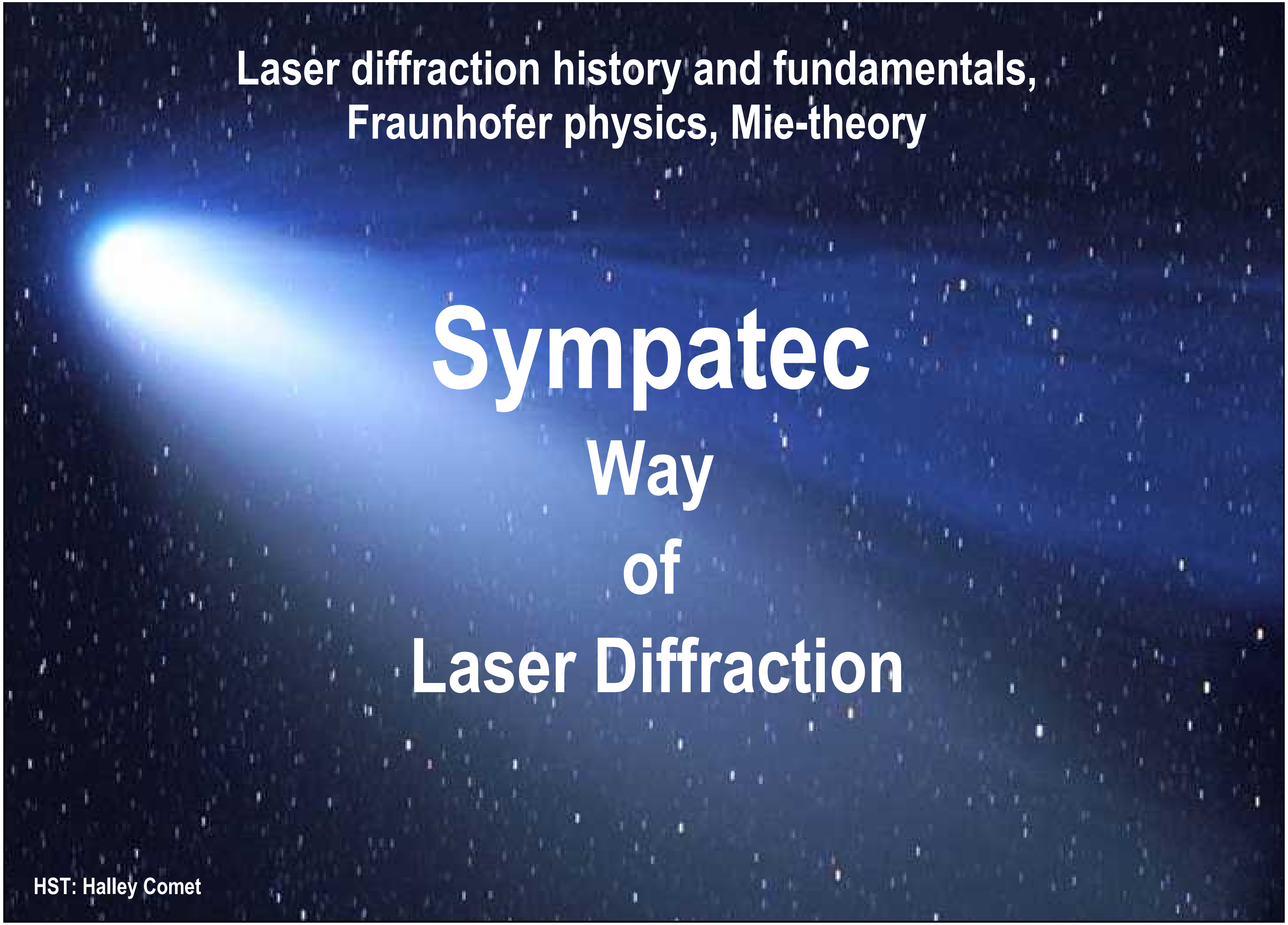


Laser diffraction history and fundamentals, Fraunhofer physics, Mie-theory

Sympatec Way of Laser Diffraction

HST: Halley Comet

Sympatec Way of Laser Diffraction

- ★ History of laser diffraction
- ★ Fundamentals
  - ☆ Generation of diffraction patterns
  - ☆ Diffraction pattern
  - ☆ Intensity distributions
  - ☆ Detection
  - ☆ HELOS auto-focus unit
  - ☆ HELOS/RODOS-system
- ★ Comparison of particle size analysis (psa) methods
- ★ Epochs of application
- ★ Fraunhofer Submicron
  - ☆ Mie theory
  - ☆ HELOS-MIE software module
  - ☆ Check list

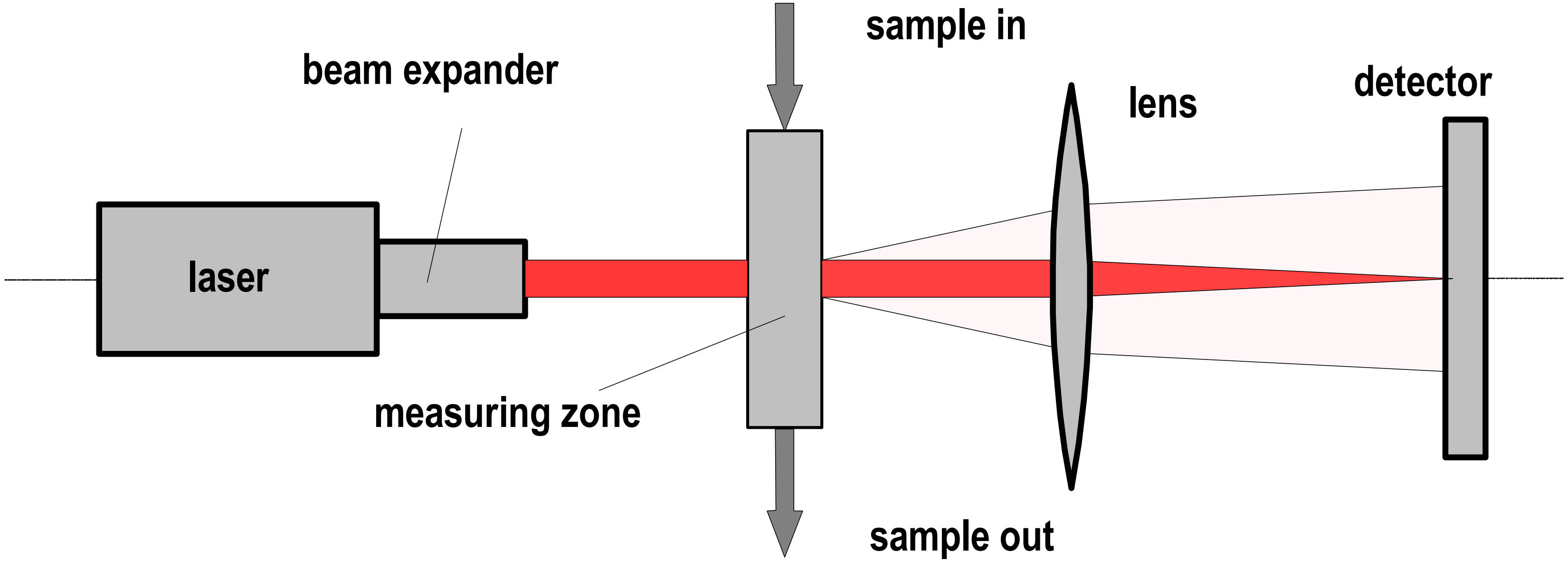
Laser Diffraction History

Advanced analysis principle for fast determination of particle size distributions in a wide size range from 0.1 - 10000 µm

Elements	Definition		Year
Theory	Fraunhofer diffraction physics		1820
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**

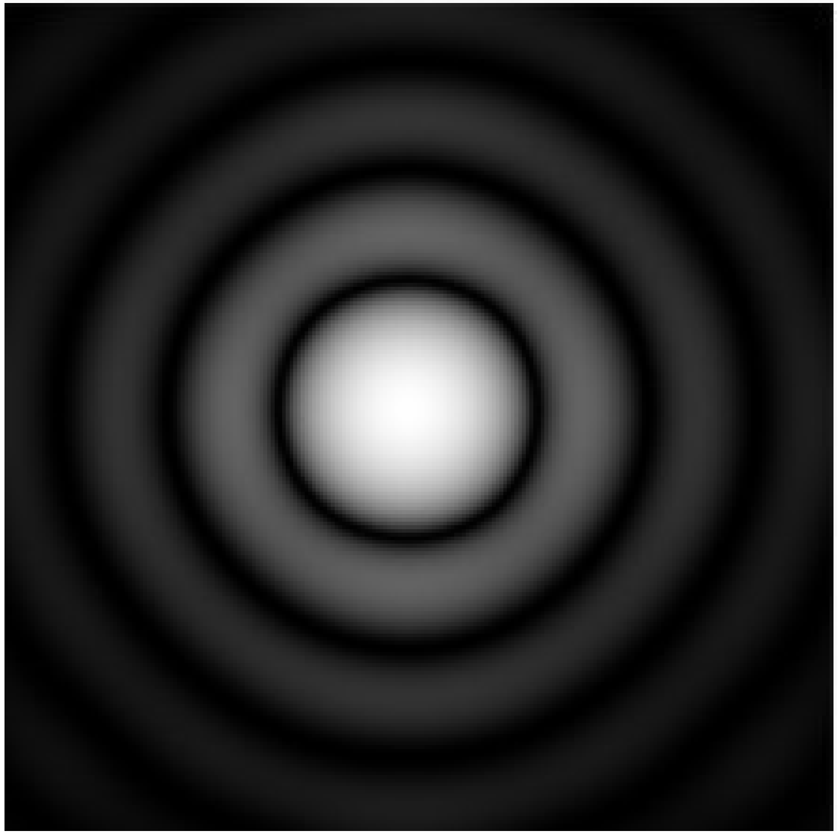
Optical set up for the generation of diffraction patterns

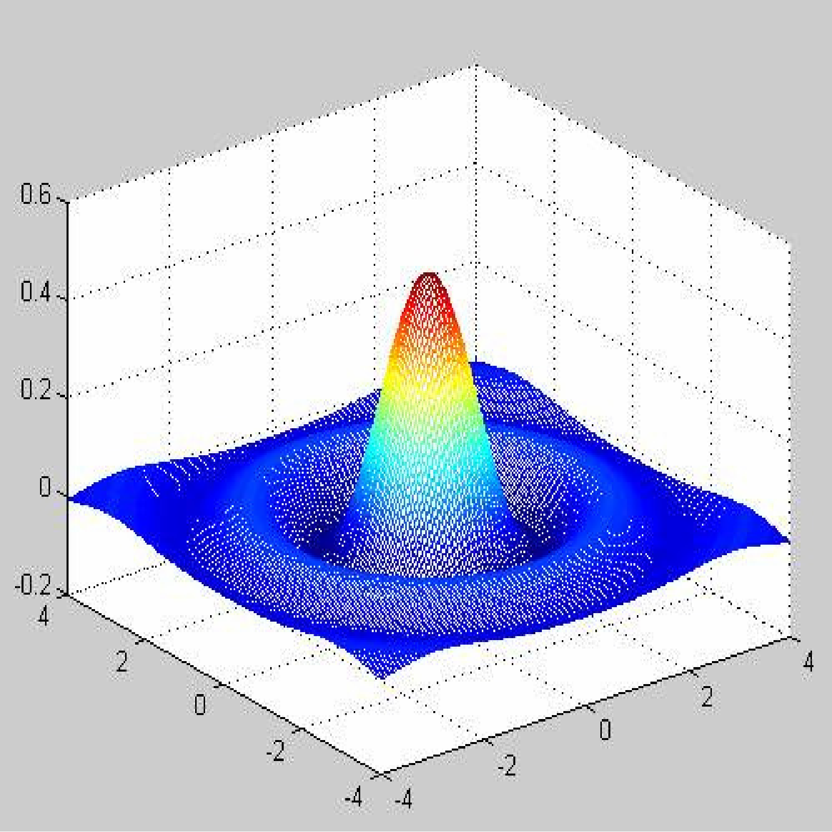
(refer to ISO 13320-1*, § 5, fig. 2)



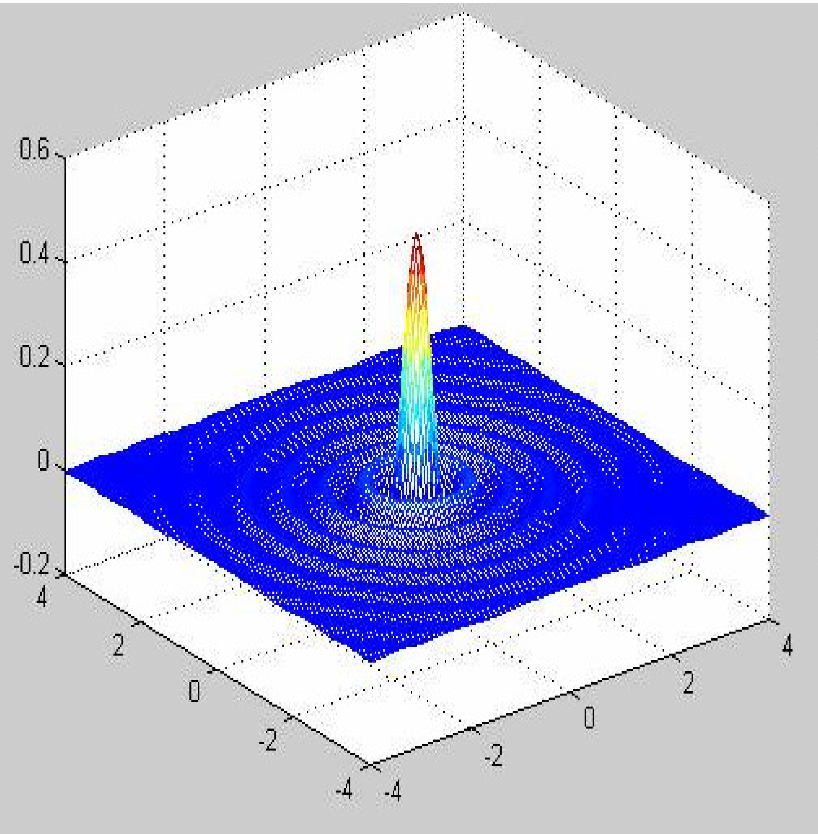
---

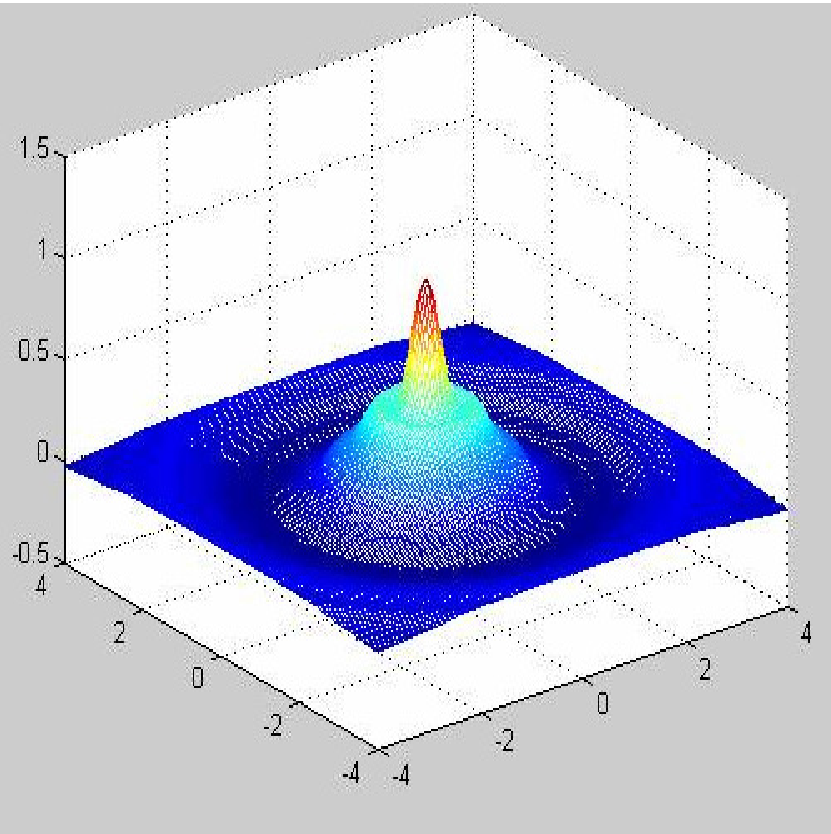
* ISO 13320-1 covers particle sizes from 0.1 µm to approximately 3.0 mm





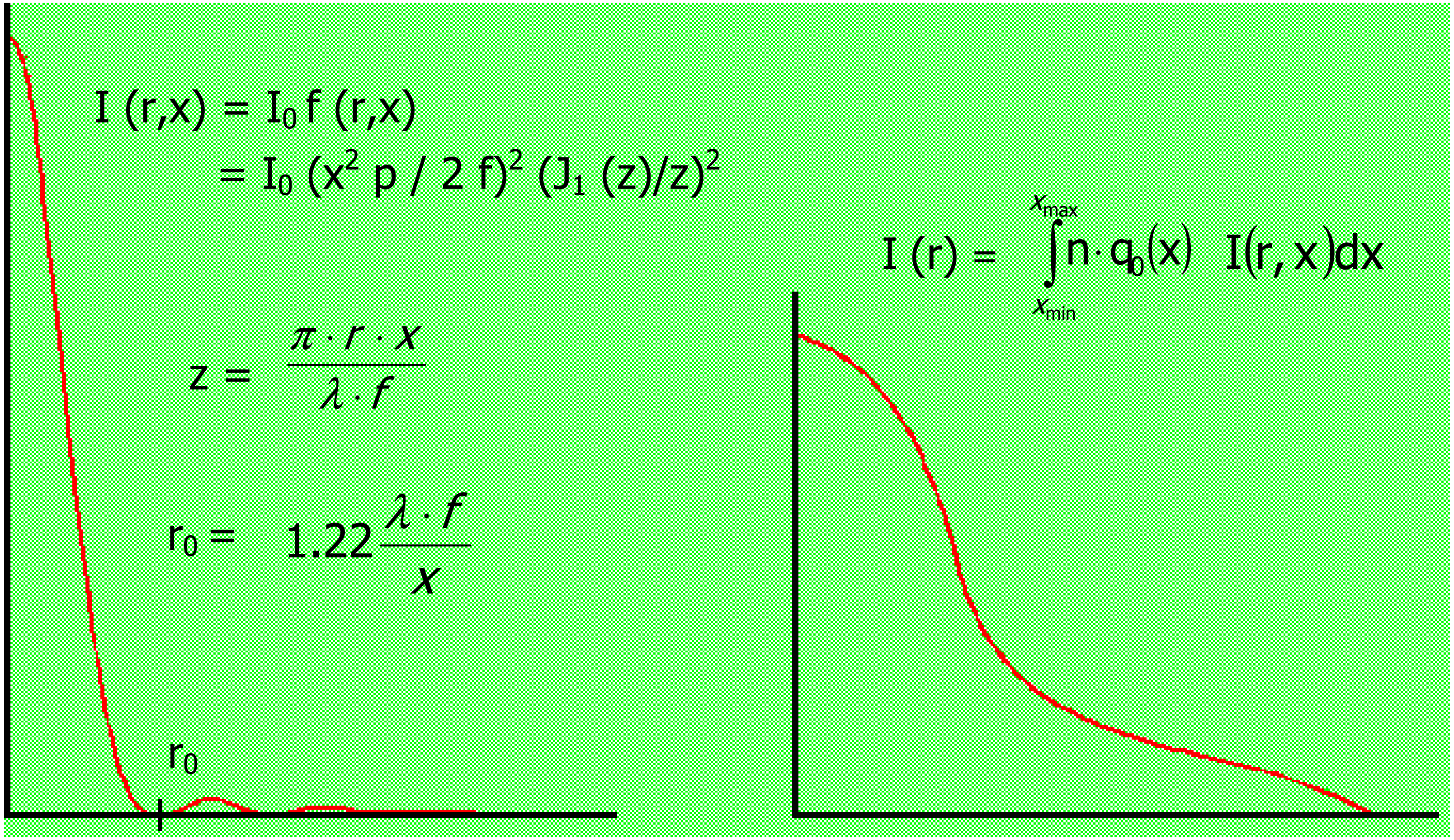
Diffraction Pattern of small spherical Particles





Single coarse particle

Overlay of fine & coarse particle



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

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

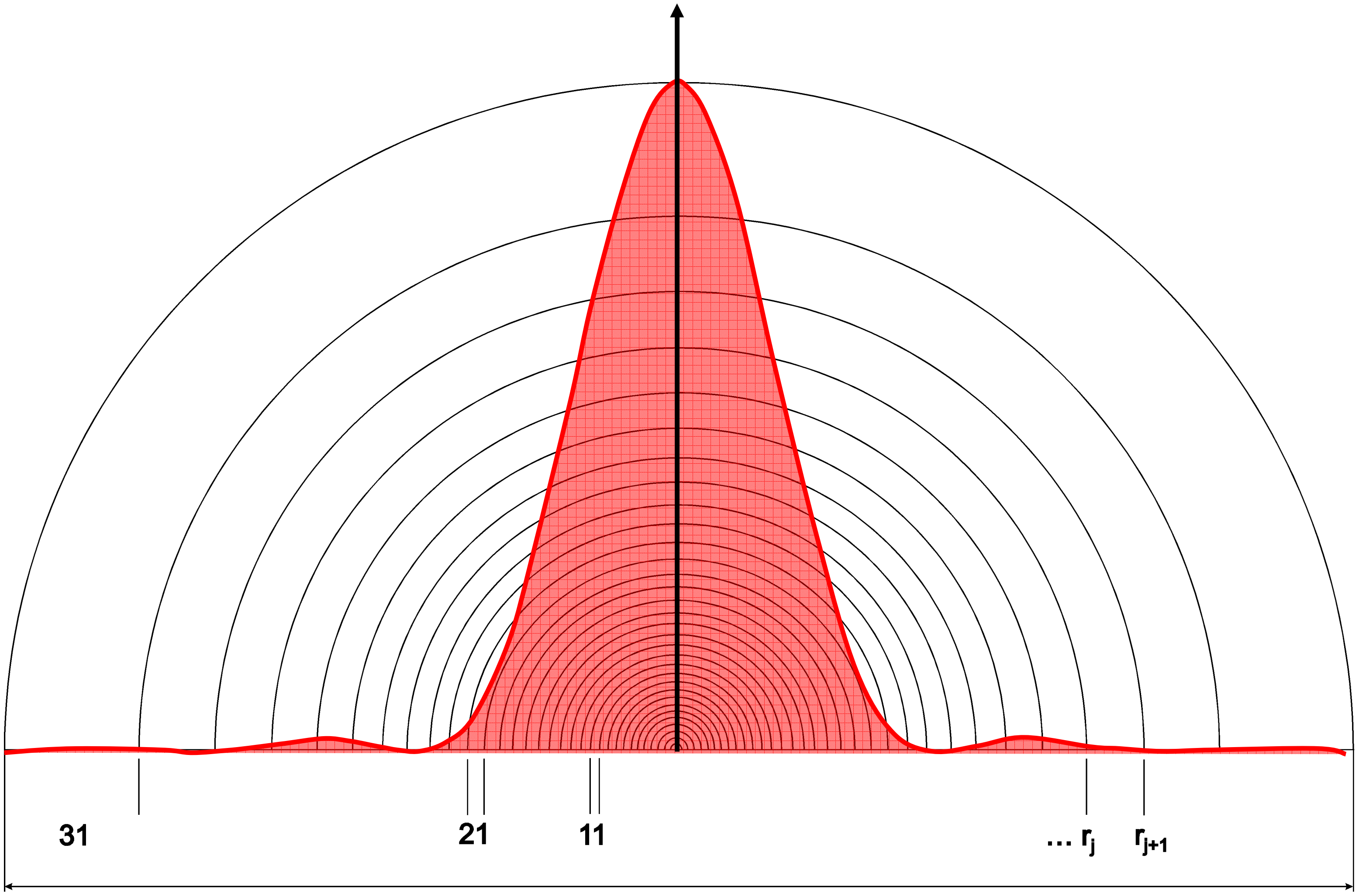
$$r_0 = 1.22\,\frac{\lambda \cdot f}{x}$$

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

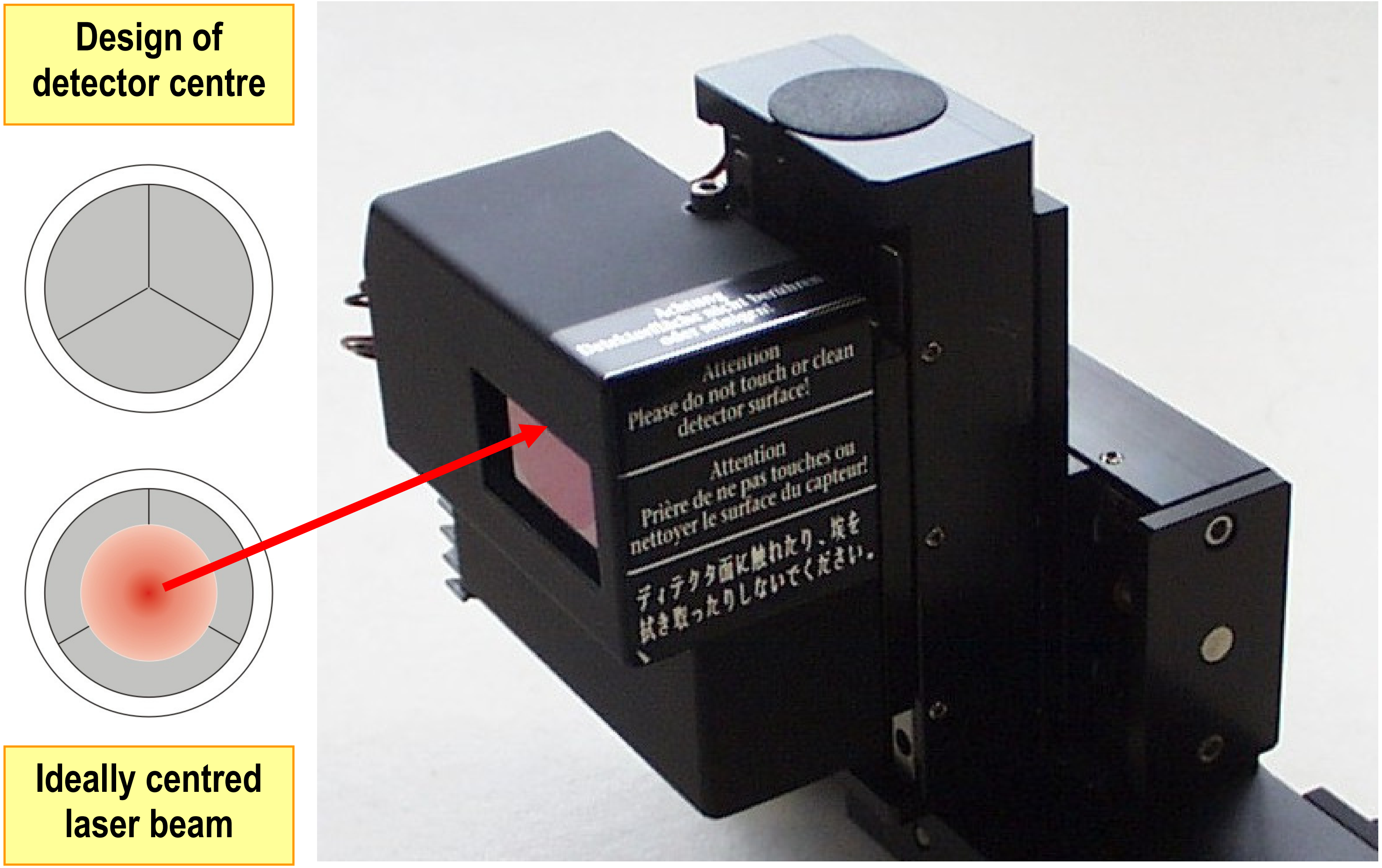
single particle of diameter x

particle size distribution q_0 (x)

Intensity distribution of Fraunhofer diffraction patterns



Multi-element detector with 31 classes and mono disperse intensity projection

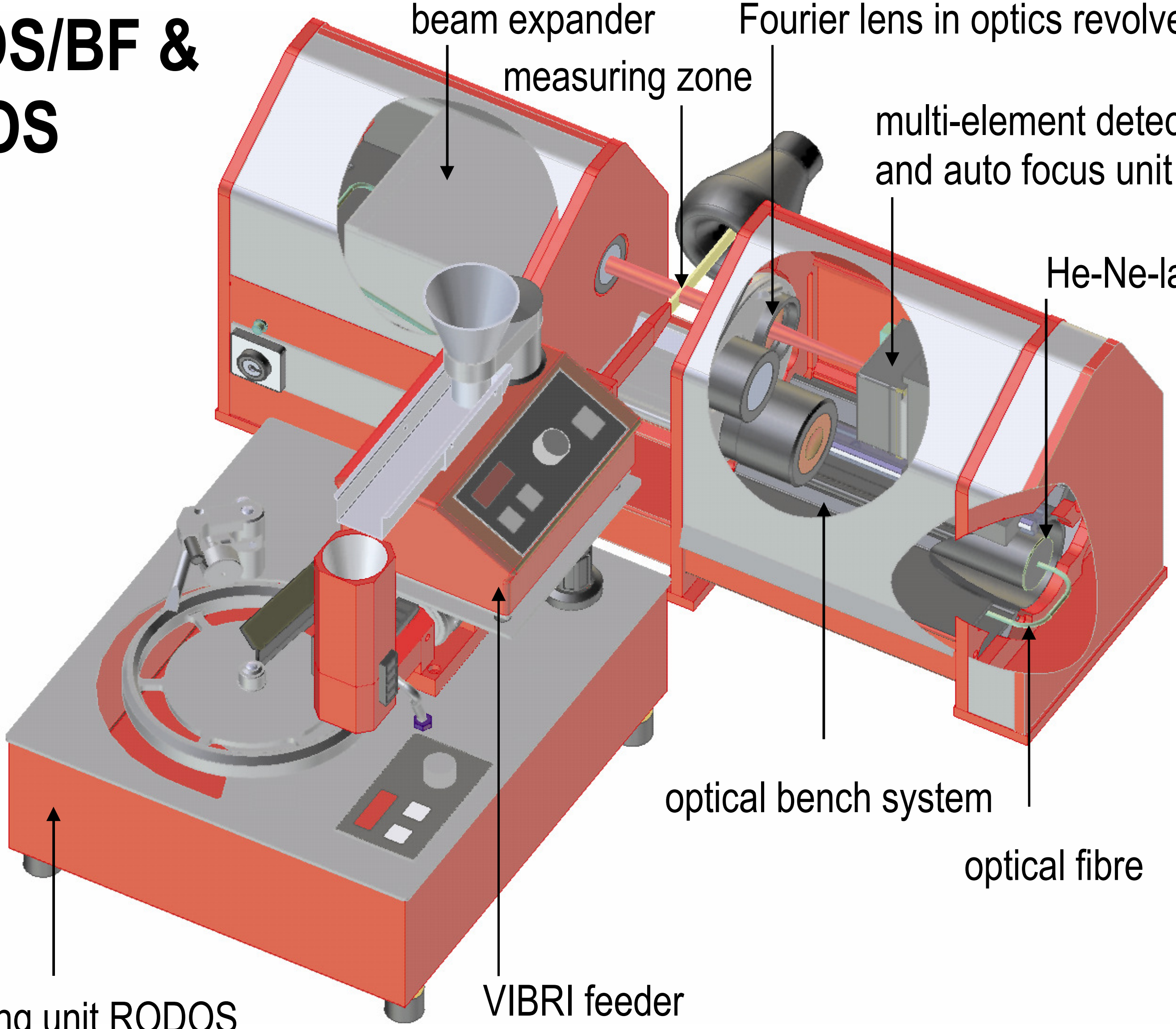


Design of detector centre

Ideally centred laser beam

HELOS detector and auto-focus unit

HELOS/BF & RODOS



beam expander

Fourier lens in optics revolver

measuring zone

multi-element detector
and auto focus unit

He-Ne-laser

optical bench system

optical fibre

VIBRI feeder

dry dispersing unit RODOS

Comparison of Particle Size Analysis Methods

year	method	dry	wet	x_{min} μm	x_{max} μm	Δx μm	$\Delta x/x_{min}$	t_A s	$\Delta x/t_A$ $\mu m/s$	invest. T€	Ω $1/(T€\cdot\mu m\cdot s)$
1900	sieving tower	x		100	10.000	9900	99	3600	2,75	5	1,38E-05
1925	sedimentation balance		x	1	100	99	99	150.000	0,0007	13	1,32E-05
1930	pipette		x	1	100	99	99	50.000	0,0020	8	6,60E-05
1950	wet sieving		x	5	200	195	39	30.000	0,0065	13	5,20E-06
1960	air jet sieve	x		30	1000	970	32	10.000	0,0970	15	1,80E-06
1965	x-ray sedimentation		x	0,5	100	100	199	7200	0,0138	40	3,69E-04
1972	laser diffraction		x	1	200	199	199	1500	0,1327	45	7,40E-04
1985	laser diffraction	x		0,5	1750	1750	3499	100	17,50	65	2,80E-01
1987	laser diffraction		x	0,1	100	100	999	60	1,67	50	8,40E-01
1988	x-ray sedimentation		x	0,1	300	300	2999	2500	0,1200	40	7,50E-03
1988	laser diffraction		x	0,1	700	700	6999	200	3,50	45	1,95E+00
1990	laser diffraction	x		0,5	2625	2625	5249	10	262	65	4,04E+00
1993	laser diffraction	x		0,25	3500	3500	13.999	5	700	60	4,48E+01
1995	laser diffraction	x		0,1	8750	8750	87.499	5	1750	75	5,90E+02
1996	laser diffraction	x	spray	0,5	175	175	349	5E-04	349.000	50	7,00E+03

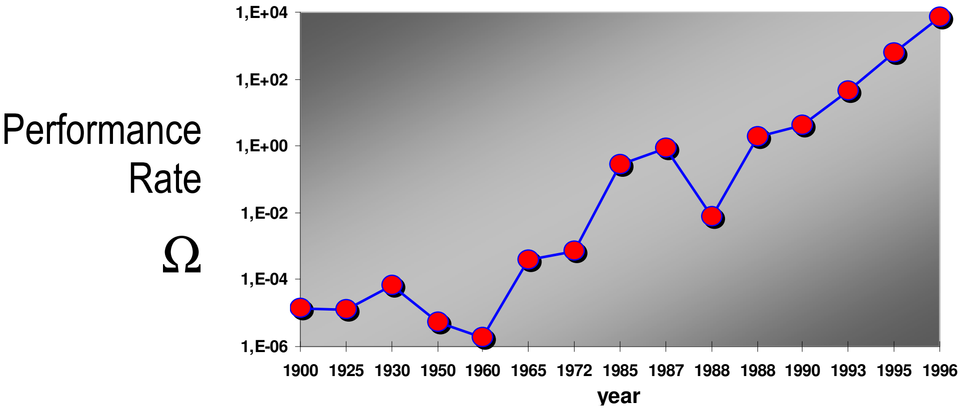
$$\Omega = \frac{\Delta x / x_{\min}}{x_{\min} \cdot t_A \cdot i} \quad [(€ \cdot \mu m \cdot s)]^{-1}$$

Δx : Measuring range (μm)

x_{min} : Finest particle size

t_A : Measuring time

i : Cost for investment



Application of Laser Diffraction

Stadium decade	Sensor & size ranges	Dispersing systems & product consciousness	Sampling & process coupling
Introduction 1970	Basic application 1 - 200 µm	Suspension period	Exclusively via laboratory
Penetration 1980	Sub-micron and extra millimetre 0.1–2000 µm	Fruitful dry period	Coupling with transport systems and samplers
Propagation 1990	Completion of extension 0.1 – 8750 µm	Robotic era	Cultivation With sample couplers
Qualification 2000	Standardisation ISO 13320 CFR21Part11	Certification validation	Completion

Fraunhofer Extension for Submicron Application

Examination of lower limit of Fraunhofer solution:

- ★ Established limit: $\alpha \gg 1$ (Mie factor $\alpha = \pi x/\lambda$)

  what does it mean ">>" ?
  is ">" possible ?
  or even "≥" ?

- ★ Study of the boundary condition: $\alpha = 1$

  $\lambda = 632.8$ nm $\Rightarrow x = 0.20$ µm
  for liquids applies: $\lambda = \lambda_0/n$

- ★ Examples:

Liquid	Water	Tetrachlor ethylene
Refractive index	1.330	1.504
Wave length [nm]	476	1.504
x ($\alpha = 1$) [µm]	0.151	0.134

- ★ Advantages of Fraunhofer extension to 0.1 µm
  - ✩ Independence of optical constants
    - ✳ No need for determination
    - ✳ No unknown relations
    - ✳ No influence of changes on the result
  - ✩ Only faint influence of shape on Fraunhofer solution
  - ✩ Improved sensitivity due to less intensive matrix smoothing

The Theory of Mie

Conditions for Application of Mie's Theory to Particle Size Analysis

- ★ Knowledge of complex refractive index *m* = n - ik, comprising
  Refractive index (n) and absorption coefficient (ik)
- ★ Problems related to application of Mie's theory for particle size analysis:
  - ✰ Related to the particles:
    - ✳ Applicable to spherical particles only
    - ✳ Unknown refractive index and absorption coefficient
    - ✳ Changes of the refractive index during the production process due to concentration and temperature
    - ✳ Mixtures of particles with different m and unknown mixing ratio
    - ✳ Influence of coated particles
  - ✰ Related to the mathematical algorithm:
    - ✳ Unpredictable consequences on algorithm for non-spherical particles
    - ✳ Highly different matrices due to smallest changes of *m*
    - ✳ New matrix determination in any case
    - ✳ Poor conditioning of matrices requests intensive smoothing procedure

HELOS MIE Software module

- ★ Complete realisation of exact Mie Theory
- ★ Matrices determined for continuous range of complex refractive indices *m* = n – ik
  - ☆ Available for all known materials
- ★ Evaluation time per set of parameters ≤ 1 second
- ★ Virtually no delay time
  - ☆ Study of parameter variations for extremely wide boundaries feasible
- ★ Easy selection of Mie parameters via HELOS software menu
- ★ Available for focal distances: f = 20, 50, 100, 200 mm
- ★ Available for laser wave lengths: λ = 632.8, 670, 685 nm
- ★ Requires not more than 5 Mbytes disk space per size range
- ★ Software tool:
  - ☆ Applicable to all HELOS sensor configurations
  - ☆ No hardware modifications necessary

$0.01 \leq n \leq 3.0$

$0.00 \leq k \leq 5.0$

HELOS MIE Software module

Check List:

☑ Refractive index *n* of particles and fluid known ?

☑ Absorption coefficient *k* of particles known ?

☑ No mixtures of different components ?

☑ Spherical particles ?

☑ Smooth particles surface ?

↳ Application of HELOS MIE Software for most precise results

However:

Due to rare availability of requested parameters, in practice very few cases of useful application only, e.g. beads, droplets