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Laser Diffraction - unlimited ?

1 Introduction

Within the past twenty years, particle size analysis with laser diffraction (LD) has been subject to rapid development; evaluation methods based on pure physics, such as the well-known Fraunhofer /1/ and Mie /2/3/ solutions, have been introduced. The size range was extended stepwise from 1µm to 200µm to approximately 0.1µm to 3500µm. The introduction of dry dispersers /4/ has extended the fields of application to aerosols. Meanwhile LD has evolved into an independent, extremely efficient method for the analysis of particle size distributions. It has therefore become the dominant technique for optical particle sizing in laboratory (off-line) and production environments as in-line, auto-line and, in combination with sample couplers, on-line applications /5/.

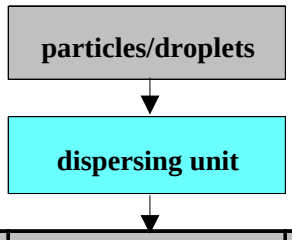
The development of LD continues - unlimited? In the present paper, an attempt is made to appraise the current situation of LD and to give some answers and examples about its possibilities and limitations.

2 Present situation

At present, several renowned manufacturers of particle measuring equipment offer LD instruments. We will focus our interest only to those instruments that use pure LD and do not combine LD with other methods such as dynamic light scattering etc. These instruments offer extended measuring ranges but in principle require a precise knowledge of the optical constants of the sample and the transporting medium, which are often not known or even undefined (for mixtures like cement, coffee etc.).

The structure of a pure LD instrument is demonstrated in fig. 1. It always consists of a light source, usually a laser, which generates a monochromatic coherent parallel beam. This is followed by the beam processing unit, usually a beam expander with integrated spatial filter, producing an extended and nearly ideal wave field for the illumination of the dispersed particles. The particles are moved through the measuring zone by some transporting media. The movement of the particles is necessary, because this smears out the phase relationships of different particles and allows superimposition of intensities instead of amplitudes of the light waves of different particles. The particles interact with the incident wave-field in terms of diffraction, deflection, absorption and refraction generating an angular dependent

intensity distribution $I(\theta)$.



light source	beam processing	measuring zone	detection	data acquisition	mathematic. evaluation	data output
generation of monochrom. coherent light, λ	beam expansion, spatial filtering	**Interaction:** diffraction (deflection, absorption, reflection)	$I(\theta) \rightarrow I(r)$; $I(r) \rightarrow i_n$	i_n –> intensity vector *L*	Fraunhofer-, Mie-theory, Philips Twomey	size distribution $Q_3(x)$
laser	**beam expansion unit, pin hole**	**transporting medium**	**fourier optics, multi-element detector**	**electronics I→U; A→D**	**computer, software**	**display, paper, files, database**

Fig. 1: Functional schematic of an LD system for particle size analysis

$I(\theta)$ is converted by the fourier optics (usually a lens) into a spatial intensity distribution $I(r)$ which is measured by a multi-element photo-detector and converted into a set of photo-currents i_n. The subsequent electronics convert and digitise the photo-currents and supply the evaluating computer software with input data in terms of an intensity vector *L*. With the help of the wavelength λ, geometrical parameters of the detection unit (and the complex refractive index *m* if Mie-theory is used) *L* is converted into the particle size information, such as $Q_3(x)$ etc. that is finally output.

For Fraunhofer the spatial distribution $I(r,x)$ yields /1/

$$I(r,x) = I_0 \int_{x_{\min}}^{x_{\max}} N_{total} q_0(x) \cdot \left(\frac{kx^2}{2}\right)^2 \cdot \left(\frac{J_1(krx)}{krx}\right)^2 dx \quad \text{with } k = (\pi / \lambda f) \tag{1}$$

For monodisperse particles a first dark ring occurs at r_0 with

$$r_0 \cong 1.22 \frac{\lambda \cdot f}{x} \tag{2}$$

From equation (2) it is obvious, that the possible size range of an LD instrument is linearly dependent on the wavelength λ and the focal distance f of the fourier optics.

3 New improvements

3.1 Light sources

Fig. 2 offers a selection of possible light sources (lasers) in combination with the operating range of detectors and the transmission range of materials to be used for the fourier optics. Most frequently the HeNe laser with $\lambda = 632.8nm$ is used with a Si-detector and the standard Crown Glass or similar.

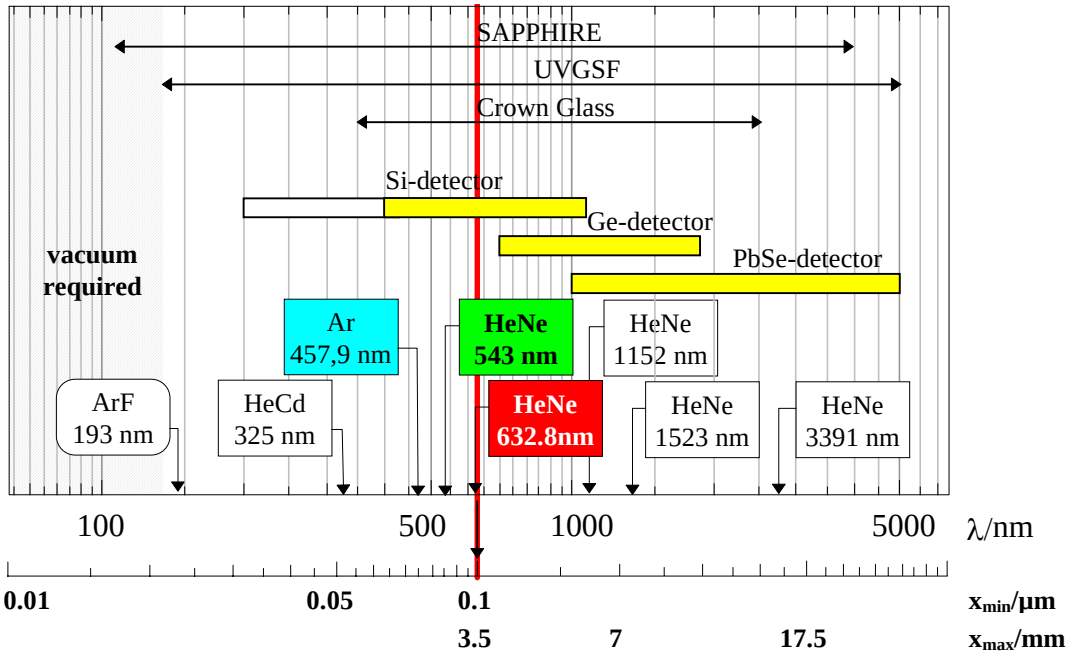


Fig. 2: Possible light sources, detectors and materials for fourier optics.

On the fine side, the range of λ is limited by the transmission of air, so below *180nm* LD would need to operation in a vacuum. The ArF laser is currently the technological limit, but it allows for pulsed operation (10ns/50Hz) only. The most interesting candidate is the continuous wave (CW) HeCd laser, which would extend the lower size limit by a factor of two. So the parameter free extended Fraunhofer solution could become applicable down to 0.05µm. But the price of the HeCd laser is currently about 30 times that of a HeNe laser. The green HeNe laser does not give any significant progress.

On the coarse side the size limit is currently 3.5mm. This could be extended to more than 7mm with semiconductor lasers or infrared HeNe lasers. Here the problem is the detector material which cannot be produced in precise multi-element detector geometries or is very expensive (InAs). The background from infrared radiation at room temperature would complicate the application. So perhaps the HeNe laser with 1152nm would be a good compromise and nearly double the maximum size to about 6mm.

Semiconductor lasers are widely used in the range from *650nm* to *1.55µm*. Their

advantage is size, but they produce an elliptical beam, which is expensive to correct, and a large wavefront distortion (typ. $\geq \lambda / 6$) resulting in a poor quality of the wave-field. They have to be stabilised and cooled. So currently the HeNe laser with $\lambda = 632.8nm$ is the best solution. Our company no longer uses semiconductor lasers.

3.2 Adaptable beam expansion

For an LD instrument the spatial intensity distribution on the detector surface is the sum of the fourier transformed projected areas of the illuminated particles. The largest „particle“ which is transformed is the beam diameter itself. As the TEM_{00} mode output of a laser has a Gaussian intensity distribution with

$$I(r) = I_0 e^{-2r^2/w_0^2} \tag{3}$$

and $2w_0$ is the full width at $I / I_0 = 1/e^2$. The fourier transform is also Gaussian with the width *w* expressed by /6/

$$w = \frac{\lambda \cdot f}{\pi \cdot w_0} \tag{4}$$

if the detector is set into the focal point $s'' = f$. For the analysis of large particles it is important, that $w_0 >> x_{\max}$ because large focal lengths f have to be used and significant intensity would be detected on the important inner rings of the detector. Additionally it is difficult to avoid statistical errors caused by the position of the particles inside the beam.

For the detection of small particles wide scattering angles θ have to be detected. For an extended particle ensemble it is important, that the diffracted intensity significant for $x_{\min}$ of all particles is caught by the opening of diameter *d* of the fourier optics. As *d* is limited, especially for the requested small *f*, it is important to reduce the beam diameter w_0 to maintain a useful working distance *z*.

From fig. 3 the simple geometrical relationship can be expressed as

$$d(z) = w_0 + 2 \cdot z \cdot \tan(\theta) = w_0 + 2z \frac{r_D}{f} \tag{5}$$

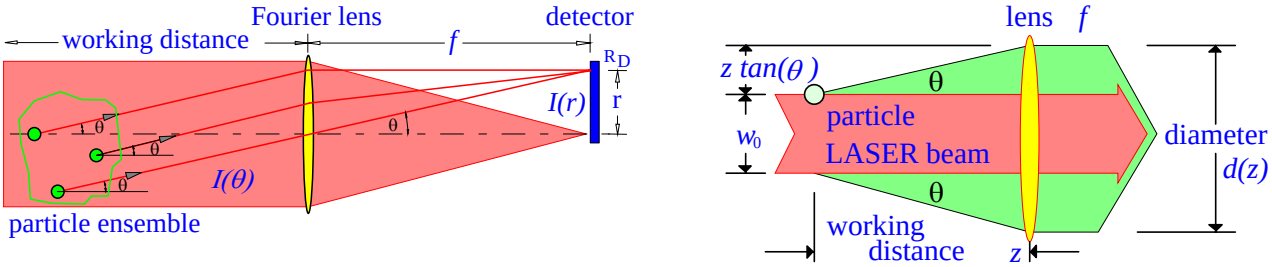


Fig. 3: The required lens diameter d(z) as function of the beam diameter w_0

So we have introduced a variable beam expansion unit, which allows for the automatic selection of one of three different beam diameters according to the appropriate size range.

$2w_0$/mm	2,2	13	26
measuring ranges/µm	0.1/0.18-87.5	0.5/0.9-875	0.5/9.0-3500
maximum focal distance *f*/mm	50	500	2000
theoretical spot width *w*/µm	4,58	7,75	15,49

Table 1: Related beam diameters of the adaptable beam expander (ABE)

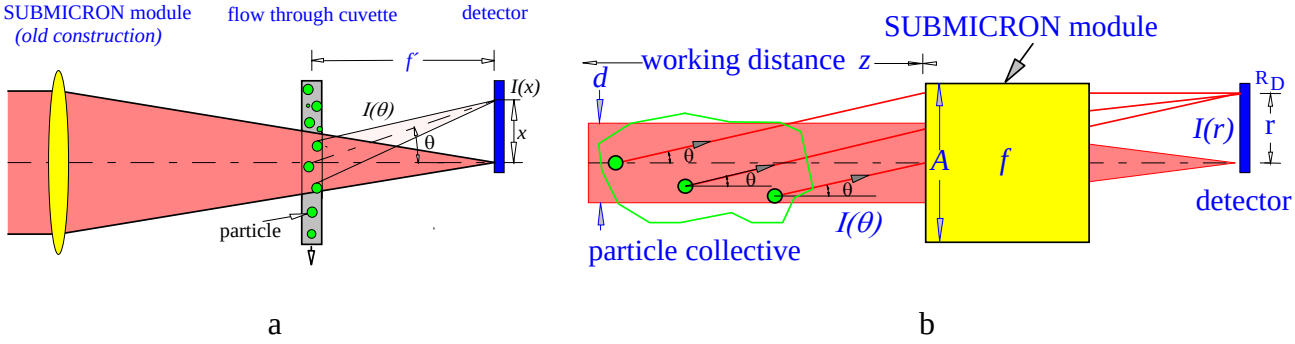
3.3 Extended fourier optics

Fourier optics usually are simple lenses with a focal distance *f*. Improved instruments use achromatic doublets to minimise imaging errors especially for small focal distances. Due to the fact that the detector diameter is fixed, the measurement of large particle diameters requires long focal distances. Instruments with *f=1500mm* and an upper size limit of 2625µm have been successfully introduced. They mark the limit of this technology because of the need for mechanical stability.

In 1992 we have introduced the TOPMICRON technique. Here an optical system which acts as a linear angular amplifier precedes the fourier optics. This combination has an effective focal distance *f'* which yields the amplification factor times the focal distance *f*. TOPMICRON modules with *f'=200mm*, *1000mm* and even *2000mm* are available. They have scaled down the size of the instruments by a factor of two and strongly improved the mechanical stability. So LD instruments can be used up to 3.500µm even under conditions of industrial production (vibrations).

On the fine side of the measuring range one has to use small focal distances (e.g. $f = 20mm$) with a fixed detector geometry. From chapter 3.2 we know, that large apertures are requested. Lenses with an opening diameter of e.g. *50mm* and a focal length of *f=20mm* are not available. The focus would be inside the lens. For this reason the well-known inversed Fraunhofer /7/ set-up was often used, as shown in fig. 4a. This set-up using a convergent beam, is mathematically equivalent to the

operation in the parallel beam (but with a phase factor). As the effective focal distance *f'* is now defined by the distance of the particles from the detector, extended particle collectives cannot be measured. So narrow cuvettes have to be used, limiting the applications to wet dispersion and still introducing the uncertainty $\Delta f = w_{cuvette} / f'$ due to the finite width of the cuvette $w_{cuvette}$. The incident angle on the detector becomes very large so the angular dependence of the sensitivity has to be taken into account.



a b

Fig. 4: Set-up for measuring fine particles using a convergent beam (a) and a SUBMICRON module (b) in the parallel beam

Following the principle of the TOPMICRON technique, it was possible to design SUBMICRON modules by extensive use of beam tracing calculations that overcome these restrictions. They can operate with a small parallel laser beam (*2w=2,2mm)* and convert the scattered intensity $I(\theta)$ of an extended particle collective into the spatial intensity distribution $I(r)$ as if it had a focal distance $f' = 20mm$ or $f' = 50mm$ as demonstrated in fig. 4b. The modules consist of a series of lenses which convert even large scattering angles of up to 38° stepwise to nearly horizontal beams. So the detector is illuminated close to its normal incidence. As the first lens of the modules is concave it is well adapted to the large scattering angles.

The modules are optimised for large working distances. The measuring range from 0.25/0.45µm to 87.5µm is covered by the *f'=50mm* module. For this module it was possible to achieve $z \leq 65mm$. So it can be used in combination with dry dispersers such as RODOS or auto-RODOS module, at the output funnel of inhalers or for dry in-line applications in addition to standard wet dispersing systems such as SUCELL or CUVETTE.

The measuring range from 0.1/0.18µm to 35µm is covered by the *f'=20mm* module. This module is optimised for $z = 20mm \pm 1mm$ and can be used for dry and wet dispersing systems. Fig. 5 shows the modules.

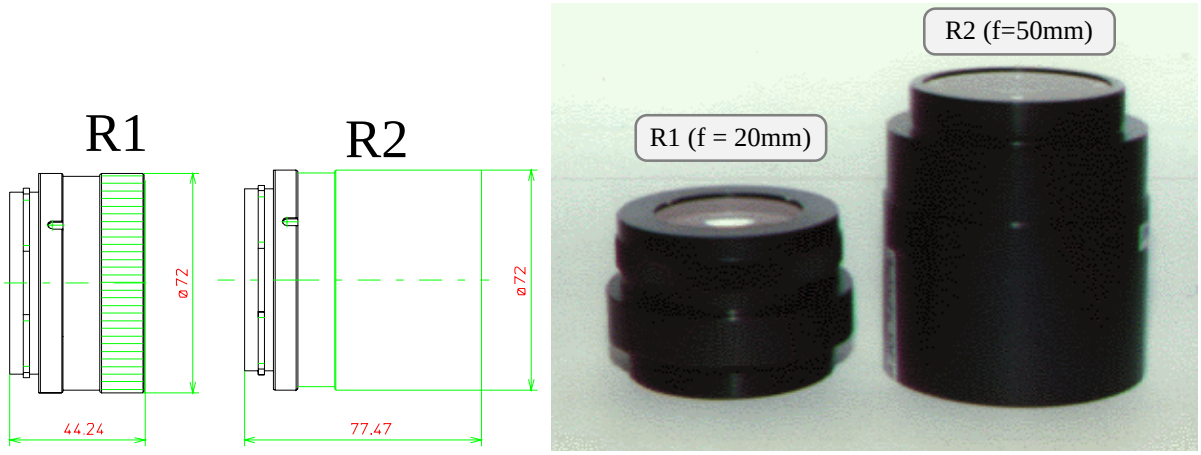


Fig. 5: SUBMICRON modules 0,1 (R1, *f'=20mm*) and 0,3 (R2, *f'=50mm*)

For the first time these modules extend the application of LD for dry powders and aerosols into the SUBMICRON range. This is done without breaking the cylindrical symmetry of the optical set-up in contrast to possible solutions with off-axis detectors and lenses.

There is a strong demand for an open measuring zone for applications with sprays and aerosols. Daylight which scatters at the particles nearest the detector cannot be compensated for by a reference measurement due to the absence of the particles during the reference. The solution is a bandpass-filter at the input of the fourier optics, which is only transparent for a small band of wavelengths centred at the wavelength of the laser. It was discovered, that a combination of two edge-filters can reduce the influence of daylight by more than a factor of ten. Interference filters cannot be used due to the fact that the transmission is poor and strongly dependent on the incident angle. So all the fourier optics of our LD instruments are now equipped with a bandpass-filter.

3.4 Detector design

The detector is the most sophisticated element of an LD instrument. It should have a number of discrete sensitive elements of precise geometry, usually rings of homogeneous sensitivity and a reflectivity of zero. The latter is very important, because the intensity in the central maximum is about 100 times larger than the diffracted intensities on the rings, and any reflection of the central beam at the fourier optics can become significant on the ring elements. This list could easily be extended taking the dynamic requirements of the subsequent electronics into account. Consequently, many different designs have been developed and patented.

Since the intensity on the detector is the fourier transform of the spatial properties of the particle ensemble, any symmetry of the ensemble is conserved on the detector. If the particles are e.g. needles and the needles are not completely randomly distributed,

(perhaps the horizontal orientation prevails slightly due to the flow conditions), the intensity distribution has a horizontal symmetry axis. The best way to overcome this problem would be to use a detector with circular elements which integrate over 360°. Due to the bonding requirements this is currently not possible and in reality not necessary /8/. So since the very beginning we used the best solution: a detector with semicircular-ring elements which integrates over 180°. Detectors with smaller integration angles are cheaper but sensitive to symmetry-breaks.

The detection of very coarse particles requires very small detector elements and an exact alignment of the highly intensive central-beam to the centre of the detector. The centre of the detector consists of three sector-fields which now allow for dynamic re-centring even during the data acquisition.

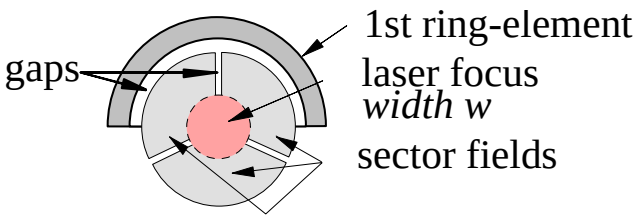


Fig. 6: Arrangement of sector-fields in the centre of the detector.

For a typical detector, the active elements are usually p-doped areas in an n-doped Si-substrate. They are separated by gaps covered by a layer of SiO_2 to minimise the crosstalk between the elements (see fig. 6). Nevertheless, the light crosses the SiO_2 and generates some holes in the p-doted wings under the SiO_2. So the active area of the element is apparently increased and the geometry is no longer precisely known causing systematic errors in the evaluation.

So a new manufacturing process was developed for the new detector design. Now the detector is completely covered with a homogeneous active area which is separated by virtual border lines into rings and central segments. Thus the geometry is now only limited by the known tolerances of the manufacturing process. The centering of the central beam is improved and its intensity and the resulting optical concentration C_{opt} can be precisely evaluated. Also the reproducibility for coarse particles is increased.

The exact knowledge of the optical concentration is necessary for the correct subtraction of the background signal from the measured signal, the former being measured during the reference measurement and attenuated by the particles. A precise subtraction is important especially for measurements at very low concentrations such as spray or inhaler applications, and for the detection of small fractions of coarse particles in a large amount of fines.

4 Results

4.1 Reproducibility and Stability

The reproducibility of LD systems has been tested with standard HELOS/KA and LA systems using BCR samples /9/. The samples were split with a rotating riffler into partial samples. Each partial sample was measured „no. of measurements“ times.

Material	BCR 66	BCR 67	BCR 68	BCR 69	BCR70
Measuring range/µm	0.1/0.18-35	0.25/0.45-87.5	0.5/9.0-1750	0.5/1.8-350	0.25-0.45-87.5
Dispersing unit	SUCELL	SUCELL	GRADIS	SUCELL	SUCELL
Sample	0.1-0.2g from paste	sample split; complete partial sample 0.4g	sample split; complete partial sample 0.4g	sample split; complete partial sample 0.4g	sample split; complete partial sample 0.4g
no. of partial samples	6	6	6	6	6
no. of measurements	≥3	3	1	3	3
x_{10}/µm	0,81	3,20	194,1	21,0	1,27
x_{16}/µm	0,94	5,26	223,1	25,4	1,57
x_{50}/µm	0,61	4,78	357,4	48,7	3,33
x_{84}/µm	2,72	21,10	537,8	82,6	7,10
x_{90}/µm	3,13	24,36	593,3	93,5	7,79
x_{max}/µm	8,60	43,50	870,0	146,0	30,50
σ for 6 partial samples KA	**0,15%**	**0,08%**	n.a.	**0,38%**	**0,22%**
σ for 1 partial samples KA	**0,04%**	**0,04%**	**0,34%**	**0,13%**	**0,03%**
σ for 6 partial samples LA	n.a.	**0,02%**	n.a.	n.a.	**0,28%**
σ for 1 partial samples LA	n.a.	**0,02%**	n.a.	n.a.	**0,04%**
σ for 6 partial samples KA	n.a.	**0,58%**	n.a.	n.a.	n.a.
σ for 6 partial samples KA after 5 h	n.a.	n.a.	n.a.	n.a.	**0,64%**

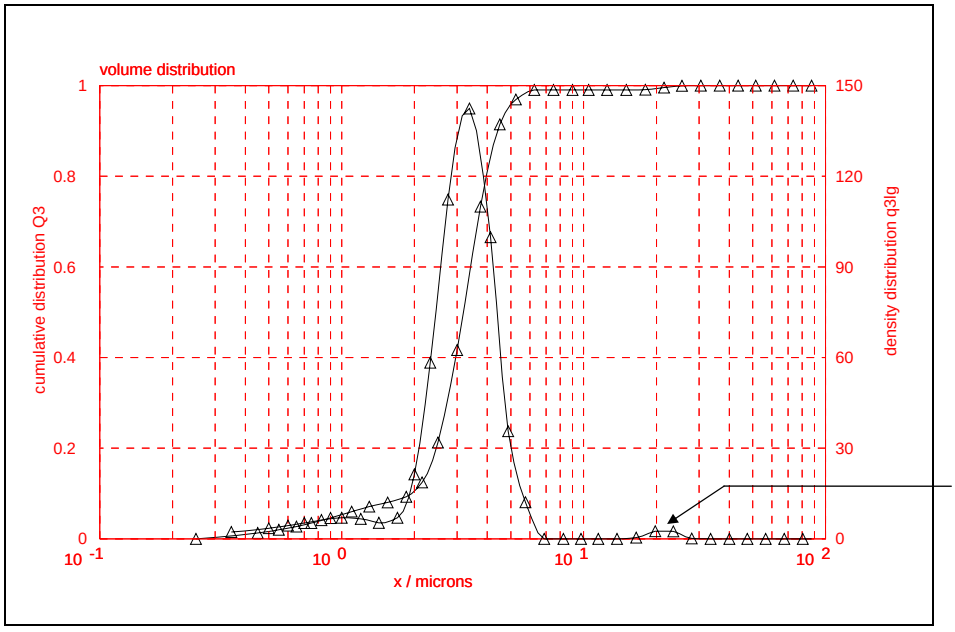
Table 2: Test of reproducibility of HELOS/KA (KA), HELOS/LA (LA) and second HELOS/KA (KA2)

The x_{10} to x_{max} values are averaged over all measurements. All standard deviations σ are much smaller than 1%. The largest is obtained for BCR68 and BCR69 due to the small sample and coarse particles. The σ evaluated on different instruments show nearly no differences. The comparability of absolute results was 0.58%. BCR70 was slightly unstable in suspension. The standard deviation for measurements of the same partial sample is typically more than a factor of two smaller than σ over all 6 partial samples. Sampling errors from the rotating riffler dominate!

The extreme stability is also proven for an in-line application in the fines outlet of an air classifier /10/. Here a single reference measurement has to be used for a full eight hour batch.

4.2 Sensitivity

Good reproducibility could be the result of reduced sensitivity, i.e. the LD sensor would not see small changes of the PSD of the sample. So it is important, always to combine reproducibility data with sensitivity data, as for example demonstrated in fig. 7.



LD-System:

HELOS/KA

Range:

0.5/0.9-175µm

Dispersion:

SUCELL

Material:

Mixture of diamonds

Coarse fraction:

2.8%

Fig. 7: Detection of a small fine fraction

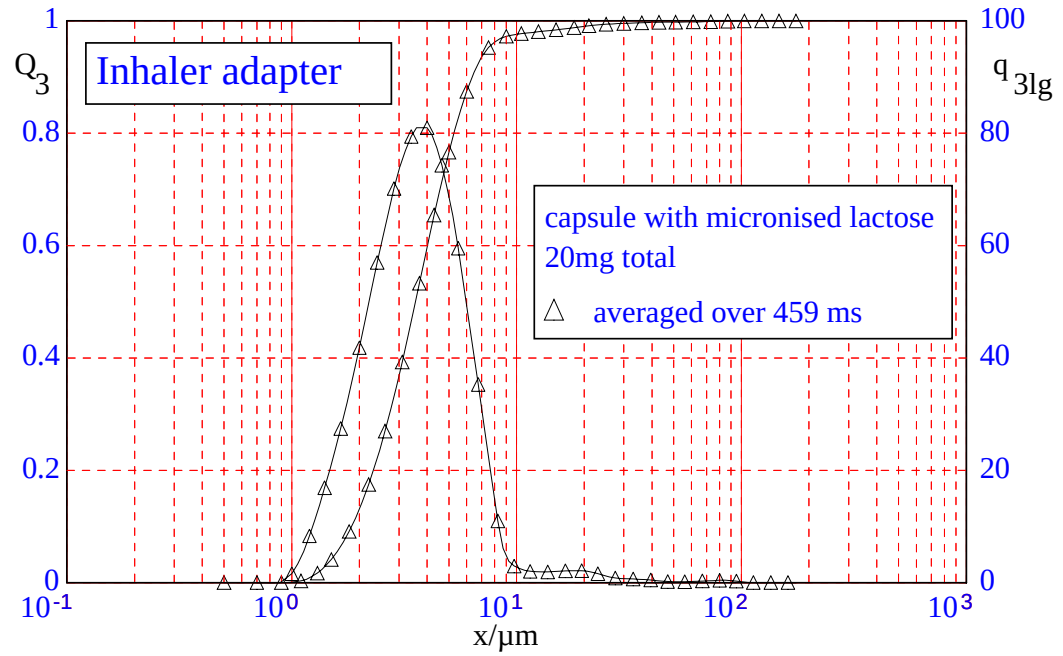
Due to the improved background correcting and evaluation algorithms even isolated populations on the coarse side of the distribution can be detected down to below 1% of volume fraction.

But what can be done, when the size of the sample is extremely small, and one is interested not only in the integral size distribution but also in the development of the size distribution with time? This is often the case for spray and inhaler investigations. Fig. 8 shows the results of a medical inhaler that was filled with 20mg of lactose powder.

The small coarse fraction when integrating over 20mg can be resolved in a number of time slices down to below 5ms/scan by use of the new trigger algorithm. From this data it becomes clear, that the origin of the coarse fraction is most probably some fragments of the broken capsule crossing the measuring zone first.

The peak at 100µm demonstrates that only a few single particles are sufficient to produce a stable output with LD. So the sensitivity limit is approaching that of single particle counting techniques.

a)



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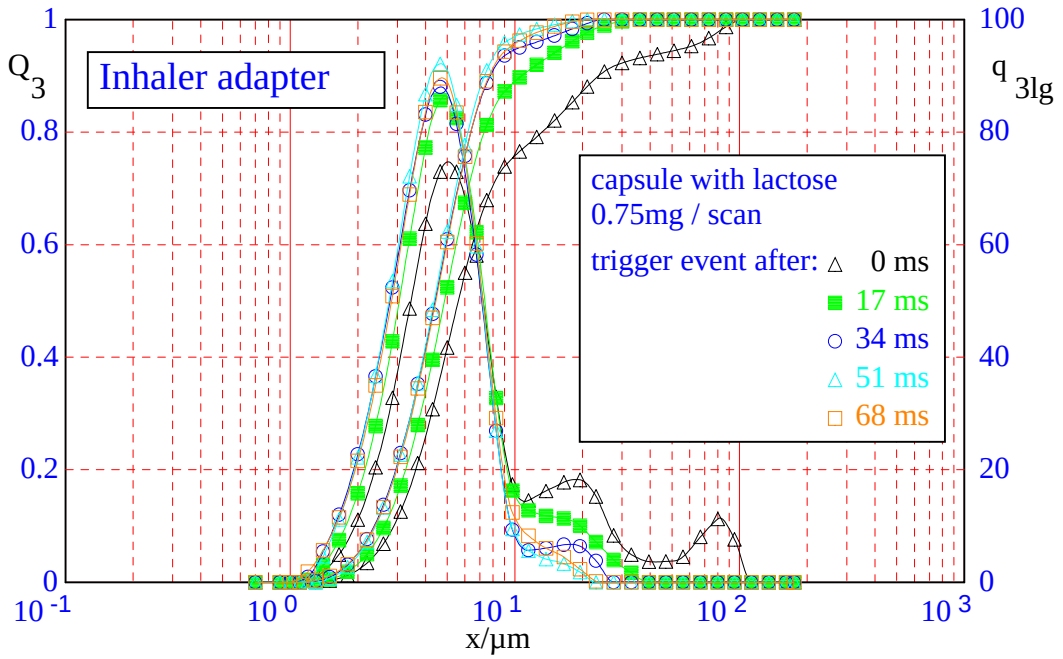

Fig. 8: Integral (a) and time resolved (b) particle size distribution (PSD) results of lactose using the INHALER-ADAPTER.

4.3 An on-line application

Finally one can try to combine all progress made in LD in a single project. This was successfully done for the on-line analysis of the PSD of coal. The primary mass flow of about 120t/h is reduced by a primary sampler to about 2t/h. This partial flow is transported to a sieve which adapts the coarse side of the PSD to the requirements of the LD system. A subsequent rotating sampler/sample splitter (ROPRON, /11/) reduces the mass flow to about 20g/min which is sufficient for about 6 measurements per hour (fig. 9).

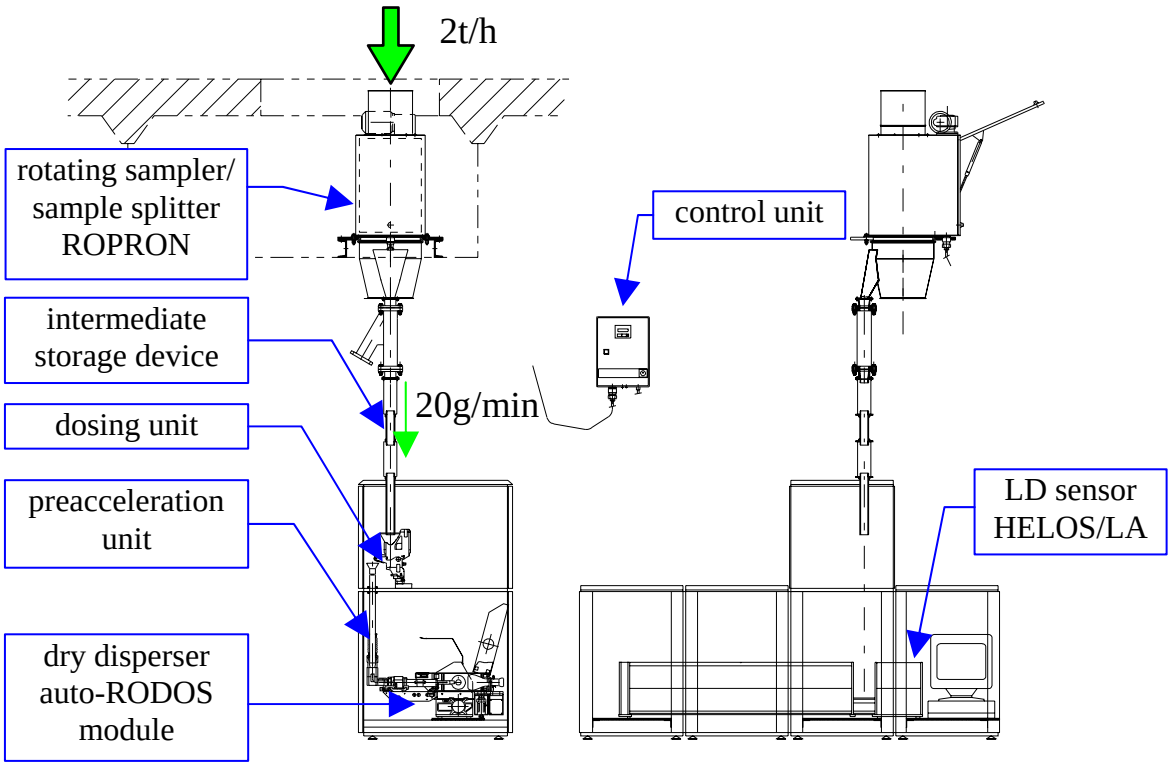

Fig. 9: on-line particle size analysis for coal, including sampling, using dry dispersion in an explosion proof environment (zone 11, dust explosion)

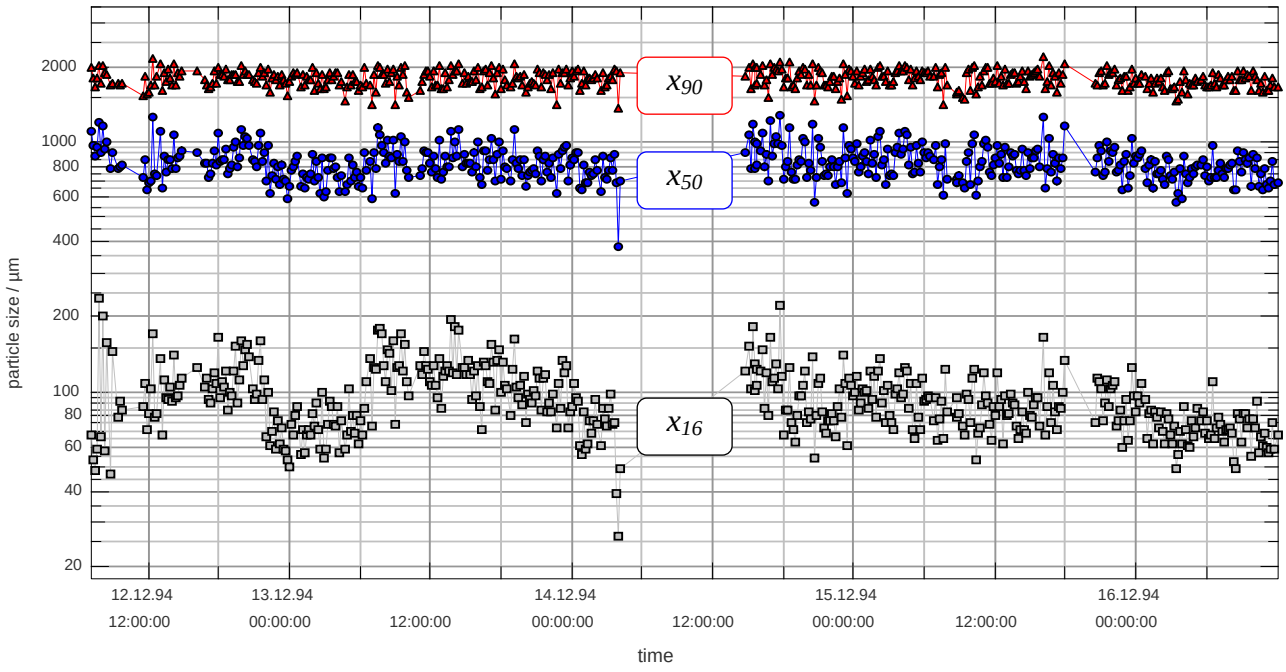

Fig. 10: Trend analysis of the production of coal generated by the new data base orientated PSD information system WINDOX.

As the coal has a very wide size distribution in combination with very coarse particles, a standard dry disperser (auto-RODOS module) with 6mm injector is used in combination with the TOPMICRON technique to achieve a measuring range of 0.5/18.0 to 3500µm. The full system is housed in an IP55 cabinet and adapted to explosion proof requirements. The strong vibration of the floor is reduced by

integrated vibration dampers. The data is transferred to a control room using a 20mA current loop over a distance of 1.5km. Usually a trend analysis as demonstrated in fig. 10 is used for production control.

5 Summary

From the discussion about light sources it would be surprising if pure LD could extend the measuring range to below 0.05µm. On the coarse side, the limit will be terminated more by the market than by the physics. Does LD for x>10mm really make sense?

The recent introduction of SUBMICRON modules has extended the dry dispersion into the SUBMICRON range. So the choice of the dispersing medium can now only rely on the requirement of the sample.

The reproducibility is already far beyond the possibilities of the sample preparation and the dispersing errors. The sensitivity is approaching the limit of single particle counting techniques. But what about system to system comparability and comparability among systems of different suppliers. Standards and standardisation has already become of decisive importance /12/. Pure LD is best prepared for this situation.

Laser diffraction - unlimited ? Naturally not, because every measurement method has its (physical) limitation! But even 25 years after the introduction of LD these limits are still movable and to some extent not even known.

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7 Nomenclature

C_{opt}	optical concentration	r_D	outer radius of a detector element
$d(z)$	opening diameter of fourier optics	s''	imaging distance of a lens
f	focal distance	w	width of the focus point
f'	effective focal distance	w_0	width of the Gaussian intensity distr.
I_0	primary intensity	x	particle diameter
i_n	photo-current of detector element n	$x_{\min}$	minimum particle diameter
$I(\theta)$	angular intensity distribution	$x_{\max}$	maximum particle diameter
$I(r)$	spatial intensity distribution	x_{10}	particle diameter at Q_3=10%
$I(r,x)$	spatial intensity at radius r for size x	x_{16}	particle diameter at Q_3=16%
J_1	Bessel function of 1. order	x_{50}	particle diameter at Q_3=50%
L	intensity vector	x_{84}	particle diameter at Q_3=84%
m	complex refractive index	x_{90}	particle diameter at Q_3=90%
N_{total}	total number of particles	z	working distance
$Q_3(x)$	cumulative volume distribution	θ	scattering angle
r	distance to the centre of detector	λ	wavelength of the light source
r_0	radius of the first minimum	σ	mean standard deviation

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Laser Diffraction - unlimited ?

Within the past 20 years, particle size analysis with laser diffraction (LD) has been subject to rapid development; evaluation methods based on pure physics, such as the well-known Fraunhofer and Mie solutions, have been introduced. The measuring range was extended stepwise from 1 to 200µm to approximately 0.1 to 3500µm. The introduction of dry dispersers has extended the field of application to aerosols. Meanwhile LD has become the dominant technique for optical particle sizing in laboratory (off-line) and production environments as in-line, auto-line and, in combination with sample couplers, on-line applications. Is LD unlimited? This question is discussed in terms of *size range*, *sensitivity* and *reproducibility.*

The possible *size range* is not only restricted by the wavelength of the laser and the transmission limits of the medium. Its extension is mainly due to improvements in the measurement of the angular intensity distribution. Influences from stability and flow dominate on the coarse side of the measuring range and can be overcome e. g. with the TOPMICRON technique. On the fine side, the spatial extension of aerosols and the resulting demand for extended working distances, can be covered only in a parallel laser beam. The recently introduced DRYSUBMICRON technique can satisfy these requirements.

The *sensitivity* limit of LD is approaching that of single particle counting techniques. For medical spray and inhaler applications, 0.1% of optical concentration can be converted to particle size distributions even for time-resolved analyses with sampling intervals of only a few milliseconds. The detectability of small coarse fractions is normally determined by the LD principle itself. By use of an improved beam alignment and the exact measurement of the obscuration of the laser intensity, a precise intensity measurement can be performed resulting in an increased sensitivity.

The *reproducibility* of the sensor, with σ typically much less than 0.5%, is no longer the limiting factor. It is mainly dominated by the reproducibility of sampling, sample splitting, dispersion and the contamination of the optical path. The latter can be improved by the control of flow, especially for in-line and inhaler applications.