

The Application of Laser Diffraction Technology

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0 Introduction

The development of laser diffraction technology has been a story of rapid evolution during the past two decades. During the interim, this modern analytical technique for the fast determination of particle size distributions has become the most important method in the field of particle measuring engineering world-wide.

At present, laser diffraction is on the threshold to becoming the dominant standard analytical technique for off-line applications in all fields and simultaneously offers for the first time realistic chances of fulfilling expectations for on-line application.

1 Fundamentals

As a rule, diffraction spectrometers employ an optical arrangement such as that illustrated in figure 1. The diffraction patterns are generated by the particles introduced into the laser beam in the focal plane of the lens installed downstream and detected by a special sensor. The evaluation is based on the Fredholm integral equation, which describes the distribution of intensity, $I(r)$, of a particle collective by means of its number density distribution, $q_0(x)$.

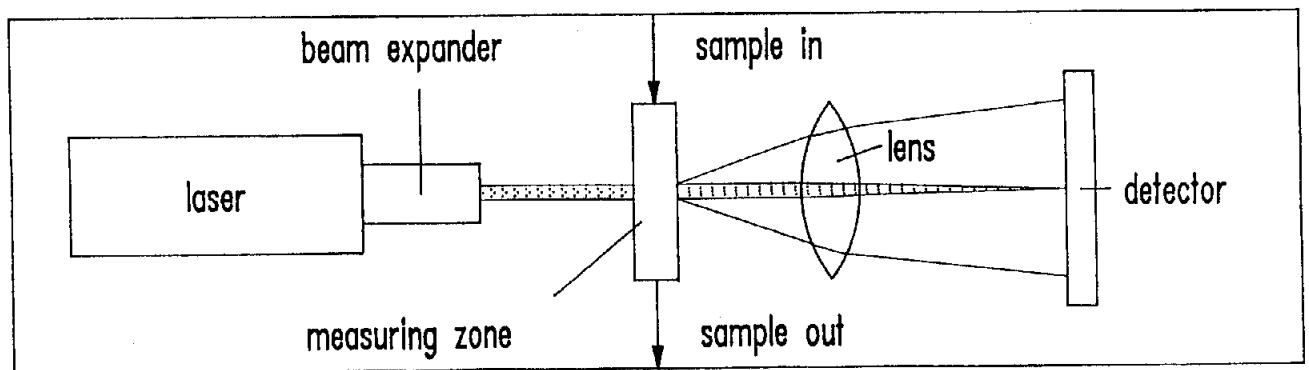


Fig. 1: Optical arrangement for the generation of diffraction patterns

A detailed presentation of the fundamentals is provided by HEUER and LESCHONSKI [1], and the methods of solution are given by RÖTHELE et al. [2].

2 Position in particle measuring technique

At the end of the present century, laser diffraction will have attained the summit of a development which has resulted in enormous progress with respect to the measuring range, Δx , covered in conformance with the method, the finest attainable particle size, x_{\min} , and the attainable analytical time, t_A . Although a comparison of methods in closed form is feasible only within certain limits, an attempt is made to describe the state achieved with the use of appropriate parameters.

The comparison is thereby restricted to sieving methods, sedimentation techniques, and laser diffraction technology. The development during the past thirty years is significant: By means of x-ray sedimentation, the relative measuring range interval is first doubled, with a simultaneous, substantial increase in the speed of analysis and initial penetration into the submicron range. If the following vital criteria, measuring range, Δx [μm], time of analysis, t_A [s], and smallest particle size, x_{\min} [μm], are compiled in correspondence with their effect on performance as attributes for comparison, the dynamic measuring range rate, Ω , referred to the finest particle size, is obtained:

$$\Omega = \frac{(\Delta x/x_{\min})}{x_{\min} \cdot t_A} \quad [(\mu\text{m} \cdot \text{s})^{-1}],$$

with

Δx = measuring range [μm]
 t_A = time of analysis [s]
 x_{\min} = minimum particle size [μm]

Until midcentury, the progress was concentrated on the time of analysis and the conquest of the measuring ranges down to 1 μm by various methods.

Initially, sedimentation was regarded as the ideal supplement to sieving in the fine-particle range. In accordance with the classical view, a given method was capable of spanning only two powers of ten. The speed of analysis remained low; as a rule, an analysis required a day's work. In the case of sedimentation analysis including the 1 μm range, it could even become the biblical creation cycle of one week.

If the measuring range rate, Ω , is employed as a measure for appraisal, it is evident that sieving has not progressed any further for decades. Hence, its evolution can be viewed as complete.

year	principle	dry	wet	x_{min} (μm)	x_{max} (μm)	Δx (μm)	$\Delta x/x_{min}$	t_A (s)	$\Delta x/t_A$ ($\mu m/s$)	Ω (μms) ⁻¹
1900	set of sieves	X		100	10,000	9,900	99	3,600	2.75	$\sim 3 \cdot 10^{-4}$
1925	sedim.balance		X	1	100	99	99	150,000	0.0007	$\sim 7 \cdot 10^{-4}$
1930	pipette		X	1	100	99	99	50,000	0.002	$\sim 2 \cdot 10^{-3}$
1950	wet sieving		X	5	200	195	39	30,000	0.006	$\sim 3 \cdot 10^{-4}$
1960	air jet sieve	X		30	1,000	970	32	10,000	0.1	$\sim 1 \cdot 10^{-4}$
1965	x-ray sedim.		X	0.5	100	99	200	7,200	0.01	$\sim 1 \cdot 10^{-1}$
1972	laser diffr.		X	1	200	199	200	1,500	0.1	$\sim 1 \cdot 10^{-1}$
1985	laser diffr.	X		0.5	1,750	1,750	3,500	100	17.5	$\sim 7 \cdot 10^{+2}$
1987	laser diffr.		X	0.1	100	100	1,000	60	1.7	$\sim 2 \cdot 10^{+2}$
1988	x-ray sedim.		X	0.1	300	300	3,000	2,500	0.12	$\sim 1 \cdot 10^{+1}$
1988	laser diffr.		X	0.1	700	700	17,000	200	3.5	$\sim 4 \cdot 10^{+2}$
1990	laser diffr.	X		0.5	2,625	2,625	5,250	10	262.5	$\sim 1 \cdot 10^{+3}$

Table 1: Parameters for comparison of methods

Sedimentation, in contrast, has undergone considerable further stepwise development all the way to automation of x-ray sedimentation, initially by more than three powers of ten. An additional achievement resulted after conversion to digital techniques of computation; the measuring time thereby persists in the four-digit second range because of the measuring principle.

Nevertheless, the significant development thus initiated additionally acquired the decisive dynamism in the past twenty years with the advent of laser diffraction.

The time of analysis, t_A , was first shortened by one power of ten with respect to x-ray sedimentation for a comparable measuring range interval Δx . The methods are still equivalent as far as the measuring range rate, Ω , is concerned. With the development of dry dispersion, the measuring range interval was then extended by a factor of more than ten, and the time of analysis was simultaneously shortened by the same order of magnitude.

Correspondingly, this achievement is definitely expressed as an innovative measure with respect to the measuring rate range.

Subsequently, by means of the Fraunhofer method, the submicron range was successfully included with laser diffraction. For the last decade of the present century, there are indications of progress into the two-digit second range for suspension analysis, and into the one-digit second range for dry applications as far as the time of analysis is concerned.

3 Application of laser diffraction

Until the present stage, the classification of laser diffraction has been confined to a comparison of performance for the sensor system and the measuring ranges.

However, laser diffraction possesses an additional dimension which must be incorporated into the practical aspects for realizing and describing the full performance capabilities.

Ideally, products manufactured in the dry state should also be analyzed in the dry state, whereas wet products should be analyzed in suspension. This conclusion can be effectively realized if it is imposed as a requirement on the technology. The solution is provided by adapting the laser sensor system with an appropriate dispersing device to match the particular product under investigation in each case.

Flexible, modular analytical systems consisting of a sensor and a dispersing device are the result. With the use of these systems, conclusive information can be obtained on the primary size distribution; it is highly probable that these data are also relevant with respect to the production process.

The concept is characterized by a high degree of product consciousness.

Finally, correct coupling and matching with the production process, with the object of preparing an appropriate sample for analysis, then has to be ensured in the last step.

This technical delineation of the individual functions and the consistent design of instruments in compliance with the associated demands in three consecutive epochs are illustrated in table 2.

epoch	sensor and size ranges	dispersing system and product consciousness	sampling and process coupling
1970	basic application 1 - 200 μ m	suspension period	exclusively via laboratory
1980	extension to submicron and extra-millimeter 0.1 - 2000 μ m	fruitful dry period	coupling with transport systems and samplers
1990	completion of extension 0.1 - 3000 μ m	robotic era	cultivation of sample couplers

Table 2: Epochs of application of laser diffraction

3.1 Launching phase

The seventies may be viewed as the introductory period for basic applications, with a limited measuring range between 1 and 200 μ m. The results attainable from the measurements initially did not require any methodical independence, but were based, for example, on sedimentation analysis, or conformed with distribution types specified in advance.

Figure 2 proves this situation for a reference sample of portland cement supplied by the National Bureau of Standards.

During this epoch, no specific product consciousness was recognizable from the dispersing devices. This phase can be regarded as the age of monostructural suspension applications. No on-line ambitions were yet detectable in sampling and process coupling at this time, either.

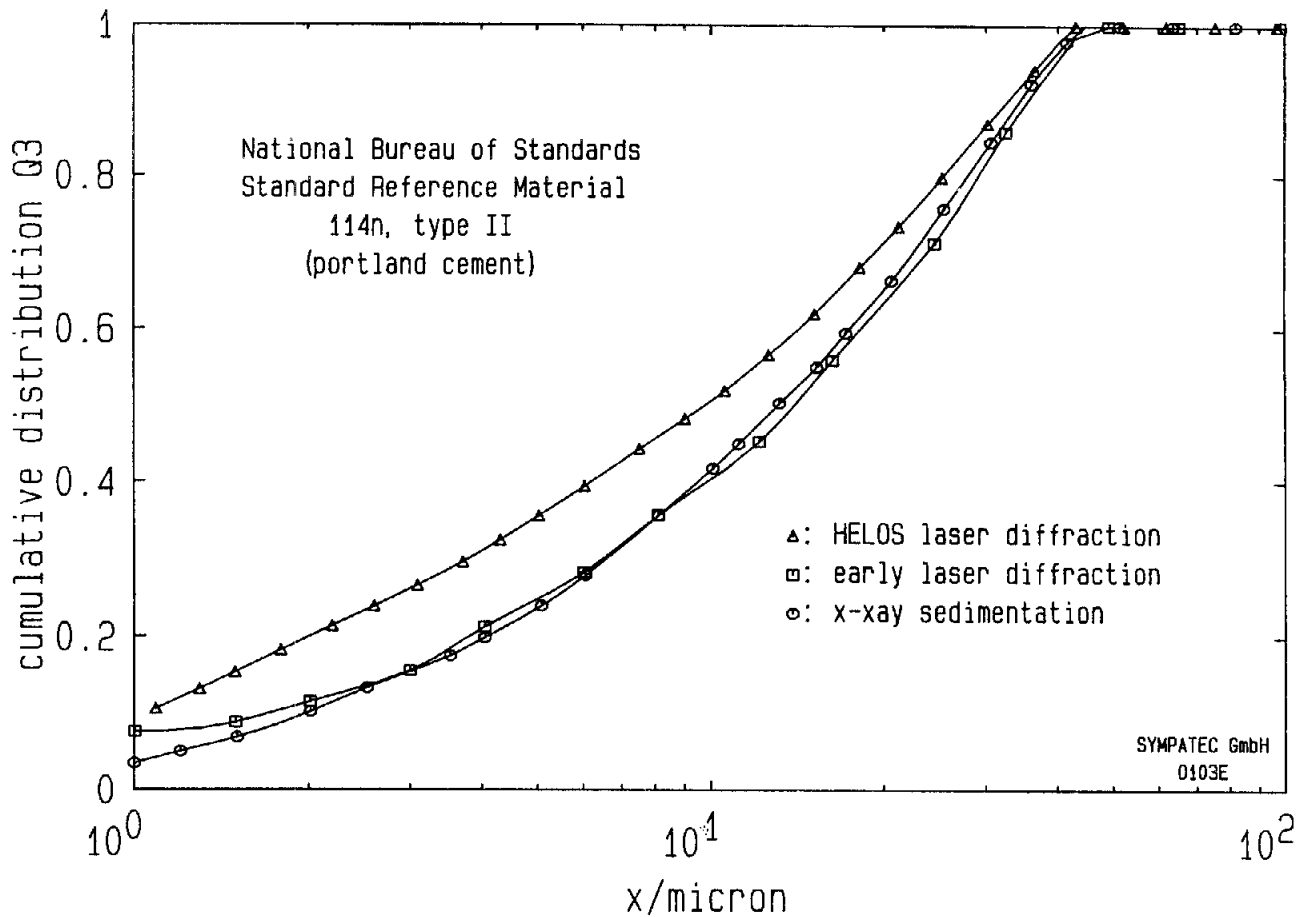


Fig. 2: Comparison of different measuring principles

3.2 Extension of the application limits

During the following decade, the performance characteristics of the sensors were greatly improved, and the limits of application with regard to the detectable particle sizes were extended by one power of ten upward and downward to

$$0.1\mu\text{m} < x < 2000\mu\text{m}.$$

At the same time, the independence of laser diffraction results has been firmly established in comparison with other methods. This was accomplished with the development of new mathematical techniques and the user-oriented application of the Fraunhofer scattering function to values of the Mie-parameter, α , approaches which had previously been viewed as inadmissible.

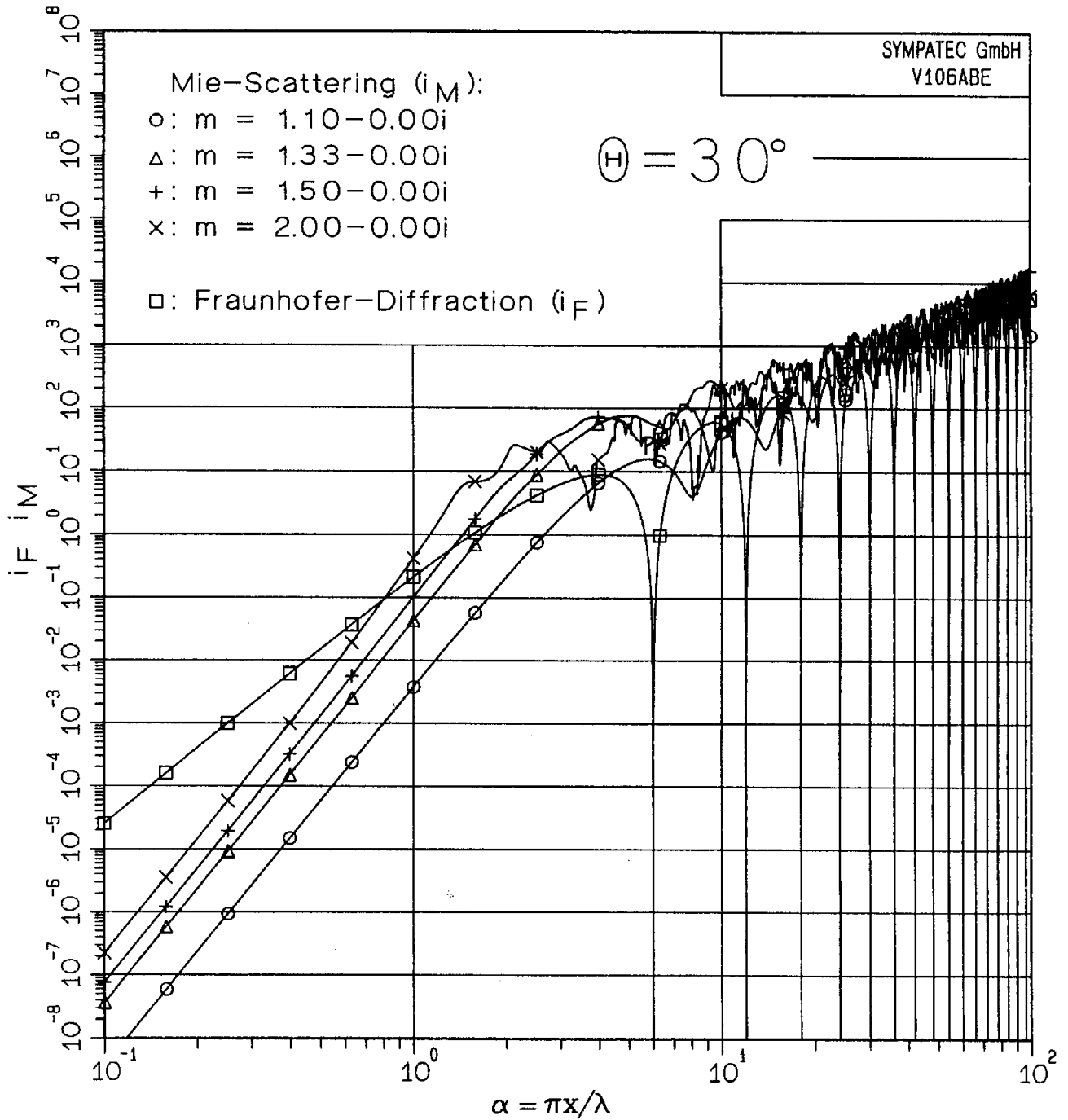


Fig. 3: Comparison of the Fraunhofer scattering function and the Mie scattering function, referred to [2].

The direct comparison shown in figure 3 indicates that in the decisive limiting range,

$$1 < \alpha < 10,$$

the Fraunhofer scattering function passes through the middle of the range which is spanned by the varied indices of refraction yielded by the Mie solution.

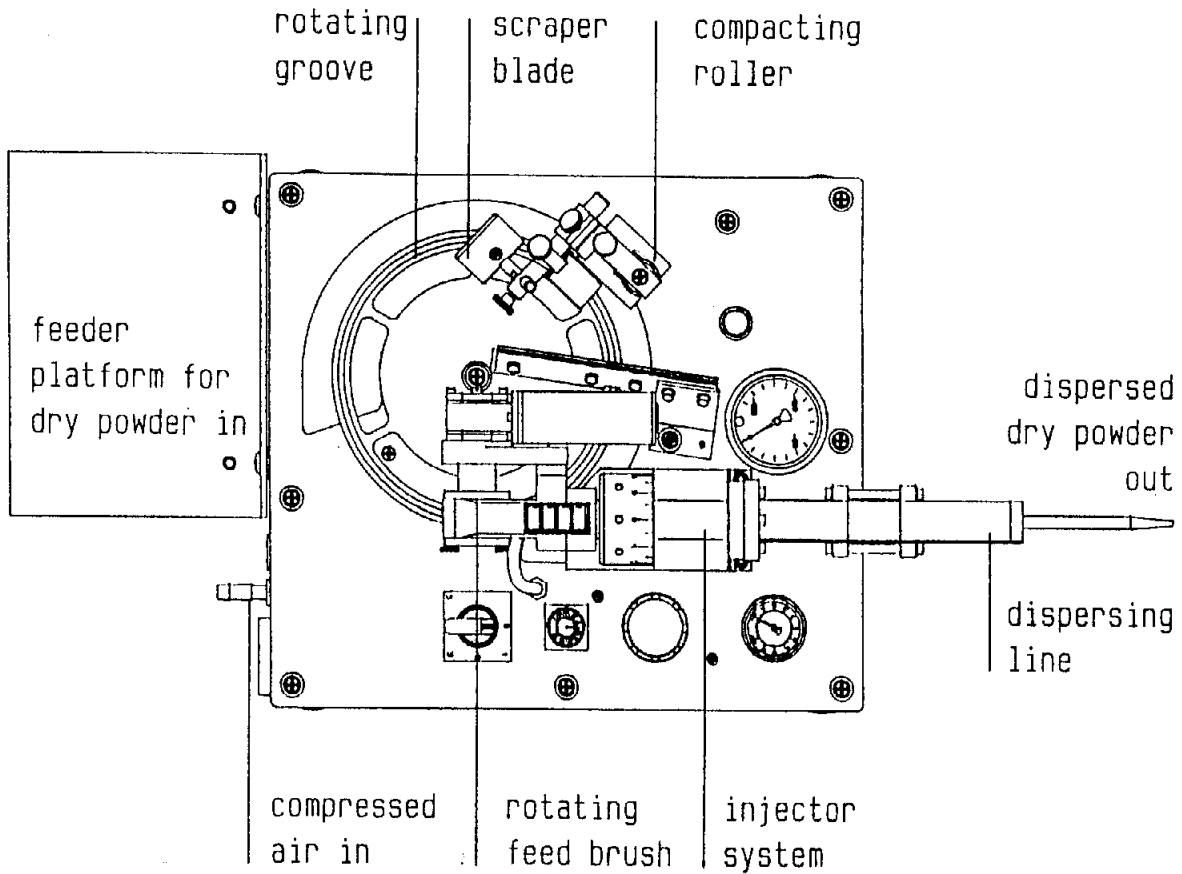


Fig. 4: Feeding and dispersing unit RODOS [3]

achievable results, will become more firmly established. The integration of intelligent elements will round off the established method and help in attaining the shortest possible time for analysis.

This trend is already evident in the case of dispersing devices. Preliminary, fully automated prototypes have been developed to the extent that they can operate with commercially available robots for routine analysis.

However, the major accent in the development will be placed on so-called compact sample couplers. The function of these devices is to provide a process-specific link with the production plant and to prepare the sample taken from the process in a manner appropriate for the analysis system downstream.

The conception of sample couplers and its technical realisation was introduced and presented by RÖTHELE, NAUMANN and BRANDIS [4].

An example for an on-line suspension system is the Sampling Finger Robot SAFIR.

3.4 on-line analysis, e.g. with SAFIR

SAFIR is a compact sample coupler for performing on-line analysis with suspensions.

With the use of SAFIR, samples are also taken from highly concentrated suspensions and prepared for the subsequent particle size analysis with a laser diffraction sensor fully automatically under computer control.

The sample coupler offers flexibility in selecting the measuring intervals by means of the cycle interval setting, which must be longer than five minutes. Matching to the suspension concentration in production is effected by means of the sampling frequency.

The tasks for the user are reduced to the individual configuration of the product- and process-dependent sequence and are as a rule limited to the beginning and end of analytical campaigns and possibly control operations.

A functional principle is shown in figure 5.

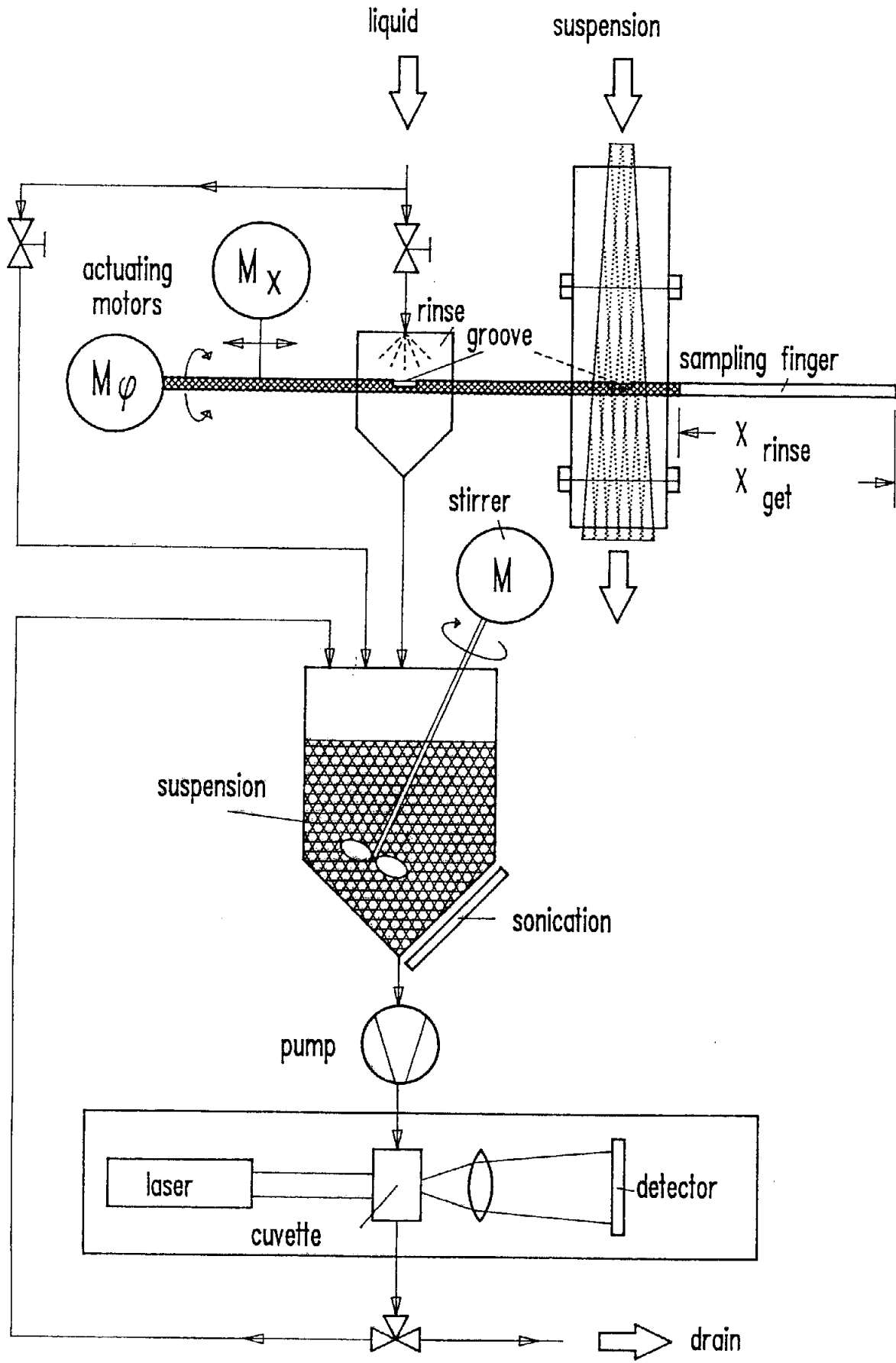


Fig. 5: SAFIR/HELOS functional principle

In figure 6 an example is presented for the reproducibility of measurements supported by SAFIR. The scatter curves about the average Q_3 value are compared here. The two series of measurements shown were performed under different boundary conditions.

The first series (Δ) shows the result of six repetitive measurements on the same sample. The result indicates that the laser sensor always reproduces the particle size distribution decidedly better than a scatter less than ± 0.2 per cent in every size class and preponderantly even better than $< \pm 0.1$ per cent.

The second series (\square) indicates the scatter for six different samples taken in succession in the same population. The scatter curve is comparable. The influence of the sampling procedure, being composed of the mixing effect of sensor and sample coupler can not be detected or isolated.

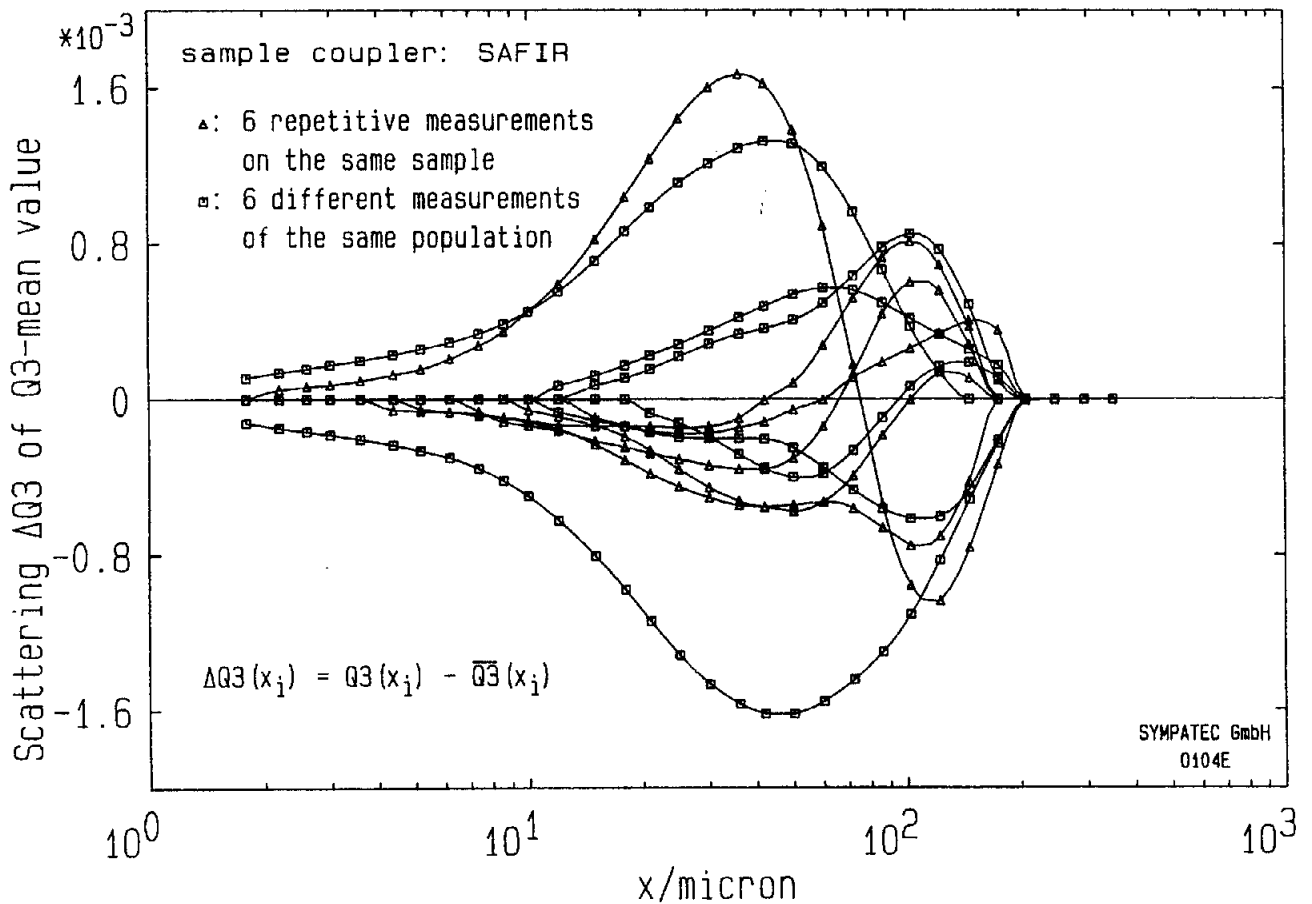


Fig. 6: Reproducibility for sensor and sample coupler

4 Conclusions

The development of applications of the laser diffraction technology over the last twenty years as a modern method for fast and high precision analysis of particle size distributions has opened a new dimension.

The superiority is resulting from the cooperation of different elements being most advantageous for its further propagation. The most important are:

- * fast measurement with a resolution unknown to industrial aspects
- * broad measuring range meanwhile between $0.1\mu\text{m}$ to nearly 3mm covering today's most important size range without change of the measuring method
- * independence of product parameters and its resulting simplicity of operation even for high expectations.

The most successful instruments applying this method in addition to the other features already mentioned above are adapting themselves to the products. I.e. dry manufactured powders can be analysed dry and wet manufactured powders respectively can be analysed in suspension. This is the most important step into automatic and on-line application of the Fraunhofer method.

Meanwhile sample couplers are available that can be used for the realization of standard project solutions for process control.

Together with the increasingly appearing on-line applications the laser diffraction technology is on its way to install itself as dominating standard for particle size analysis at the end of this century.

5 Nomenclature

List of used formula characters and indices

$I(r)$	intensity distribution of scattered light
i_F	diffraction function of the Fraunhofer-solution
i_M	scattering function of the Mie-solution
$Q_3(x)$	cumulative distribution by volume
$q_0(x)$	differential distribution by number
t_A	analysis time
x	particle size
x_{\min}	minimum particle size
x_{\max}	maximum particle size
Δx	measuring range
α	Mie-parameter
θ	scattering angle
λ	wavelength of laser
Ω	dynamic measuring range rate

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