Application of Laser Diffraction with Dry Dispersion for Automatic Particle Size Analysis

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Introduction

The characterisation of the particle size distribution of cement has become one of the important parameters for the determination and the control of quality. With the introduction of the laser diffraction principle into industrial quality control some 15 years ago the basis for further development was laid.

Sympatec in 1984 introduced a new generation of laser diffraction analysers, based on the simple idea that the instruments used for the characterisation of quality parameters have to adapt to the products and processes and not vice versa, i.e. that the products have to adapt to the instruments, which was the common way of how analytical equipment was designed.

This basic rule consequently led to the development of a range of laser diffraction analysers and dispersing systems that are of modular design thus being easily adaptable to a wide variety of different products and processes.

Dry powders have to be dispersed dry, wet products and suspensions have to be dispersed in the wet state. This is the practical and consequent conclusion.

Sympatec GmbH being founded in 1984 in Germany have developed a to date unique instrument for the dry dispersion of powders in the particle size range from below 0.5 μm to more than 3 mm. This patented instrument, the RODOS, allows for a smooth, complete and product adaptable dispersion.

In combination with the laser diffraction instrument, HELOS, a powerful system for industrial particle size analysis of the powders is available.

Company Profile and Technical Highlights

The development of Sympatec GmbH can be taken from the company profile (fig. 1). Sympatec, standing for SYsteM PArticle TECnology, since its foundation in 1984 as a hightech spinout of the University of Technology Clausthal, Germany (famous for its mining tradition), has featured remarkable Highlights in the development of the laser diffraction technology (fig. 2). This certainly underlines Sympatec's claim for technical leadership.

Laser Diffraction Principle

The history of Laser Diffraction as an advanced optical analysis principle for fast determination of particle size distributions in a wide size range, i.e. from 0.1 to x.000 μm, begins in the early 19^{th} century with the experimental work of Mr. Fraunhofer in Munich (fig. 3).

Those experiments are the basis of what is known today as Fraunhofer diffraction physics.

From these first experiments it took about 130 years for the technical realisation of the first laser diffraction particle size analyser in 1972 [1].

The optical setup for the generation of diffraction patterns consists of a laser as light source, an optical beam expander that widens the very fine laser beam to more than 10 mm in diameter, and a measuring zone where the sample is fed and taken out.

The measuring zone is the position where the interactions between the monomochromatic light and the particles take place and the diffraction patterns are generated (fig. 4).

These phenomena of intensity distribution are focussed on the surface of an optical detector with the help of a Fourier transformation lens of a distinctive focal distance. The detector usually consists of up to 31 semicircular elements. They are tranforming the optical light intensity into the corresponding electric current (fig. 5).

A mathematical relationship can be established between the diameter of the particles and the intensity patterns [2]. For a particle size distribution, consisting of fine, intermediate and coarse particles, the diffraction pattern monitored with the detector, comprises a two parameter information; it is the distribution of particle sizes and the quantities (fig. 6).

An extremely important feature that determines the quality of the measurement to a very large extent is the accurate alignment of the optical setup, i.e. the precise focussing of the central laser beam to the centre of the detector. Sympatec have realised an focussing system that automatically and constantly checks this alignment and if necessary corrects it before a measurement can be released (fig. 7).

In a cutaway view of the HELOS/RODOS system the important elements of a technical realisation are shown (fig. 8).

Sympatec Assortment

As a result of the idea of adaptation of the instruments to the products and processes a completely modular conception of the Sympatec product range was realised.

The Sympatec assortment comprises instruments for off-, auto- and on-line particle size analysis with laser diffraction (fig. 9).

The HELOS Laser Diffraction Sensor is manufactured in four different types for a size range between 0.1 μm and 3.500 μm.

Dispersing Systems are provided for dry and wet applications in up to more than 20 variations.

Analytical Software allows for the processing of particle size analysis data. Sample couplers are used for the connection of the analysis system to the production lines if a complete on-line particle size analysis for continuous quality control is required.

Independent of the type of HELOS sensor all instruments show the same technical features. The Central Unit, made of high precision mechanical components, houses also the optical and electronical elements. The Systems Computer, being integral part of the instrument, underlines Sympatec policy of using high quality parts for best system performance (fig. 10).

A typical HELOS/RODOS configuration for off-line application including the Process Control System and a printer, is shown in the photo (fig. 11).

The variety of Dispersing Systems for dry and wet dispersion allows for the best choice with regard to the products to be analysed. The Matrix of dispersers shown can be used for the selection of the appropriate system (fig. 12).

The RODOS, standing for Rotating Dosing and Dispersing System, is a patented disperser for dry powders [3]. Due to its unique two stage design it can be used with powders in a very wide size range from below 0.5 μm to more than 3 mm (fig. 13).

Depending on the dispersion characteristics different adaptors are applied to feed the sample to the gas-solids injector, being the centre piece of the disperser.

For very fine and sticky powder the sample is fed in surplus into a rotary groove with the help of a vibratory feeder. Additional scrapers and rollers are compacting the powder in the groove. Thus a homogenous mass flow of powder is presented to the rear end of the disperser. The gas solids injector is driven by compressed air. In a nozzle it is accelerated to velocity of about 50 m/s.

Due to the accelaration of the compressed air, a vacuum is produced at the rear end of the disperser and the previously homogenized powder is taken into the disperser. In a zone of high velocity gradient between the air and the agglomerated particles, shear forces are applied to destroy these particle aggregates. Additional particle-to-wall and particle-particle collisions and centrifugal forces generated by the rotation of such aggregates completely and smoothly take the agglomerates apart, down to the primary particle sizes.

The powder leaves the disperser as an aerosol in a free jet and crosses the laser beam of the HELOS instrument at a very short distance from the exit of the disperser. After passage of the laser beam the aerosol is completely taken out of the air with the help of an extraction nozzle and a vacuum cleaner.

The advantages of the dry dispersion with RODOS over suspension (wet) dispersion are manifold and include complete and proper product specific dispersion, adaptable dispersing forces and variable sample size, highest measurement frequency available to day, as well as a guaranteed long life time of the disperser with minimum operating costs (fig. 14).

With regard to application in the cement industry RODOS is the natural way for the dispersion of

- cement
- raw meal
- coal dust
- fly ash
- .....

Automation

In addition to the off-line instruments designed for application in labs of all branches with manually operated dispersing systems, Sympatec have created a new line of instruments, called auto-line, for application in automatic laboratories for continuous quality control. These instruments automatically perform a particle size analysis once a sample has been filled into the disperser. They can be operated as stand alone solutions and, due to their ability for cooperation with robots and central process control systems, they can be integrated into the laboratory automation systems supplied by well known plant manufacturers.

A typical cycle time for a measurement with an integrated Sympatec auto-line system with the automatic dry dispersing unit auto-RODOS module totals to less than two minutes (fig. 15).

Included in the cycle is the information exchange between the central control computer of the automatic lab and the HELOS systems computer, the fill in of the sample, the dispersion and measurement of the size distribution, the evaluation of data and transfer to the host computer [4].

The most significant differences in the differential distribution curves are those between the charge 1 and charge 2: Charge two is showing a remarkably higher amount of coarse particles (fig. 23).

With regard to the long time stability of the dry dispersing system auto-RODOS, Sympatec guarantees a life time of 25.000 measurements of PZ 35 F, i.e. a life time of typically one year in an automatic production control laboratory. The standard deviation over the life time is less than 1 % or expressed in terms of the BLAINE surface, inferior to +/- 40 cm^2/g for 4000 cm^2/g (fig. 24).

Compared to the operating costs of systems that are using suspension dispersion in alcool, the dry dispersing system RODOS has an advantage of probably more than 50%.

Conclusion

As a conclusion the following important facts can be stated.

HELOS and auto-RODOS module are the superior components for automatic production control of dry powders they

* represent the state of the art of laser diffraction technology
* have been established world wide over the last years
* are using a unique dry dispersing system
* allow for product specific dispersion of powders
* are designed for a high measurement frequency of up to 200 measurements/day
* have a guaranteed life time of 25000 measurements
* are thus allowing for lowest operating costs.

References

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Results Obtained with a New Instrument for the Measurement of Particle Size Distributions from Diffraction Patterns,
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[3] *Kurt Leschonski, Stephan Röthele, Ulrich Menzel*:
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[4] *Stephan Röthele, Heinz Naumann, Udo Brandis*:
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Company profile Sympatec GmbH
SYsteM-PArticle-TEChnology

★ Foundation	1984	
★ Location in Germany	Clausthal	(Sales, Administration, After-Sales)
	Goslar	(R & D, Distribution, Manufacturing)
★ Venture Capital	Swiss Holding Company	
★ Products & know-how	Powder Technology Department TU Clausthal, University of Karlsruhe (TH)	
★ Awards	1985: technology transfer award of IHK Braunschweig (chamber of commerce); dry dispersion technology: RODOS	
	1986: technology transfer award of the German Federal Minister of Education and Science; **on-line-particle-size-analysis**	
	1992: Arnold-Euken-Award; GVC: **OPUS ULTRASONIC EXTINCTION**	
★ Marketing strategy	- **laser diffraction for dry** dispersed bulk materials **to below 1 µm** - development of dispersing systems for the **adaptation of laser diffraction to products** - **modular conception** of instruments	
★ Manufacturing strategy	- **lean production** - **assembly-plant** - **high-tech-product**	
★ Employees	48 in 1993	

Highlights of technical leadership

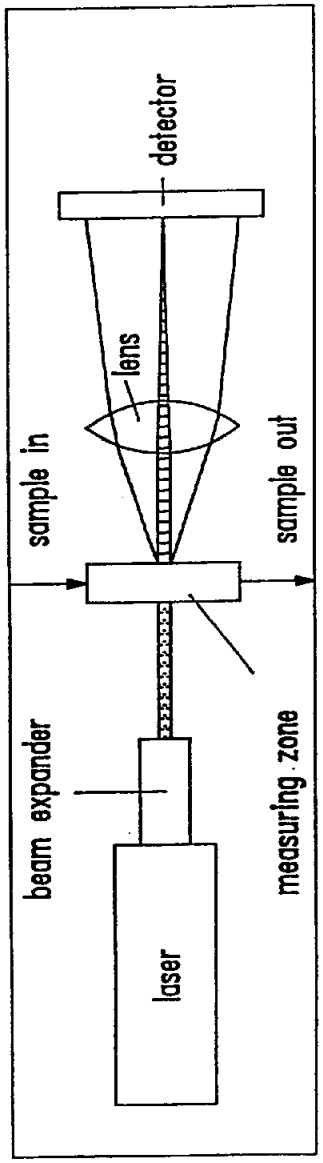
1984	RODOS	adaption of laser diffraction for dry analysis from less than 1 µm, with patented two-stage dispersion system
	HELOS	highly accurate for auto-aligned 31 classes, combined with parameter-free Philips-Twomey solution
1985	QX	programme package for universal processing of particle size distributions (psd)
1986	ROPRON	patented sample-coupler with two-stage in-stream sample splitter (dry pocesses)
1987	SUBMICRON	pioneering application of Fraunhofer diffraction from 0.1 µm, with 11 points measured below 1 µm
1988	REMO/PARA	Software modules for comparison of different psa-methods, for example, laser diffraction with sieve analysis
1989	auto-RODOS auto-SUCELL	automatic versions of dispersing systems, computer controlled, robotor-adaptable
1990	auto-GRADIS	
	TRIMO	dynamic trigger-ignition
	SAFIR	Sampling Finger Robot (wet processes)
1991	QT	Quality time-accelerator
	Spray-Sizer	universal adaptor for spray-applications
	MAGIC	HELOS-Sensor with auto-ranging
	OPUS	acoustic on-line-psa using ultra sonic-extinction
1992	auto-RODOS-module	integrated dry disperser with extremely extended long-term stability
	TOPMICRON	range extension with deflection amplifier
	MIE	high speed tool with unlimited refractive index stock

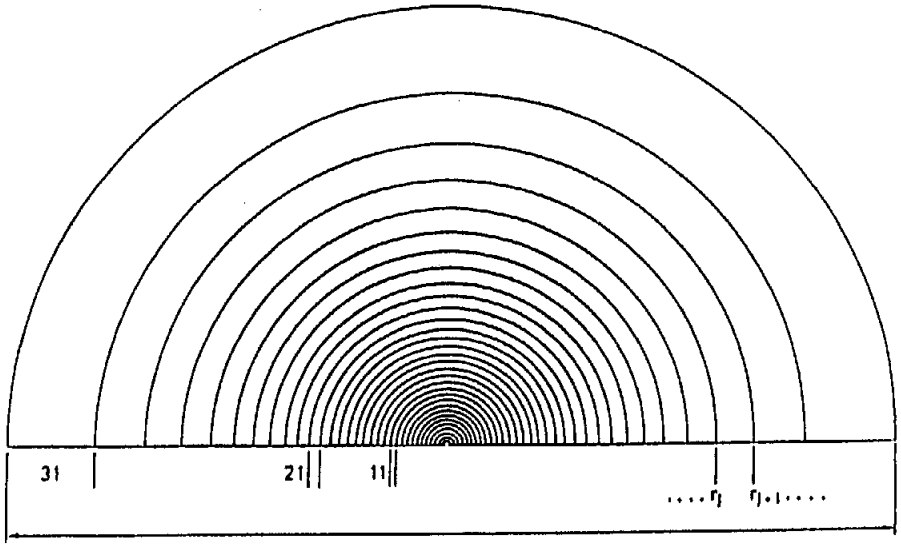
Laser Diffraction History

Advanced analysis principle for fast determination of particle size distributions in a wide size range i.e. from 0,1 - X.000 µm

Elements	Definition	Year
Theory	**Fraunhofer** diffraction physics	1840
Method	He-Ne-laser as high energy, coherent light for the generation of **particle-light interaction**	1960
Sensor	**multi-element semiconductor** first used as aiming device for military application	1965
Processing	high performance **microcomputers**	1970
Result	**particle size analysis with laser diffraction**	since 1972

optical set up for the generation of diffraction patterns

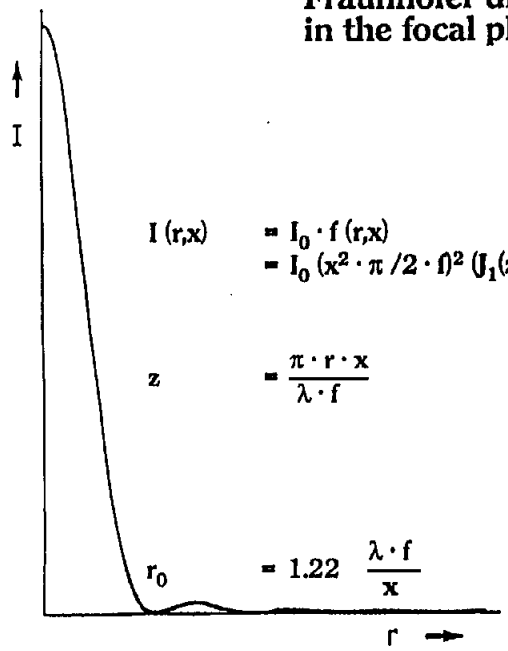




Multi-element detector with 31 classes

3E08K10U3E

Intensity distribution of Fraunhofer diffraction patterns in the focal plane

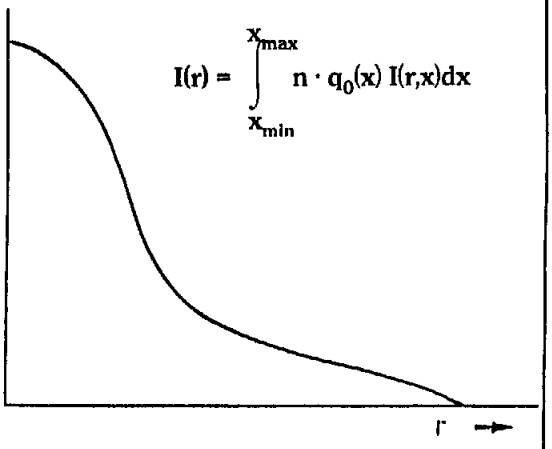


$$I(r,x) = I_0 \cdot f(r,x) = I_0 \, (x^2 \cdot \pi / 2 \cdot f)^2 \, (J_1(z)/z)^2$$

$$z = \frac{\pi \cdot r \cdot x}{\lambda \cdot f}$$

$$r_0 = 1.22 \, \frac{\lambda \cdot f}{x}$$

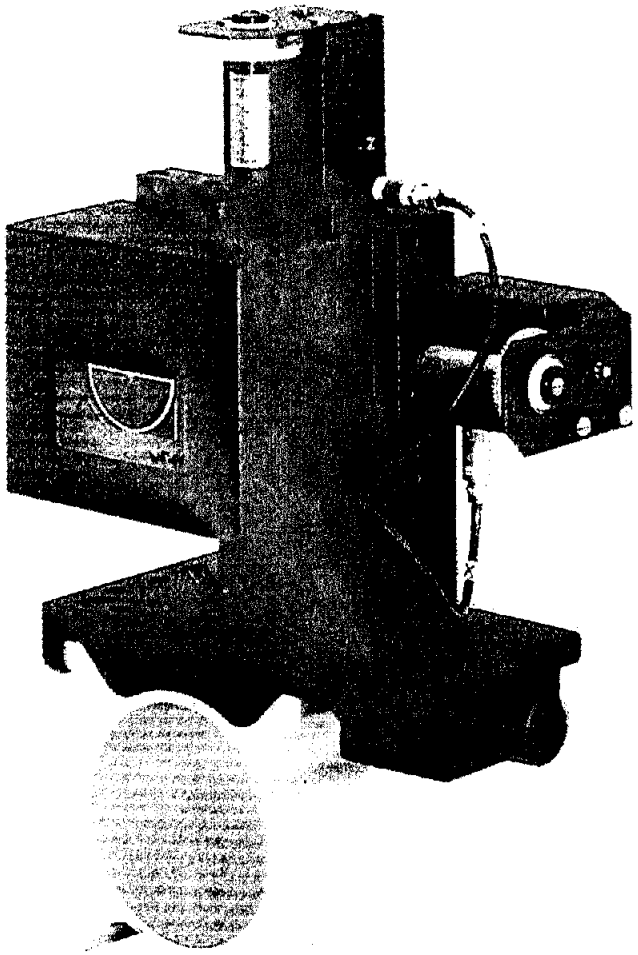
single particle of diameter x

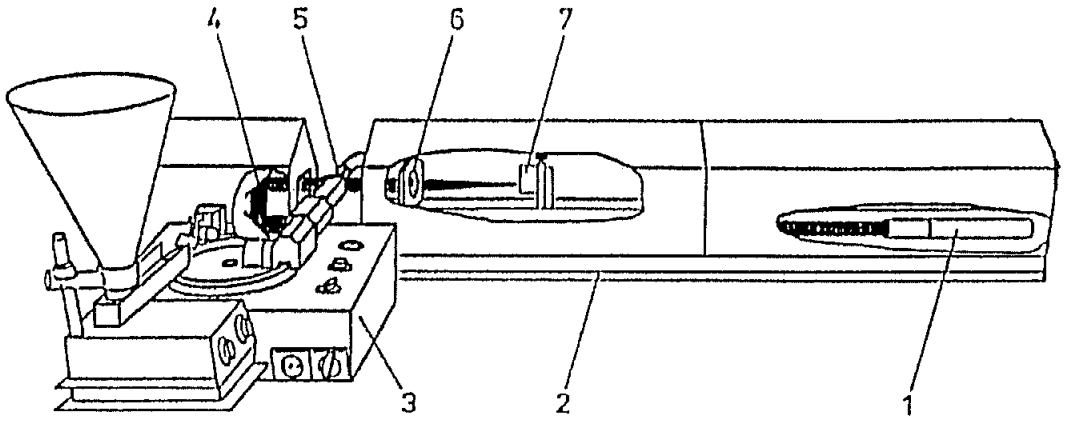


$$I(r) = \int_{x_{min}}^{x_{max}} n \cdot q_0(x) \, I(r,x) dx$$

particle size distribution $q_0(x)$

3E08K10U2E





cutaway view of
HELOS/RODOS-system

1 He-Ne laser with beam expander
2 optical bench system
3 dry dispersing system RODOS
4 deflection mirrors
5 measuring volume
6 Fourier lens
7 multi-element detector with autofocus-system

SYMPATEC

instruments for off-, auto- and on-line particle size analysis

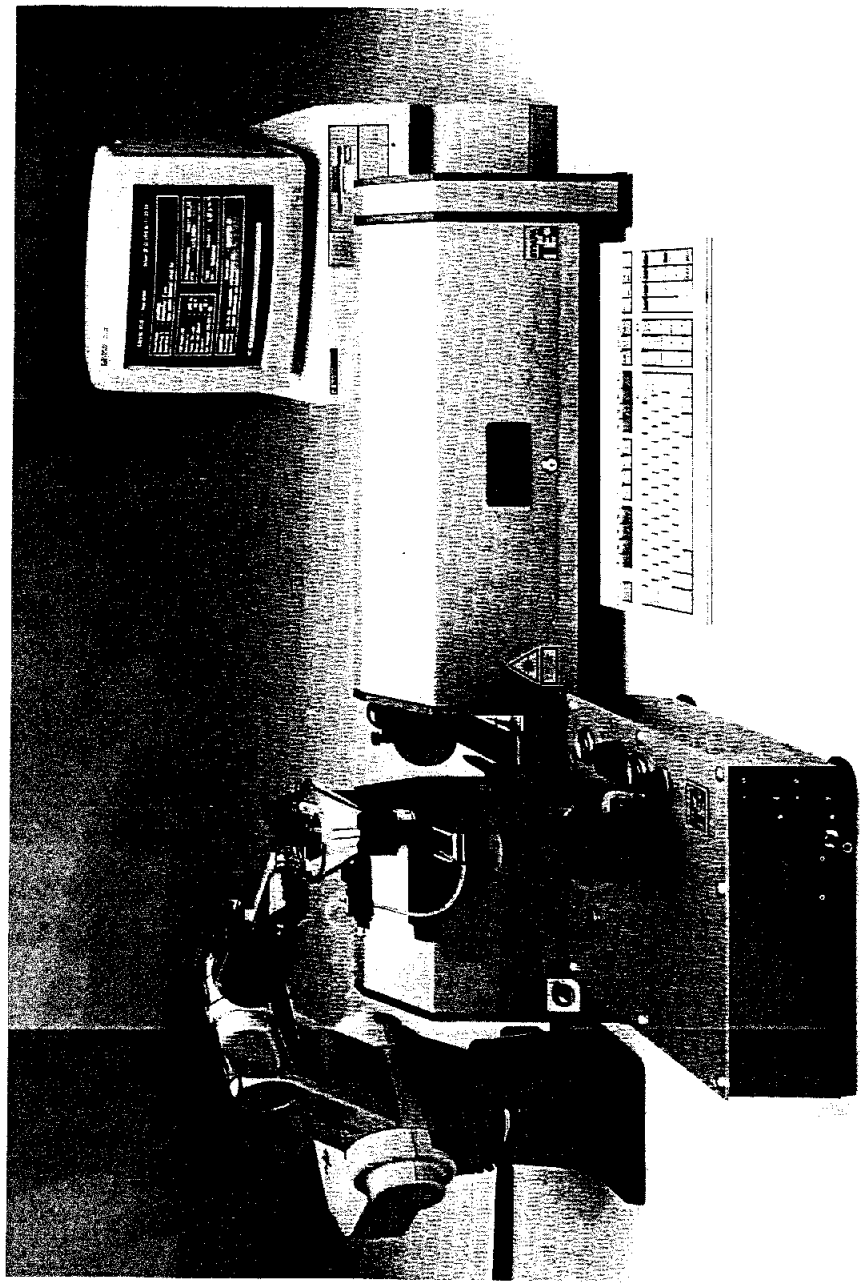
★ **Laser Diffraction Sensors** : for the analysis of particle size distributions

HELOS/BA*: 0.1 - 350 µm
KA*: 0.1 -1750 µm
LA*: 0.1 -3500 µm
VARIO*: 0.5 -3500 µm
(* also with MAGIC option)

★ **Dispersing Systems** : for the adaptation of the sensors to powders, suspensions, emulsions, sprays
dry: **RODOS***, **GRADIS***
wet: **SUCELL***, **CUVETTE**
(* also in auto-line version)

★ **Analytical Software** : for processing of psa data and correlation with other analysis principles
QX, REMO & PARA, TRIMO, QT

★ **Sample Couplers** : for the connection of the analysis systems to the production lines
dry: **ROPRON**
wet: **SAFIR**



Dispersing Systems

principle:

dispersing medium	in measuring zone of sensor turbulent	statical
dry	**RODOS**	**GRADIS**
size ranges	1 < x < 2500 µm	100 < x < 3500 µm
quantity	5.0 g - 100 g	5.0 g - 100 g
wet	**SUCELL**	**CUVETTE**
size ranges	0.1 < x < 875 µm	0.5 < x < 1000 µm
quantity	0.5 g - 2.0 g	0.001 g - 0.5 g

Laser Sensors Basic Equipment

★ **central unit**

in BA-, KA-, LA-, VARIO-cabinet of stainless steel with super pressure ventilation, and
- optical bench system
- HE-NE-laser or laser diode
- 31 channel multielement detector incl. auto alignment system (autofocus)
- data acquisition unit MECB-HEL
- high speed fibre optic communication interface

★ **system computer**

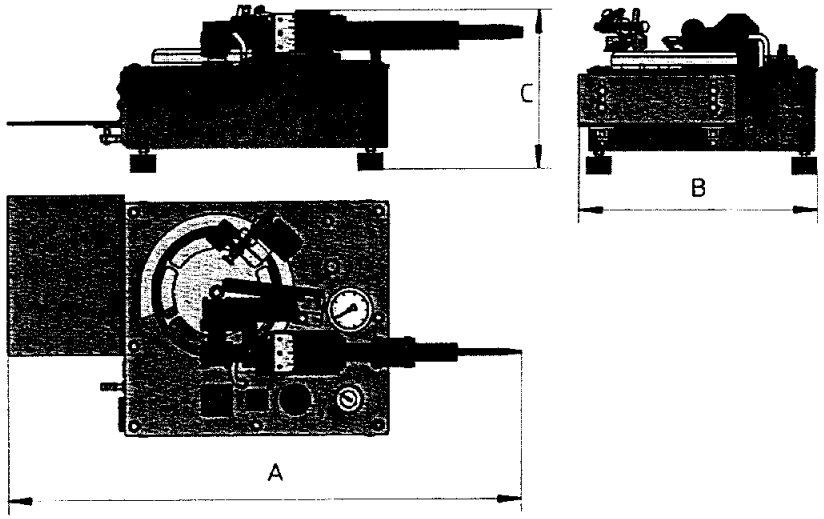
- HP VECTRA 486/XX
- HELOS software package
- high speed fibre optic communication interface
- XXX MByte hard-disk
- 3.5" floppy-disk 1.44 MByte
- HP 14" super VGA colour VDU
- keyboard
- MS-DOS license

Advantages of Dry Dispersion with RODOS

- ★ **complete and proper dispersion of all kinds of dry powders to < 0.5 µm**
  - ➪ sensitive to changes in the fine particle range
- ★ **adjustable dispersing forces**
  - ➪ best adaptation to dispersibility of different products
- ★ **variable sample size**
  - ➪ minimising the statistical sampling error
- ★ **high measurement frequency due to short cycle time**
  - ➪ no cleaning, no rinsing, of sample container
- ★ **no chemical interaction between particles and dispersing fluid**
  - ➪ no dissolution, no surface reaction, ...
- ★ **no reprocessing of contaminated liquid**
- ★ **guaranteed long life time of disperser**
  - ➪ low operating cost
  - ➪ maximum up-time

Dry Dispersing System

RODOS



A = 783 mm
B = 364 mm
C = 240 mm

auto-line cycle time

steps		auto-RODOS	auto-SUCELL
1. information in		5"	5"
2. initialisation		10"	10"
3. fill in		15"	60"
4. sampling & dispersing		10" - 30"	10" - 30"
5. take out		15" - 35"	60" - 120"
6. evaluation		5"	5"
7. information out		5" - 60"	5" - 60"
Σ 1. - 7.: [minutes]	<	2	5
8. stand-by		1" - ∞	1" - ∞

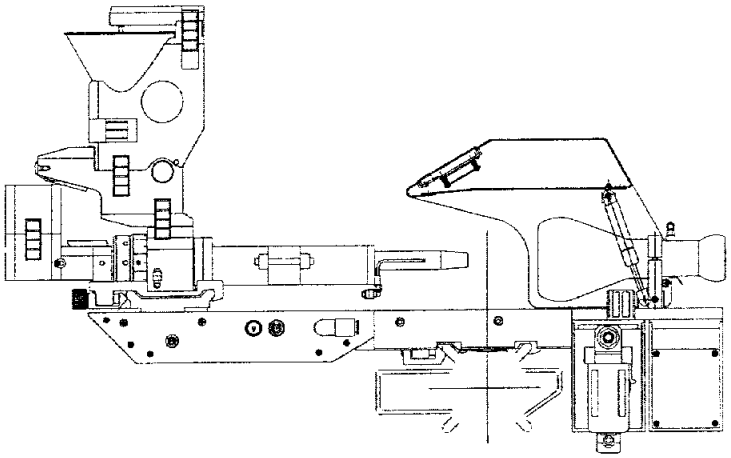
auto-RODOS module

automatic dry dispersing system for use with HELOS laser diffraction analyser

- ★ **plug-in module for direct installation in HELOS measuring zone**
- ★ **integrates all functions for automatic particle size analysis, e.g. in cooperation with robots**
  - ☆ **mechanics:**
    - fully hardened dispersing line
    - polished inlet-funnel
  - ☆ **electronics:**
    - separate box on backside, fail-safe design
    - integrated security functions
    - local/remote operation
  - ☆ **integrated ancillary services:**
    - cover to eliminate external light, incl. operational panel
    - filters for compressed air, incl. monitoring of primary pressure
    - lens cleaning system with specially cleaned compressed air
    - streamline extraction unit, incl. monitoring of vacuum
    - fixed position relative to laser beam

Dry Dispersing System

auto-RODOS module

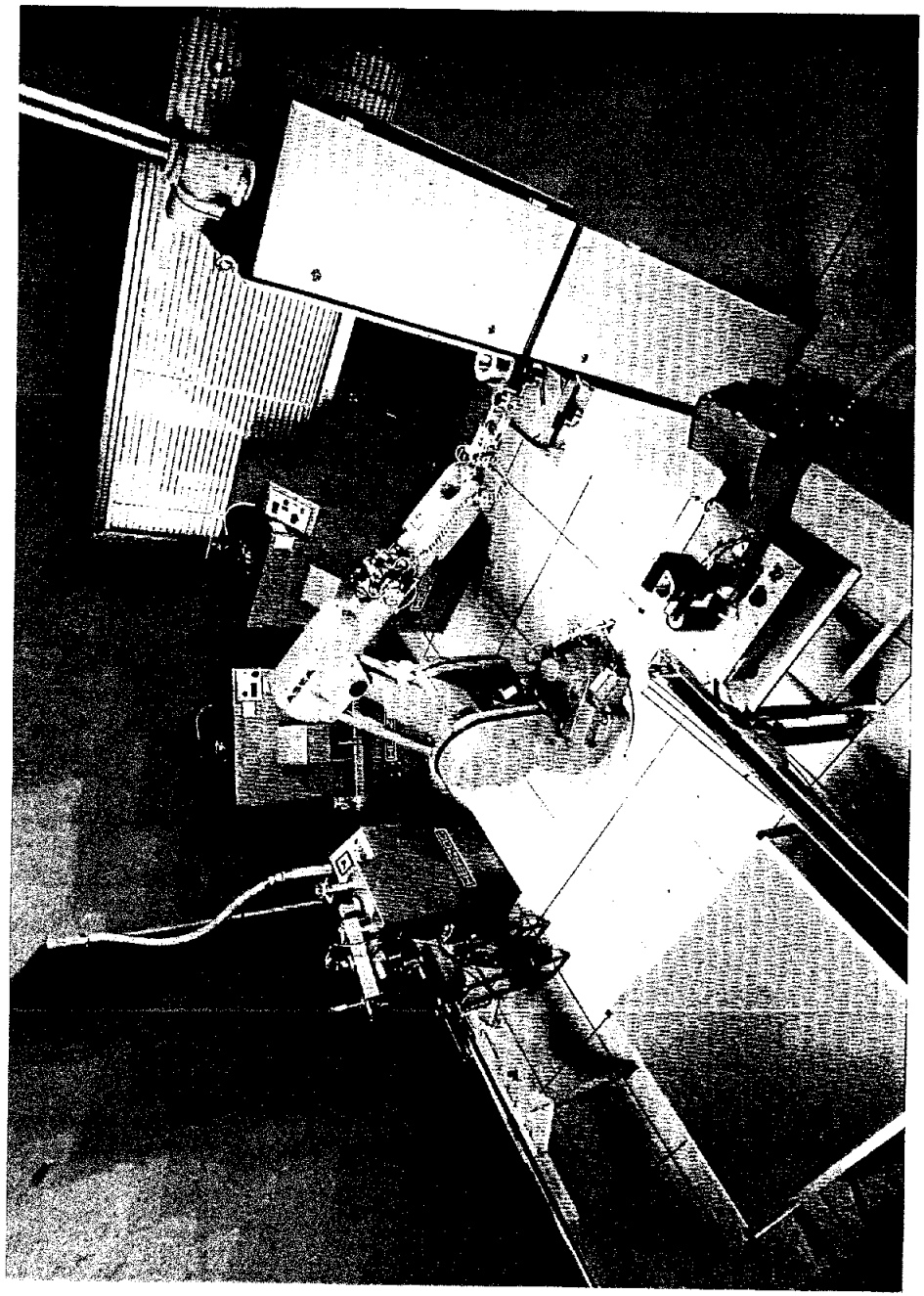


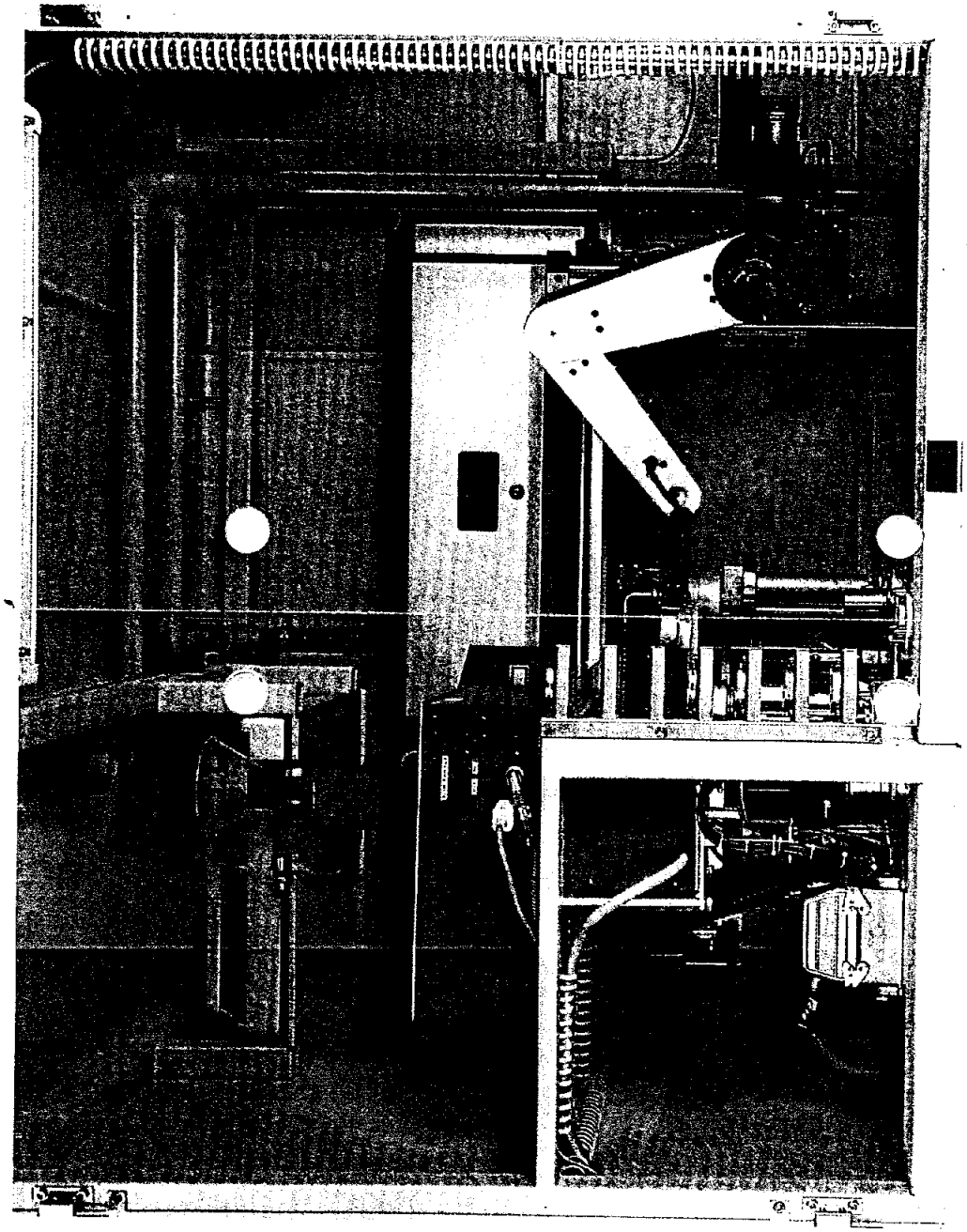
HELOS / MAGIC

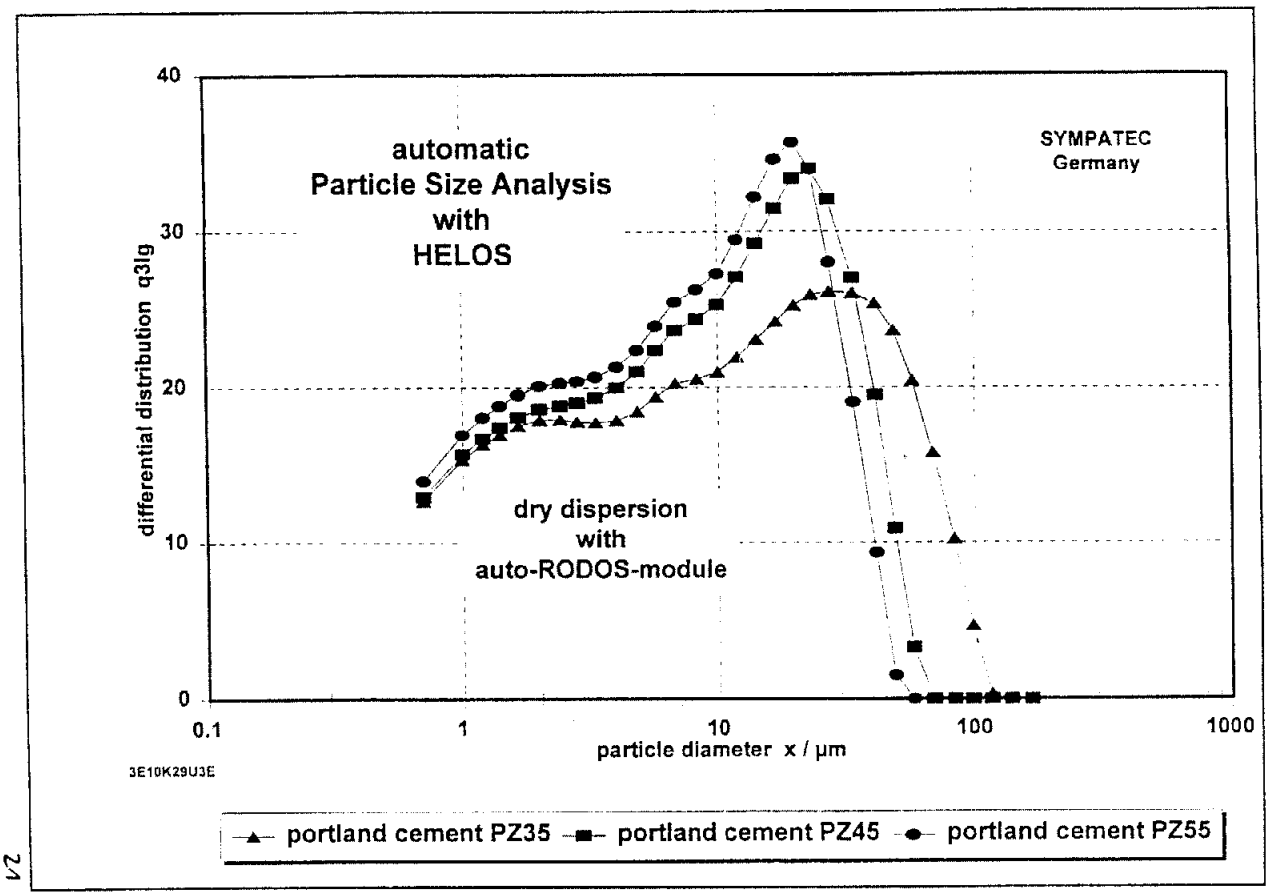
laser diffraction system with automatic change of the measuring range (auto-ranging), including auto-focus features:

- ★ lense rotator for up to 4 lenses/ranges
- ★ detector positioning unit
- ★ MAGIC electronic
- ★ MAGIC software

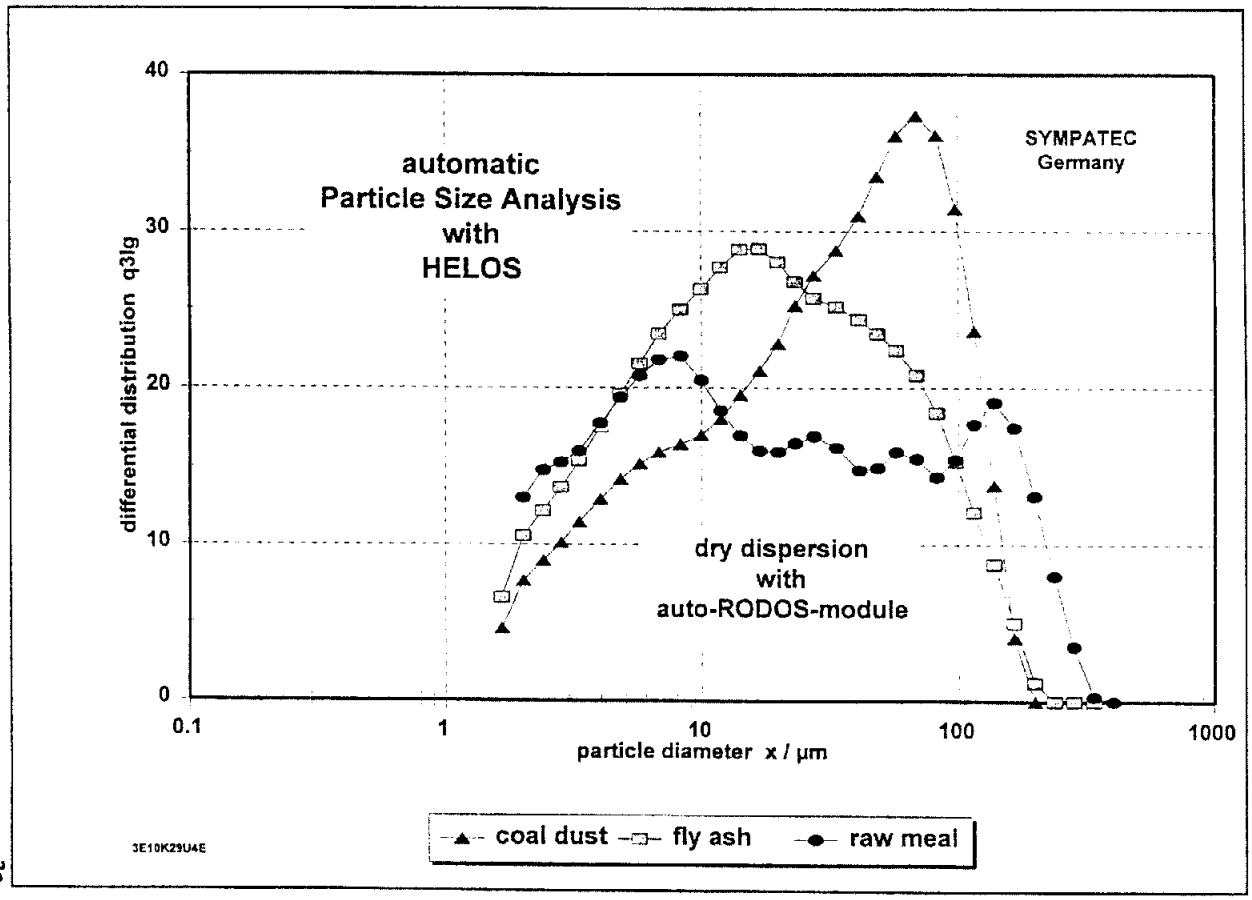
➠ **MAGIC module cooperates with all types of auto-line dispersing units**



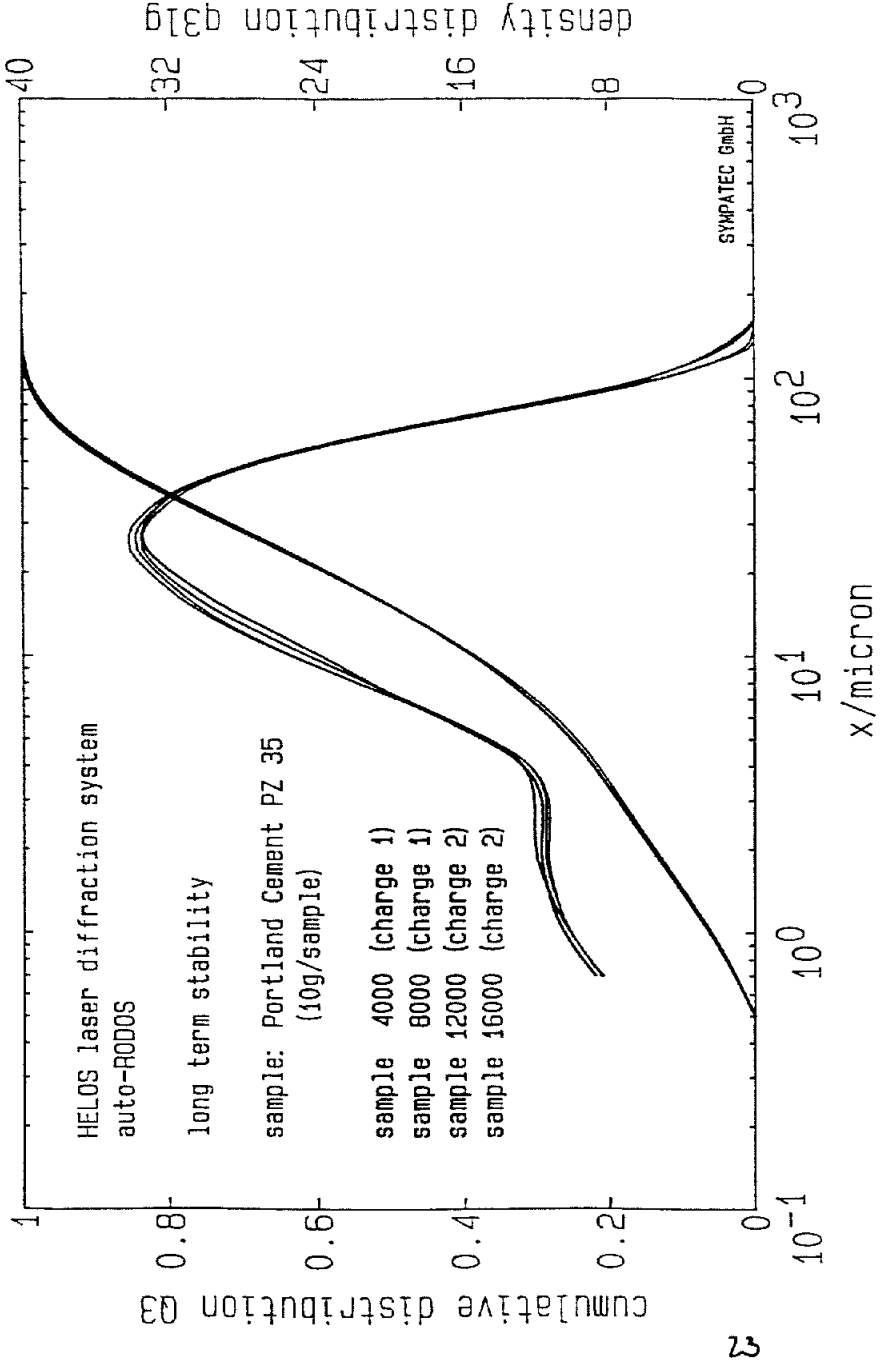



automatic
Particle Size Analysis
with
HELOS
SYMPATEC
Germany
dry dispersion
with
auto-RODOS-module
differential distribution q3lg
particle diameter x / µm
0
10
20
30
40
0.1
1
10
100
1000
3E10K29U3E
portland cement PZ35
portland cement PZ45
portland cement PZ55

21

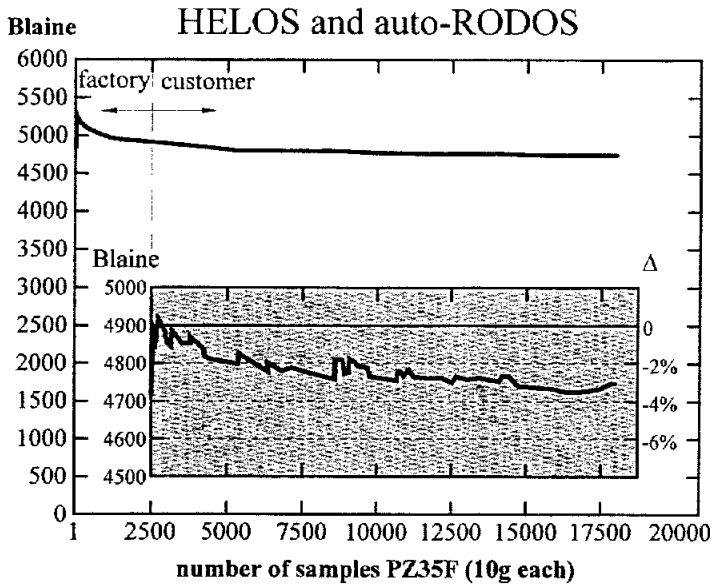

automatic
Particle Size Analysis
with
HELOS
SYMPATEC
Germany
dry dispersion
with
auto-RODOS-module
differential distribution q3lg
particle diameter x / µm
0
10
20
30
40
0.1
1
10
100
1000
3E10K29U4E
coal dust
fly ash
raw meal

22



Long Time Stability

Example auto-RODOS:



Blaine-values $S_M = \frac{6f}{\rho}\sum_i \frac{q_3(x_i)}{x_i}\Delta x_i$ (fine tail is significant)

- ★ standard deviation: < 1%, typical 0,4%
- ★ Blaine values: ± 40 cm²/g for 4000cm²/g
- ★ life time: ca. **250kg** PZ35F i. e. **25000 samples, 10 g each** or typical **1 year**