W. Witt (Speaker), S. Röthele

Sympatec GmbH, System-Partikel-Technik, Burgstätterstr. 6,
D – 38678 Clausthal-Zellerfeld, Germany

In-line Laser Diffraction with Innovative Sampling

1 Introduction

Within the past twenty years laser diffraction (LD) has become the dominating technique in particle sizing [1]. Beside the standard off-line applications in laboratories, several attempts have been made to transfer this technique into the production plant. *On-line* particle size analysers (PSAs) usually combine a standard off-line PSA with sample preparation stages which adapt the analyser to the requirements of the specific process. Some on-line applications have been reported e.g. in [1, 2]. The large flexibility of this method is now extended by the more compact design of the new *in-line* equipment.

In the simplest set-up the whole production flow passes the measuring zone of the PSA. Here the principle of LD limits the maximum throughput to a few kilograms per hour sufficient to monitor the particle size distribution (PSD) of e.g. some pharmaceutical applications [3]. The typically larger throughputs of several kilograms to several tons per hour require the operation in partial streams in bypass arrangements [4]. Thus representative sampling becomes of decisive importance.

In this paper a new in-line PSA is presented that combines for the first time representative sampling, effective dry dispersion and particle size analysis by means of LD in a single device integrated in a pipe of the production process.

2 Concept

The process stages in a production plant are usually connected by pipes. Thus for optimum flexibility an in-line PSA should be interfaced to a process pipe. Representative sampling within a production pipe requires that the complete cross-section of the pipe contributes an equally weighted amount to the partial stream being analysed and that this partial stream is taken isokinetically. The use of the LD principle adds three more prerequisites: Firstly, the optical concentration C_{opt} must be within certain limits $C_{min}<C_{opt}<C_{max}$. C_{min} is defined by the signal to noise ratio and the statistical relevance of the PSD. C_{max} is related to the sensitivity of the detector

and how multiple scattering is treated by the evaluation process. Second, the particles have to be properly dispersed if the size *x* of the primary particles is to be monitored. Third, periodical measurements of the background signal are required in order to compensate the effects of pollution and imperfections of optical components, i.e. one must be able to interrupt the partial flow through the LD stage.

2.1 Sampling

For an in-line PSA continuous sampling is required. Although proper sampling is a precondition for nearly all types of analyses adequate devices are almost unavailable: most samplers either operate discontinuously or more or less non-representatively. The exception is a sampler demonstrated in Fig. 1a. Here a sampling sector field rotates around the centre line of the pipe with a angular revolution speed ω. The partial flow $\dot{m}$ is proportional to the sector angle α, the mass flux Θ and the square of the diameter of the process pipe *D* as seen from equation (1). As Θ usually cannot exceed specific limits, large throughputs require large *D* resulting in very large partial flows. So for LD this principle has to be cascaded in general [5]. Isokinetic operation is possible, but currently not established.

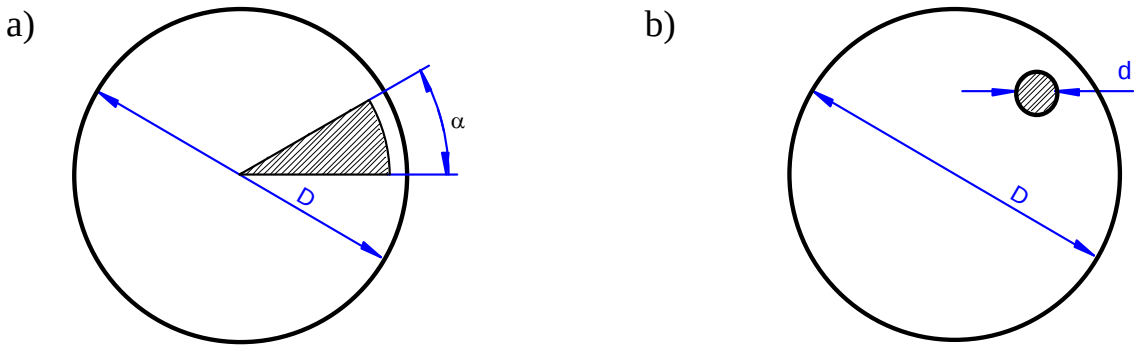

Fig. 1 Principles of continuous samplers: a) sector field, b) sampling pipe

$$\dot{m} = \frac{\alpha}{2\pi}\dot{M} = \frac{\alpha}{2\pi}\Theta\frac{D^2}{4}\pi = \frac{\alpha}{8}\Theta D^2; \qquad \dot{m} \approx D^2 \tag{1}$$

$$\dot{m} = \frac{\frac{d^2}{4}\pi}{\frac{D^2}{4}\pi}\dot{M} = \frac{d^2}{D^2}\Theta\frac{D^2}{4}\pi = \frac{\pi}{4}\Theta d^2; \qquad \dot{m} \approx d^2 \tag{2}$$

A simple sampling pipe of diameter *d* exhibits some advantages. Following equation (2) $\dot{m}$ is proportional to the square of *d* and thus for constant Θ independent of *D*.

The square dependence on *d* simplifies the adaptation to the application. Some in-line PSAs use this sampling method with a static sampling pipe, although the sampling is non-representative.

For representative sampling, the cross-section has to be scanned representatively by the sampling pipe. A linear spiral line is used in Fig. 2a. As equal areas must be covered in equal times the velocity *v* of the opening has to be adapted to the distance of the centre *r* by *1/r* creating a singularity in the centre. An improved solution is displayed in Fig. 2b. Here the scanning starts at the inner wall of the pipe, approaches the centre and returns back to the starting position. The velocity profile along the track is more complicated but very smoothly developed avoiding fast accelerations.

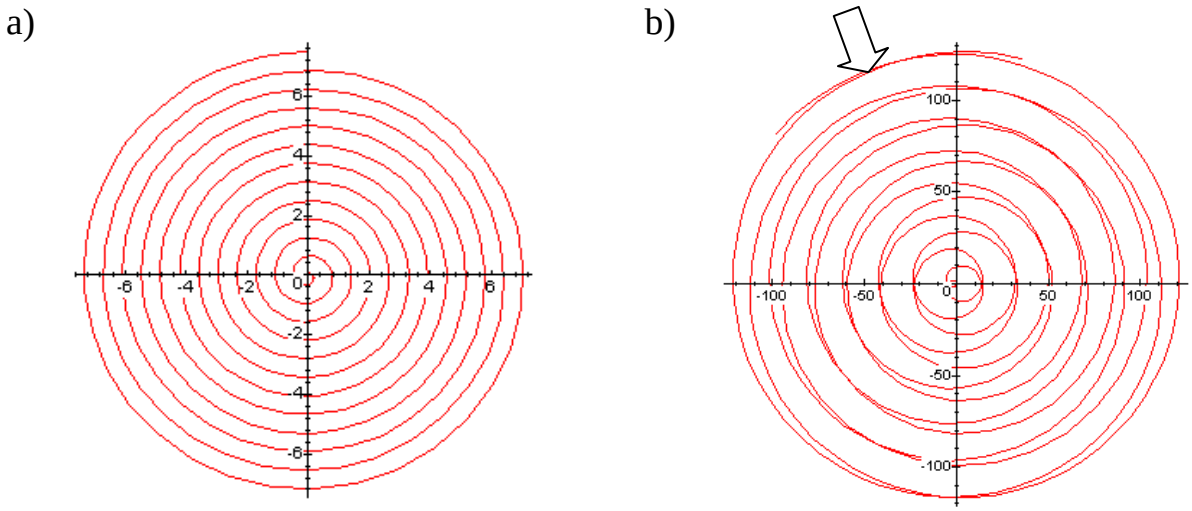

Fig. 2 Scanning methods: a) spiral line, b) realised sampling curve

The scanning of Fig. 2b allows for a shielded parking position at the inner wall of the process pipe (arrow) protecting the opening from particles in the process flow. In this position the partial flow is switched off. So the LD system can perform a reference reading without the help of additional valves, while the process flow is still present.

Fig. 3 demonstrates the preferred realisation of the sampling pipe. It is originated at the centre line of the process and is directed against the process flow at a small angle θ. The change of the projected area of the sample inlet seen from the process flow can be compensated for by adding a *cos(θ)*-term to the velocity *v*.

This arrangement has several advantages: Firstly, the partial flow is kept inside the process pipe, so any pressure inside the pipe does not effect the sampling. Space saving 90° elbows are avoided, which are generally strongly affected by wear. Secondly, the motion can be performed without moving gaskets. Bellows can be used instead, allowing for operation in hazardous areas. The outer bellow can be protected by a shield. Thirdly, only small masses have to be moved reducing the required power of the drive unit. Fourthly, all parts in contact to the process flow are of simple design and can be manufactured out of hardened materials, e.g. ceramics for extended lifetimes.

The adaptation to the mass flow of the production pipe can be performed by variation of the opening diameter *d* of an inlet cap. In addition the inlet acts as a ‘security sieve’, if diameter of *d* is chosen to be smaller than the width of all subsequent stages. The inlet can be cleaned by a scraper connected to the parking position.

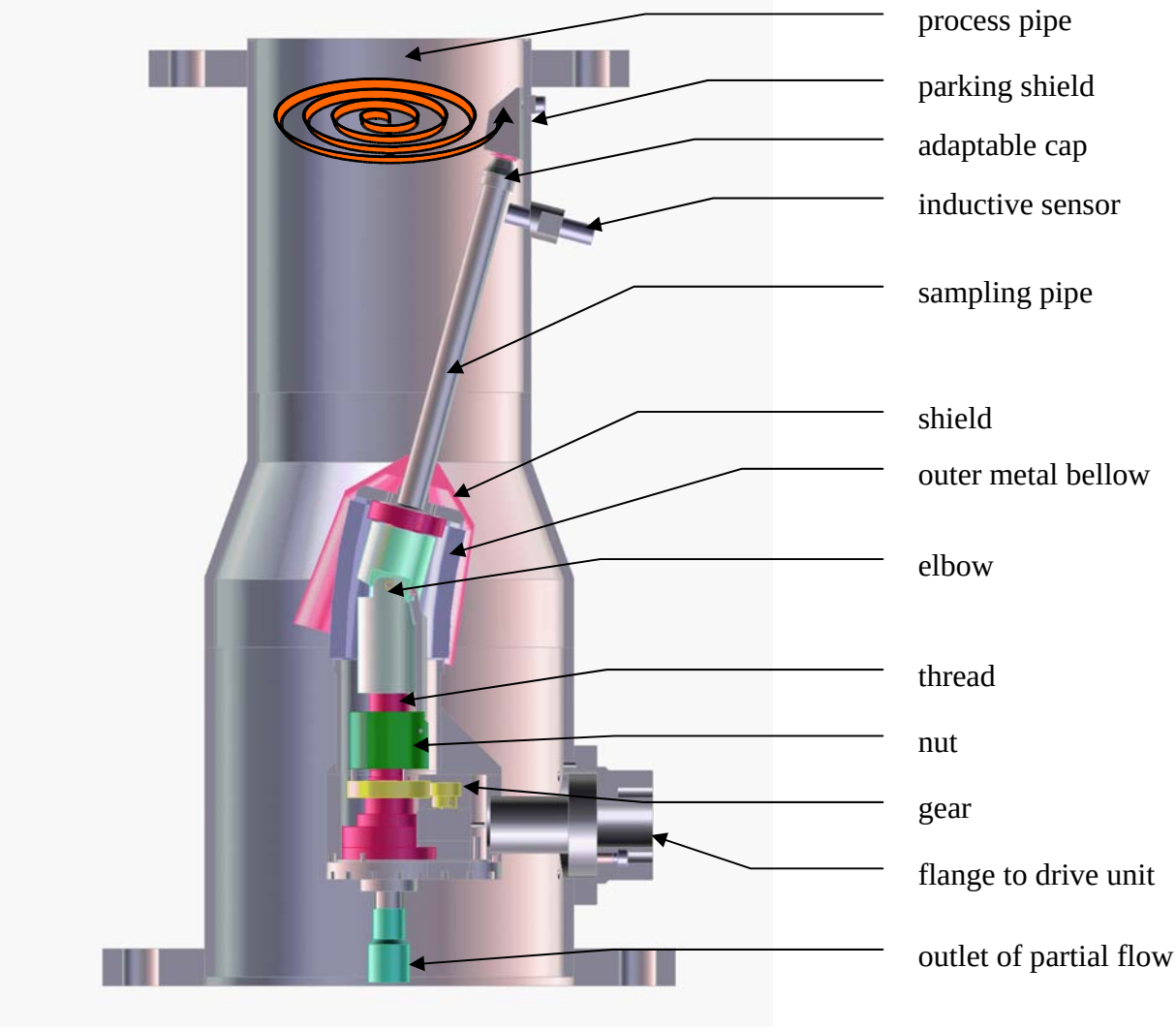

Fig. 3 Representative in-line sampler

At first sight the drive unit appears to be complicated. Several solutions e.g. the use of a Kardanic bearing for the sampling pipe agitated with x-y drives and synchronised position controllers have been investigated. The preferred solution is deduced from a simple rotational drive unit, that spins the sampling pipe along the centre axis, while a nut-tread-lever-elbow-construction varies the angle θ. Fig. 4 demonstrates the principle.

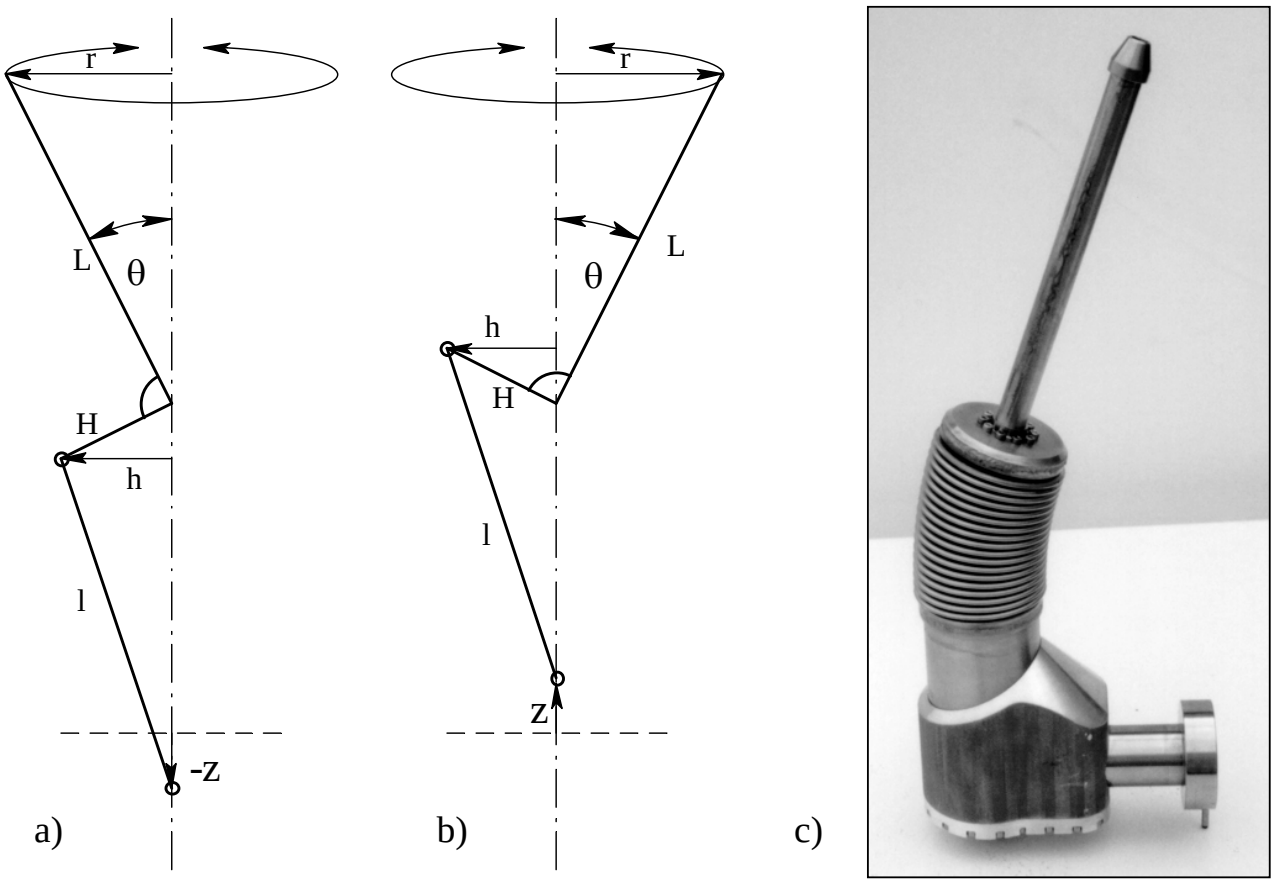

Fig. 4 Principle of the drive mechanism. a) nut in position –z, b) nut in position z; c) flange – on sampling unit with in-let cap, sampling pipe, metal bellow and integrated drive unit (shied removed). The unit is driven by a small external DC-motor through the flange.

The rotation of the whole unit along the centre line moves the nut along a tread in z direction. The connected lever l changes via elbow H-L the angle θ creating the desired spiral line.

$$r(z) = -\frac{1}{2}\frac{\left(-\sqrt{l^2 - H^2}\,z + z^2\right)L}{\left(-\sqrt{l^2 - H^2} + z\right)H} \tag{3}$$

The dependence of the radius r with respect to the position of the nut z is described by equation 3, which shows a nearly linear behaviour for typical dimensions. The

remaining non-linearity can be compensated for by adding another term to the velocity of the motor.

2.2 Isokinetic sampling

For isokinetic sampling the velocity of the particles entering the sampling pipe must be equal to the velocity in the process pipe [6]. The velocity inside the sampling pipe is adapted by a controllable flow pump, which directly feeds the subsequent dispersing stage. As precise velocity measurements at high particle concentrations in a process pipe are difficult and the equipment is usually not very robust, an empirical approach was chosen. The dependency of the velocity of the inlet was measured as function of the primary pressure p of the flow pump. With the assumption, the velocity profile v_P inside the production pipe is only a function of the radius r and constant in time, p was used to adapt $v_P(r)$ by the micro-controller accordingly.

2.3 Dispersion

The dispersion of dry particles is performed by a standard dispersing line as known from [7], which is mounted along the centre line of the process pipe (see Fig. 5).

The particles are accelerated in a turbulent flow field and perform particle-to-particle and particle-to-wall collisions or are separated by centrifugal forces, resulting from velocity gradients in the turbulent flow. This dispersing technique is suitable down to the submicron range [8]. For such device lifetimes of typically 100.000 measurements of 5g sample of Portland Cement PZ35 are nowadays standard [9]. The dispersing line is not affected by wear during static periods as the parking position of the sampler switches off the partial flow. So the number of measurements is equivalent to about two years of operation with six measurements per hour. This time can even be extended by speeding up v, i.e. shortening the sampling time.

2.4 Particle sizing

Finally, the dispersed aerosol enters the measuring zone of the LD instrument. To avoid pollution of the optical components, the measuring zone is established as a second injector, with a sheath flow used for focussing the aerosols along the centre line. An integrated rising system protects the built-in dust protection windows from spray particles. To simplify the serviceability, both light source and detector unit are connected via bayonet fastenings with the process pipe. Both units can be easily

removed in situ, with the sampler in parking position allowing for easy access to the dust protection windows.

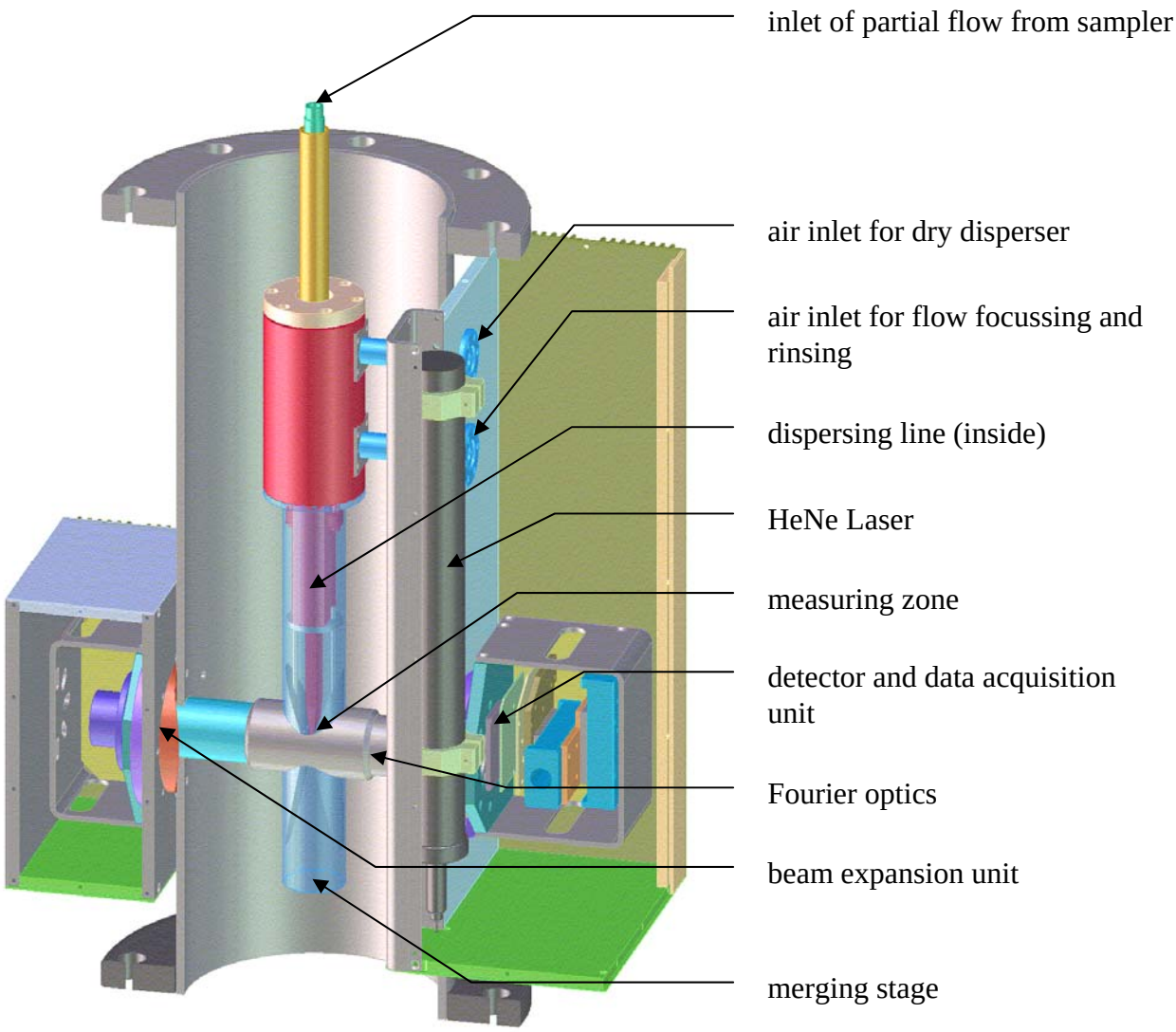

Fig. 5 Cross section of the PSA with built-in dry disperser

The components of the LD instrument are identical to that of a standard off-line series of LD PSAs [1, 10]. The light source consists of a HeNe laser with 5mW of output power, a spatial filtering using a monomode fibre and a beam expansion to 2.2 mm diameter ($1/e^2$ intensity points). On the detector side a Fourier optics, a subsequent multi-element detector, an auto-centering unit, the data acquisition and a serial communication to an external personal computer (PC) by a high speed fibre optical link are provided. An integrated RS485 interface synchronises the PSA with the sampler. The standard software calculates the PSDs and allows for automatic operation of several in-line and off-line PSAs in a distributed network [11].

2.5 Merging stage

Finally the partial flow is merged with the process flow by the merging stage. Its standard realisation is a simple pipe that directs the particles along the centre line of the production pipe. So the fast particles of the partial flow hit the walls of the process pipe under very small angles avoiding wear. Alternatively the outlet of the merging stage can be closed by a valve allowing for complete separation of the PSA from the process. Wet cleaning of the production pipes as well as the manual cleaning of the dust protection windows with the process running is possible.

2.6 Fields of application

The field of application is mainly defined by the optical concentration C_{opt} that should be between about 1% and 60% for this type of PSA [3]. C_{opt} is defined by equation 5. It results from the primary mass flow rate $\dot{M}$, the PSD, the extinction coefficient κ, the specific density ρ and the velocity of the particles in the measuring zone v and the spatial extension of the aerosol perpendicular to the illuminating beam b.

$$C_{opt} := \frac{(I_0 - I_P)}{I_0} = 1 - \frac{I_P}{I_0} \tag{4}$$

For monodisperse particles C_{opt} can be simple expressed by equation (5).

$$C_{opt}\{monodispers\} = \frac{3}{2}\frac{\kappa}{bv\rho}\left(\frac{d}{D}\right)^2 \frac{\dot{M}}{x_{50}} \tag{5}$$

PSDs generally increase the optical concentration. For logarithmic normal distributed spherical PSDs a factor $F(\sigma)$ has to be added, that can be numerically calculated.

σ	lg(x(84%)) -lg(x(16%)	lg(x(1%))- lg(x(99%)	F(σ)
0.05	0.1	0.24	1.005
0.10	0.2	0.48	1.027
0.15	0.3	0.72	1.061
0.20	0.4	0.96	1.111
0.25	0.5	1.20	1.180
0.30	0.6	1.44	1.269
0.35	0.7	1.68	1.384
0.40	0.8	1.92	1.528
0.45	0.9	2.16	1.710
0.50	1	2.40	1.940

σ	lg(x(84%)) -lg(x(16%)	lg(x(1%))- lg(x(99%)	F(σ)
0.55	1.1	2.64	2.229
0.60	1.2	2.88	2.593
0.65	1.3	3.12	3.064
0.70	1.4	3.36	3.661
0.75	1.5	3.60	4.443
0.80	1.6	3.84	5.448
0.85	1.7	4.08	6.797
0.90	1.8	4.32	8.566
.095	1.9	4.56	10.91
1.00	2	4.80	14.16

Table 1 Dependency of the correction factor F(σ) for logarithmic normal PSDs of given σ. Extreme PSDs with σ > 0.65 cannot be measured by LD.

Table 1 demonstrates, that the correction is close to unity for narrow size distributions. The extinction coefficient κ can be calculated for spherical particles using Mie theory [12]. For a HeNe laser with λ=632.8nm and particles larger than 1µm κ is usually oscillating between the values 2 and 3. Equation 6 can be used to investigate the usability of the presented in-line PSA for various applications.

$$C_{opt}\{1\,dekade;2\,dekades\} = \frac{3}{2}\frac{\kappa}{bv\rho}\left(\frac{d}{D}\right)^2 \frac{\dot{M}}{x_{50}} \cdot F(\sigma) = \frac{3}{2}\frac{\kappa}{bv\rho}\left(\frac{d}{D}\right)^2 \frac{\dot{M}}{x_{50}} \cdot \{1.12;\ 2.45\} \quad (6)$$

Two examples are given. The optimum operating conditions are given between 5% < C_{opt} < 30% allowing for sufficient safety space at both end, which may be needed by fluctuations in the partial flow. Due to the linear relationship, the diagrams can simply be scaled for other throughputs, particle sizes and densities.

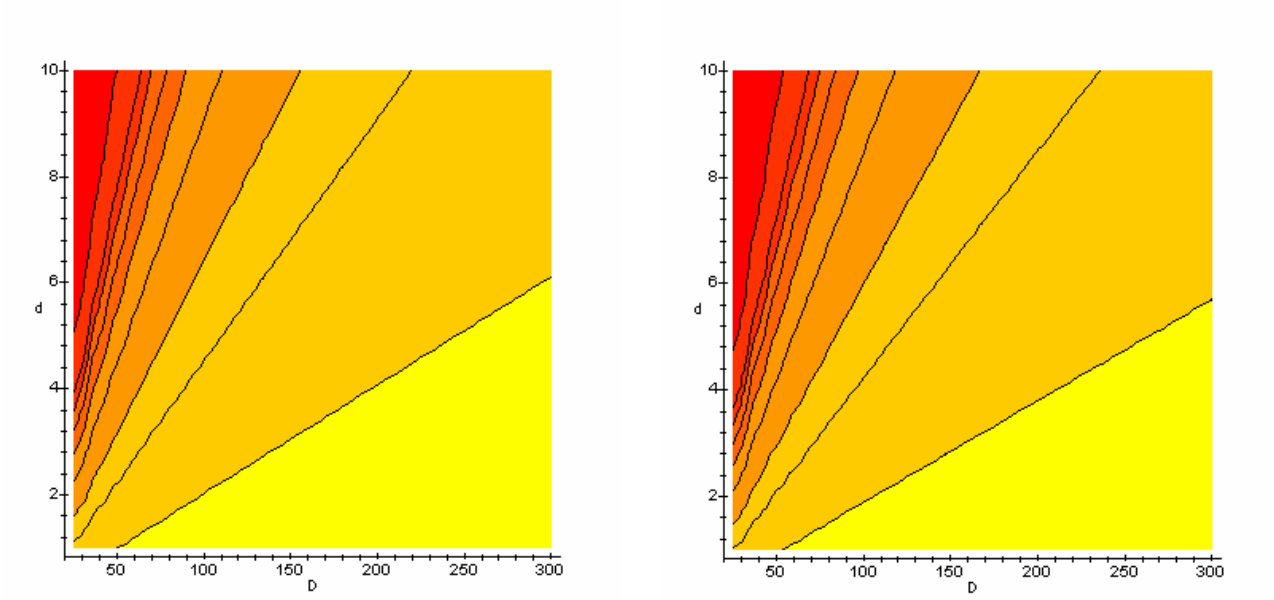

Fig. 6 Opening diameter *d* as function of process pipe diameter *D*,
parameter C_{opt}: = (right), 1%, 5%, 10%, 20%, 30%, 40%, 50%, 60%, 100%, (left)
a) $\dot{M}$ =10kg/h, x_{50}=1µm, b=15mm, v=30m/s, ρ=1g/cm³, F=1.3, κ=2;
b) $\dot{M}$ =1000kg/h, x_{50}=100µm, b=15mm, v=30m/s, ρ=1g/cm³, F=1.3, κ=2.3

From Fig. 6 it can be seen, that a typical pharmaceutical application with D ≅ 50mm results in meaningful opening diameters *d*, as well as an industrial application with throughputs of several tons per hour. It should be mentioned, that the velocity of the particles *v*, i.e. the beam focussing injector can be used in addition to *d* to adjust C_{opt}. Variation of *d* from 2mm to 10mm and *v* from 10m/s to 60m/s produces an adjustment ratio of 150:1.

3 Apparatus

Fig. 7 shows the prototype of the complete in-line PSA ready to adapt to a standard process pipe of 150mm inner diameter. The diameter widens to 200mm to avoid a pressure loss. The total height of the unit is 1200mm.

Two measuring ranges are currently available: R3 from 0.5/0.9 to 175µm and R4 from 0.5/1.8 to 350µm. The pressure range of the process pipe is limited by the bellows and dust protection windows to 10 bar max. The temperature must not exceed 100°C permanently. For operation in hazardous areas (e.g. E(x)-zone 10 or 11) the control boxes are pressurised (E(x)-p).

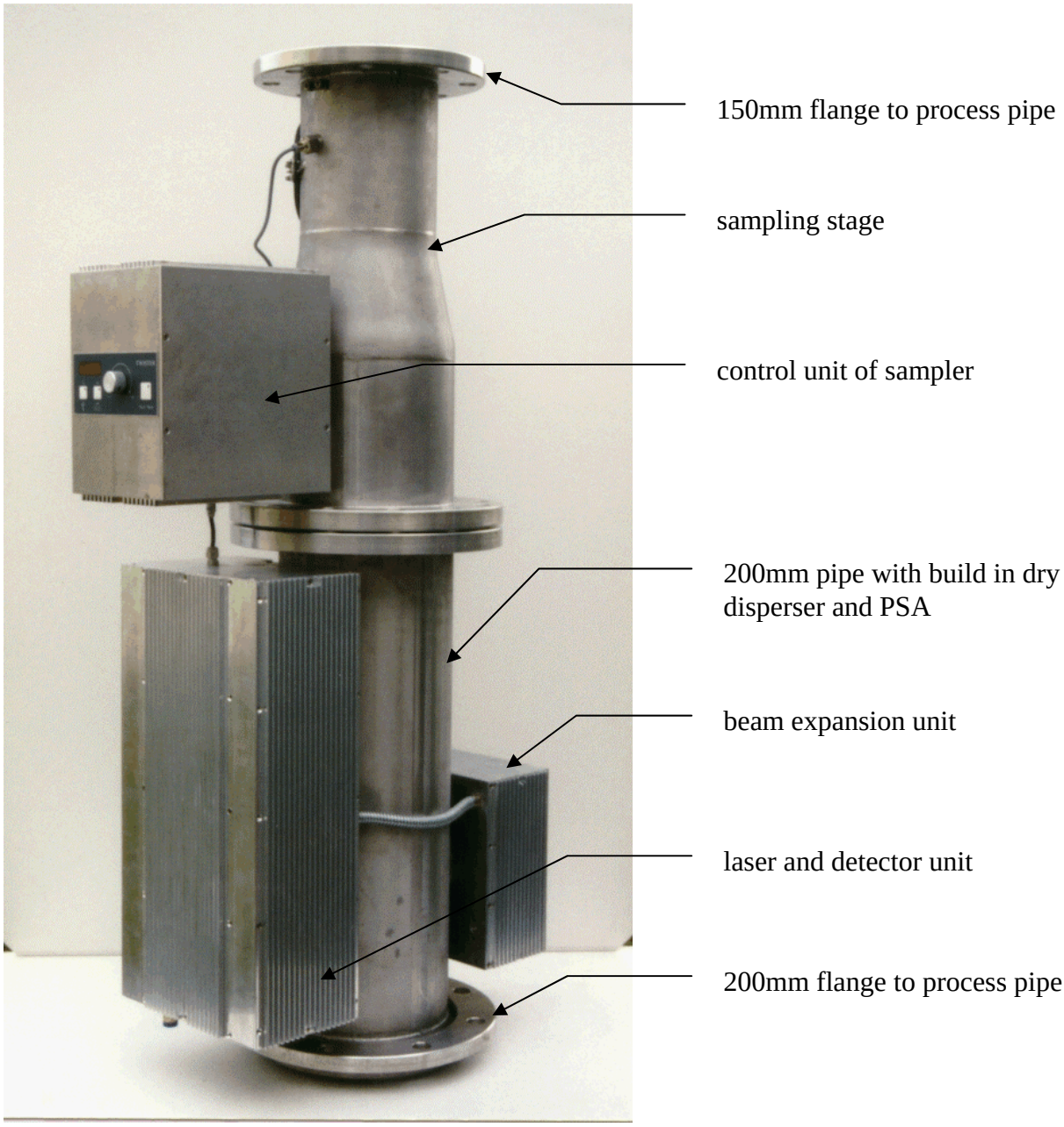

Fig. 7 Side view of the in-line PSA with preceding representative sampler

4 Results

The in-line PSA was tested under process conditions with several materials. Fig. 8 shows a comparison of the in-line PSA with a standard off-line PSA (HELOS/KF) using the measuring range R3 on both units. The test samples used were silicon carbide reference materials labelled as SiC P600 and SiC F1200 which were carefully split using a rotating riffler.

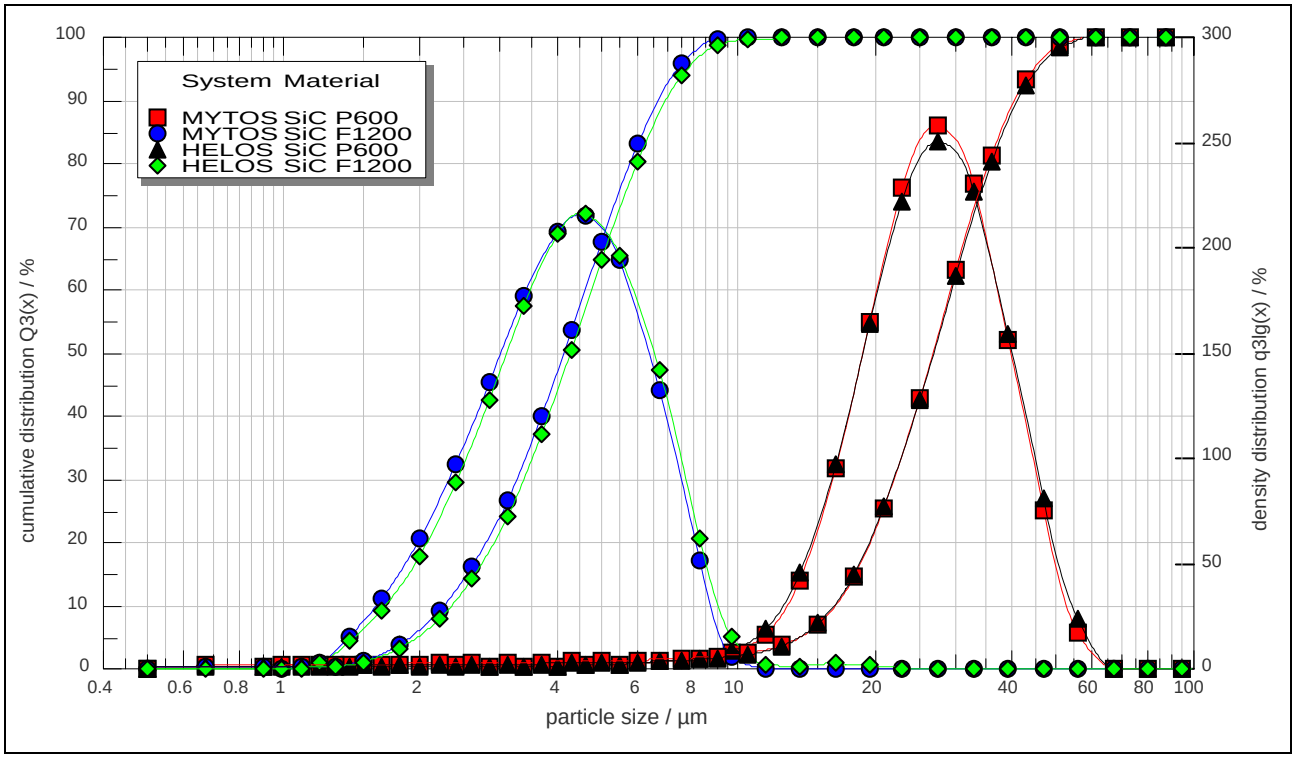

Fig. 8 Comparison of the in-line PSA with the results of a standard off-line PSA (HELOS/KF) using two different reference materials.

Fig. 8 shows an excellent agreement between both units. The differences in x(Q3) are less than 2% for all size classes and less than 1% for the coarser material. The larger differences of the fine material are probably due to an imperfect optical model of the very thick dust protection windows (10 mm).

First results of a series of measurements are displayed in Fig. 9. A milled quartz material was aplied in a loop to the in-line PSA.The measurement was performed periodically every 6 minutes automatically. Although the test loop and especially the sample transport did not work perfectly (as seen in the varying optical concentration), the trend plots of the x_{10}, x_{50} and x_{90} values are nearly stable over 6 hours. The flow focussing device in combination with the rinsing system kept the dust protection windows free of pollution.

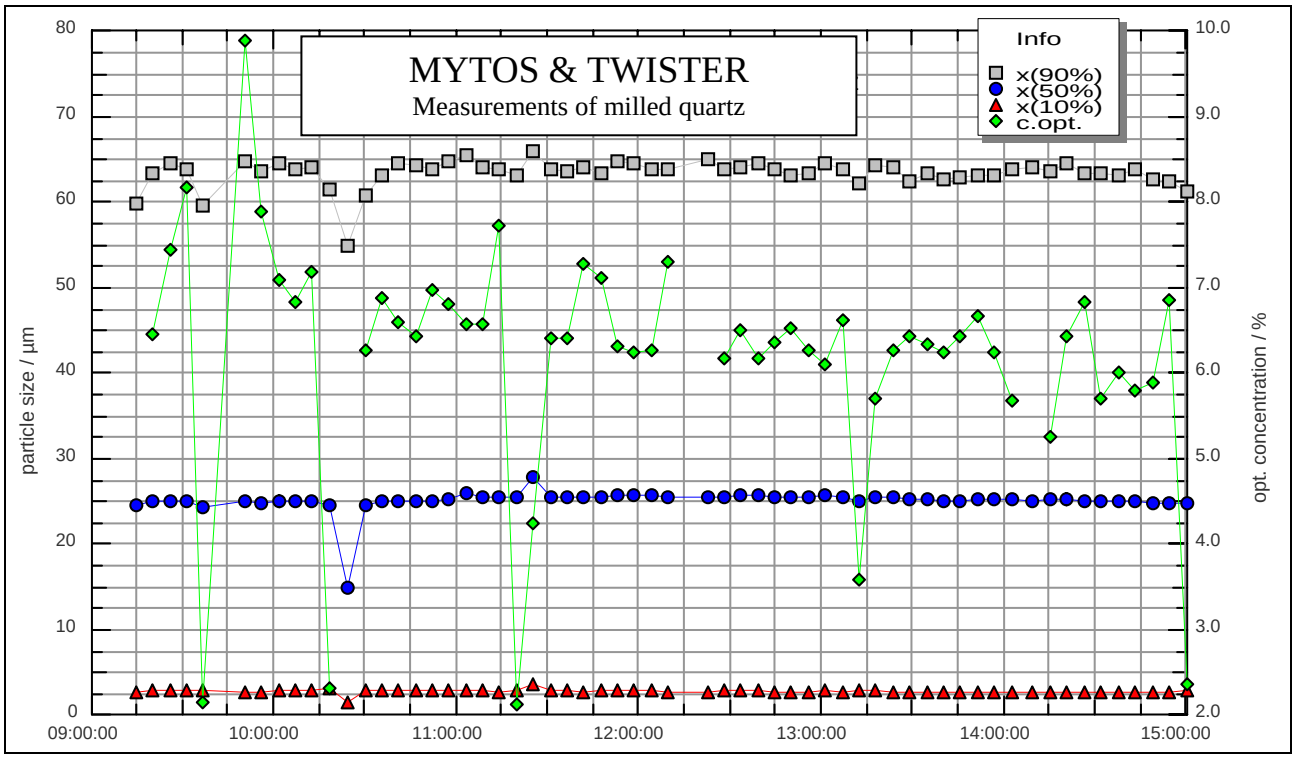

Fig. 9 Trend analysis of subsequent measurements of quartz in a test loop with the in-line PSA MYTOS & TWISTER.

5 Conclusions

For the first time, the presented in-line PSA integrates an innovative continuous and representative sampler, an effective and established dry dispersion unit and a particle size analyser based on laser diffraction inside a standard process pipe. All major components are cascaded along the centre line of the process pipe, thus the presented in-line PSA is effectively an ‘in-pipe’ solution.

The integrated sampler scans the full cross section of the pipe isokinetically and allows for operation of the in-line PSA with static or moving media in any orientation. Thus applications in fall shafts as well as in pneumatic conveying systems are possible.

The built-in dry disperser is well established and has proven it usability down to the submicron range. The parking position of the sampler prevents the measuring system from wear in idle periods between subsequent measurements and allow for reference readings prior to every measurement.

The results obtained from the in-line PSA analyser are identical to those measured by a standard off-line PSA.

6 Outlook

The sampling principle is nearly ideal for scaling up and down the device, as primarily only the length of the sampling pipe has to be adapted to the process pipe diameters.

Further more, the presented principle is not restricted to dry applications only. The parking position can be modified to a supply unit of clear fluids. For wet systems the dispersing line can be replaced by a static mixer with optionally integrated ultrasonic transducers while the flow pump can be used for dilution. Thus measuring of concentrated suspensions will become possible as well.

The sampler can also be followed by a PSA based on ultrasonic extinction and a subsequent pump. In this configuration highly concentrated suspensions and emulsions can be measured without dilution.

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8 Nomenclature

b	minimum(the width of the measuring zone, the outlet of the dispersing line)	v	velocity of the sampling pipe
		v_P	velocity of the particles
C_{opt}	optical concentration	x	particle diameter
C_{min}	minimum optical concentration	x_{10}	particle diameter to which 10 % of the cumulated undersize $Q_3(x)$ corresponds
C_{max}	maximum optical concentration		
d	opening diameter of the sample pipe	x_{50}	particle diameter to which 50 % of the cumulated undersize $Q_3(x)$ corresponds
D	diameter of the process pipe		
F	correction factor for optical concentration of logarithmic normal size distributions	x_{90}	particle diameter to which 90 % of the cumulated undersize $Q_3(x)$ corresponds
H	length of the elbow	z	variation of the nut along the centre line of the process pipe
l	length of the tilting level		
L	length of the sampling pipe	θ	angle of the sampling pipe with respect to the centre line of the production pipe
$\dot{m}$	partial mass flow rate		
$\dot{M}$	process mass flow rate	Θ	mass fluxκ optical extinction coefficient
r	distance of the opening of the sampling pipe perpendicular to the centre line of the process pipe		
		ρ	specific mass density
		ω	angular speed