$$y = \frac{\pi x}{\lambda} \sin(\vartheta) = 3.8317 \quad \text{and therefore} \quad \sin(\vartheta) = 1.22 \frac{\lambda}{x} \approx \frac{\Delta d}{f} \tag{1}$$

In sum both Airy disks exhibit a peak gap Δd and a dip between both peaks of approximately 26.5% (see Figure 6 right) at the Rayleigh criteria.

## 2.3 Resolution in Particle Sizes

For particle size analysis resolution is the ability of the method to see a particle size distribution clearly. There exists a few definitions for resolution (R) of distributions for single monodisperse PSDs (see Equation 2, right) and for two perfectly narrow PSDs (indices 1 and 2 are indicating peak 1 and 2, respectively) which can be clearly resolved as shown in subsection 2.2 (see Equation 2, left), however both definitions are based on a linear scale.

$$R = 200\% \cdot \frac{x_{50,2} - x_{50,1}}{x_{50,2} + x_{50,1}} \quad \text{and} \quad R = 100\% \cdot \frac{(x_{95} - x_5)}{x_{50}} \tag{2}$$

Using the knowledge from the above section a definition for resolution in particle size measurement could be derived even for broad PSDs by transforming the Airy disk pattern into a log-normal PSD with an adequate distribution width $\sigma_{lg}$ on a logarithmic scale. This could be done as follows:

With the dip of 26.5% 1-0.265=0.735 ≈ 2/e between two Airy disks at the Rayleigh criterion from Figure 6 (right) the sum of both Airy patterns in the middle of the gap at Δd/2 could then be expressed by (1-0.265) = 0.735 ≈ 2/e = 1/e + 1/e. Therefore transforming the single Airy pattern to an equivalent (linear) Gauß function with standard deviation σ approximating the Airy pattern (see Figure 7 left) it could be set at Δd/2:

$$y(\frac{\Delta d}{2}) = \frac{1}{e} = e^{-1} = e^{-\frac{1}{2}\left(\frac{\Delta d/2}{\sigma}\right)^2} = e^{-\left(\frac{\Delta d}{\sqrt{8}\sigma}\right)^2} \quad \textbf{(3)}$$

For the (linear) Gauß function the standard deviation $\sigma$ for one single distribution at the Rayleigh criterion is then:

$$\sigma = \frac{\Delta d}{\sqrt{8}} = \frac{1}{2}(x_{84,1} - x_{16,1}) = \frac{x_{50,2} - x_{50,1}}{\sqrt{8}} \quad \textbf{(4)}$$

For log-normal PSD the transformation leads to the following condition for $\sigma_{lg}$:

$$\sigma_{\text{lg}} = \frac{1}{2}\lg\left(\frac{x_{84,1}}{x_{16,1}}\right) = \frac{\lg\left(\frac{x_{50,2}}{x_{50,1}}\right)}{\sqrt{8}} \quad \textbf{(5)}$$

Equation (4) (on linear scale) and Equation (5) (on logarithmic scale) have to be fulfilled in order to resolve the two PSD distinguishably.

### 2.4 Example: Picket Fence Distribution (PFD)

First consequence of the above given definition for the resolution of particle size is related to PFDs. With s the number of pickets/decade (which means (s-1) intervals between smallest and coarsest fraction within the decade) the position of the pickets are given by

$$x_{50,i} = x_{50,1} \cdot 10^{\frac{i-1}{s-1}} \text{ for i = 1, ..., s} \quad \textbf{(6)}$$

The maximum distribution width $\sigma_{lg}$ of one picket out of the PFD with s pickets/decade is then given by Equation (5) with:

$$\sigma_{\text{lg}} = \frac{1}{2}\lg\left(\frac{x_{84,1}}{x_{16,1}}\right) \le \frac{1}{\sqrt{8}}\lg\left(\frac{x_{50,2}}{x_{50,1}}\right) = \frac{1}{\sqrt{8}}\lg\left(10^{\frac{1}{s-1}}\right) = \frac{1}{\sqrt{8}\cdot(s-1)} \quad \text{and therefore} \quad \left(\frac{x_{84,1}}{x_{16,1}}\right) \le 10^{\frac{1}{\sqrt{2}\cdot(s-1)}} \quad \textbf{(7)}$$

This yields a maximum distribution width $\sigma_{lg}$ of one picket of PFD 7 (s=7) of $\sigma_{lg} = 0.059$ or $x_{84}/x_{16}$ =1.31 to be resolved. With single picket widths smaller than these values it should be theoretically possible to resolve all seven pickets per decade clearly. In simulations this has been achieved already (all seven pickets resolved) with increased number of size classes in combination with the high-resolution intensity sampling procedure of HELOS/R shown in the previous section 1.

## 3 CONCLUSIONS

High-resolution intensity sampling with HELOS/R and Range Combination does improve laser diffraction systems in terms of resolution, mixing ratios, broad particle size distributions. Resolution in particle size on logarithmic scale is formulated in relation to the Rayleigh criterion and adapted to Picket Fence Distributions (PFDs).

## 4 REFERENCES

STÜBINGER, T., KÖHLER, U., WITT, W., (2008), 100 Years of Mie Scattering Theory: Expanded Size Range by Extreme Precision Calculations, PSA2008, Stratford-upon-Avon, England.

WITT, W., STÜBINGER, T., JORDAN, J., (2008), Improved Standards in Laser Diffraction, PSA2008, Stratford-upon-Avon, England.