Determination of Water Pollution in various Applications with a Focus to Determination of Particles

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(Contact: See last slide)

Contents 1

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Different kinds of water pollution

Solved pollution

High / low concentration

Particle pollution

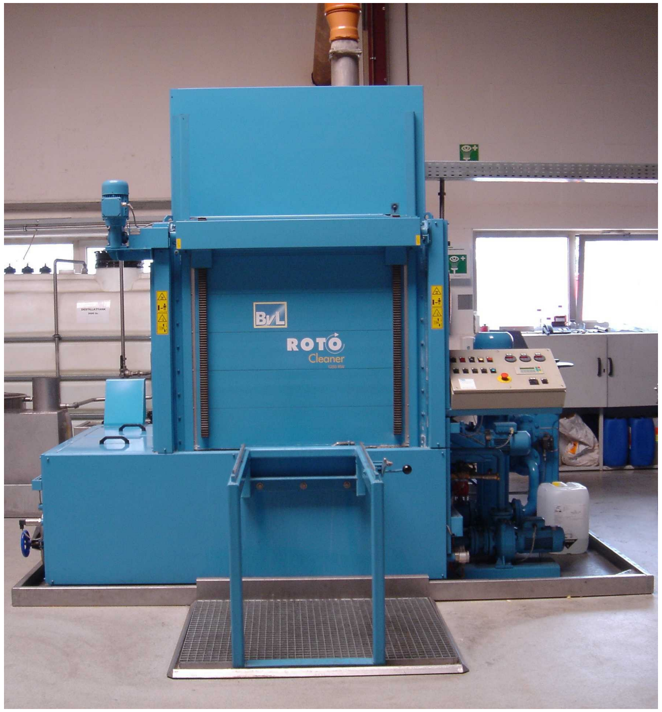
High concentration (e.g. industrial washers)

Low concentration (e.g. Clean room applications)

High concentration
The Range of industrial washers

Shoe box Size | Case Size | Room Size

Medicine / Pharmacy
e.g. Injections cleaning



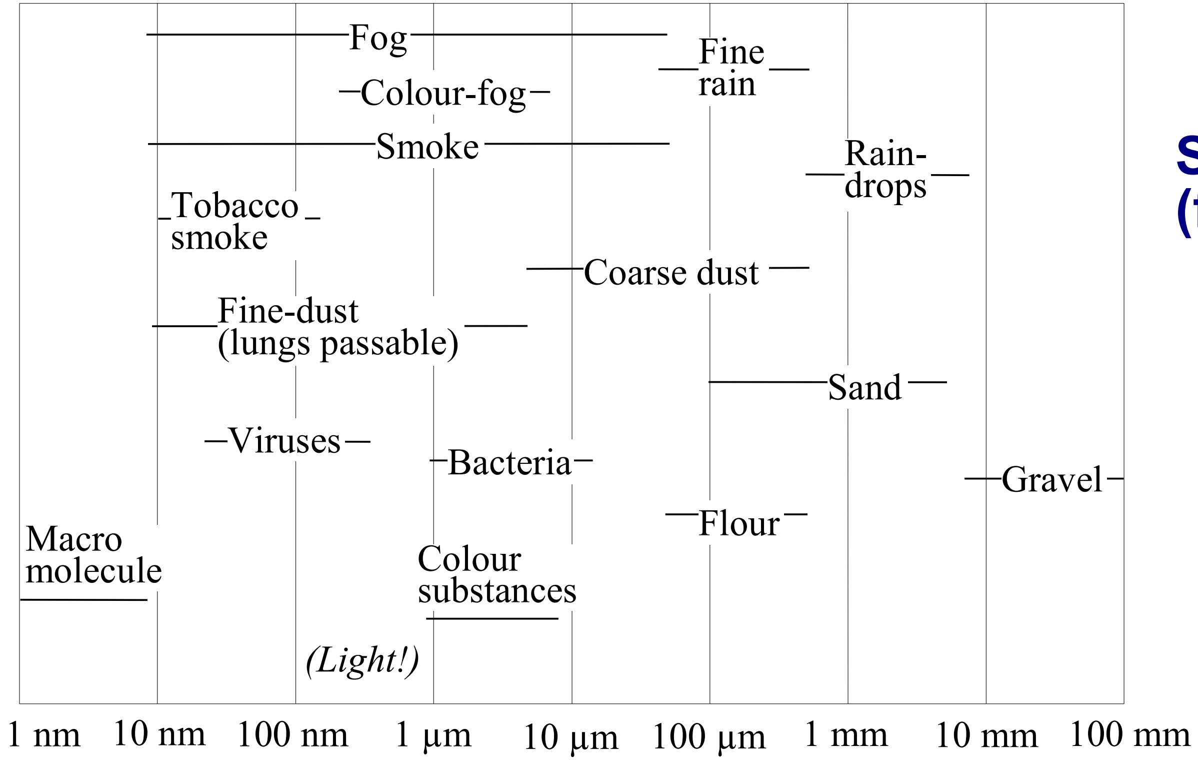
Cars and Railway washers

Metal processing industry
e.g. Cleaning of produced parts

Low concentration

- Clean room monitoring
- Particle in Volume check
- Particle on surface check

Size Ranges



Special Expressions (for example):

- **Molecular-dispersion**
- **Fine-dispersion**
- **Coarse-dispersion**

Targets of measurements

- particle size distribution
- particle concentration
- particle speed
- flowability and parameters that have influence on the flowability

... and with this parameters correlated parameters.

Calculation, presentation and interpretation of (particle) measurement results

Different definitions of the size

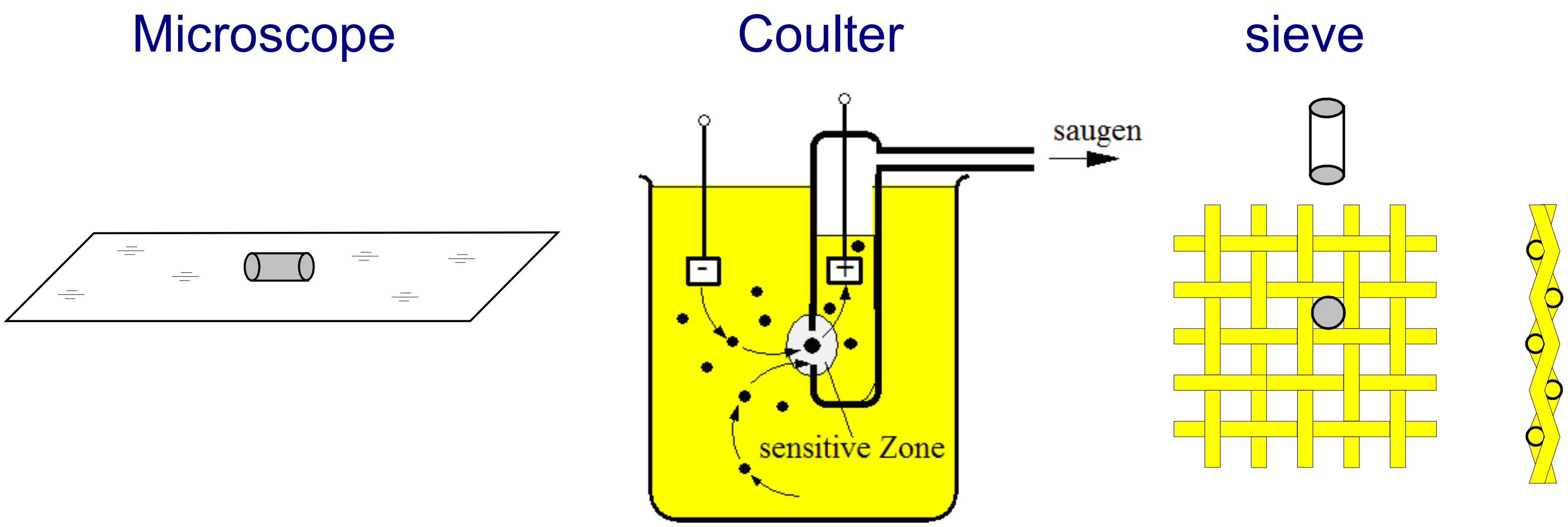
Several criterion's of size:

- Diameter of a sphere with equal volume,
- Diameter of a sphere with equal projection area,
- Diameter of a sphere with equal light scattering,
- Diameter of a sphere with equal settling velocity,
- Diameter of a sphere with equal smallest through going diameter

If the particles are non-spherical (normally), different measurement techniques will lead to different results, by reason of different criterion's of size.

These are not errors!

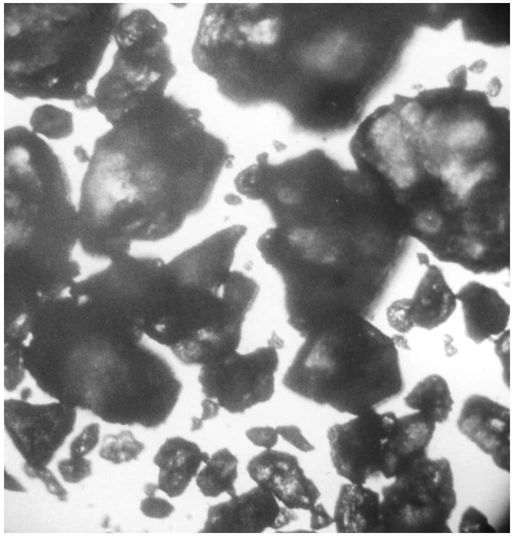
Examples for different results



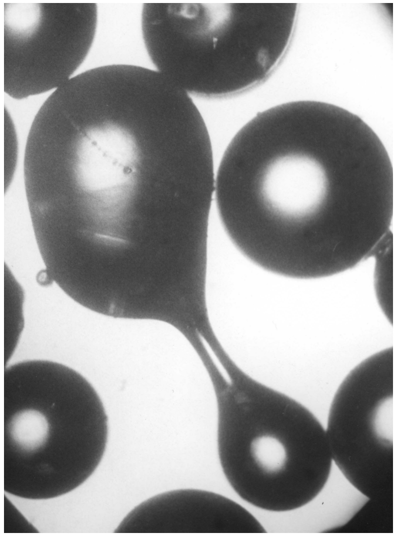
Example: cylinder Ø 10µm, l=20µm

Microscope: shadow area = 200µm² → d_S = 16µm
Coulter: volume = 1570 µm³ → d_S = 14,4µm
Sieve: mesh width = 10µm → d_S = 10µm

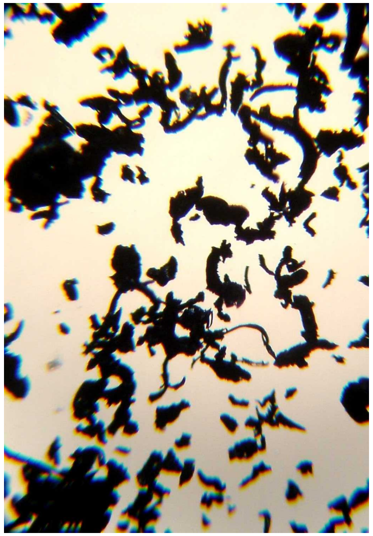
Examples for different shapes of particles



Sand

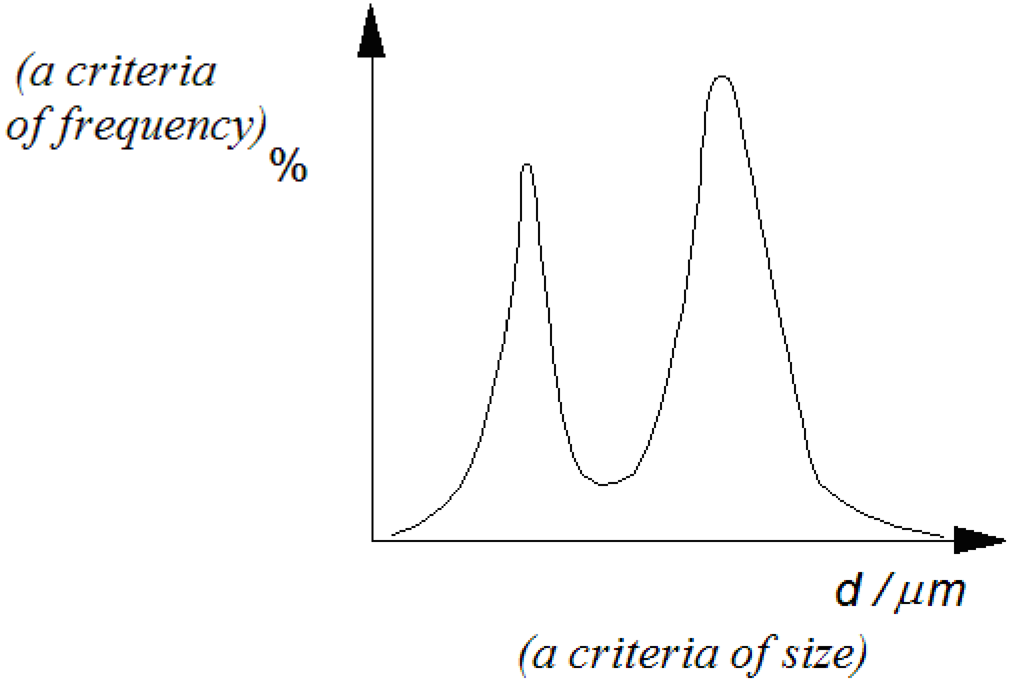


Glass



Metal

Presentation of measurement results



Particle size distribution: drawing of a criteria of the particles frequency over a criteria of size.

Criterions of frequency

Number frequency: Specify, how many particles from the total number (for example in %) are in the several classes of size.

Surface frequency: Specify, how many parts from the total surface (for example in %) are in the several classes of size.

Volume (or mass) frequency: Specify, how many parts from the total volume (for example in %) are in the several classes of size.

- The function of the mass frequency is like the function of the volume frequency because the mass is the volume multiplied by a factor (density).

- The representing of the results is possible as histogram or as cumulative histogram.

Histogram - 1

The histogram enables an easy and fast overview of the distribution of results.
The ranges in which the values of a series of measurements are divided into equal classes with a width of *Y*. The number of measurement results will be assigned to these classes in whose range they are. It follows the absolute frequency H_{abs_i} , that means the number of measurement values per class.

The absolute frequency of a class divided by the total number of the measurement values *n* results in the relatively frequency H_i:

$$H_i = \frac{n_i}{n}$$

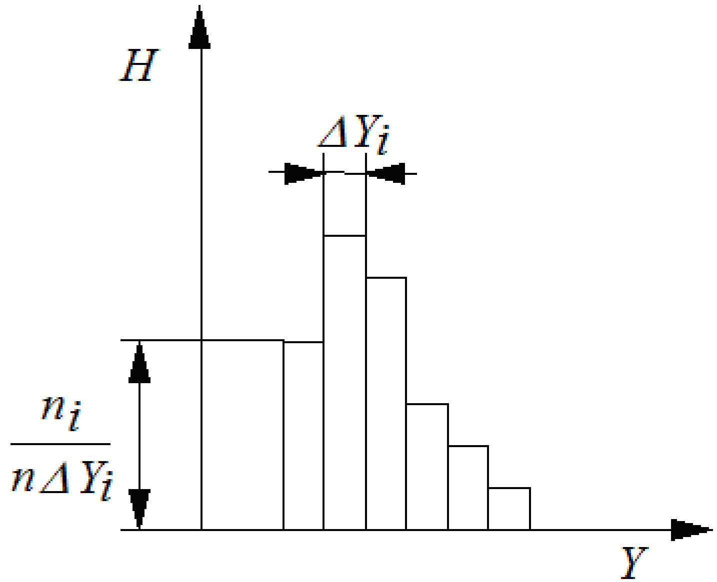
If the class width is unequal, then relative frequency of the class must be divided by the respectively class with. So follows the relative frequency, standardized by the class width:

$$H_i = \frac{n_i}{n \Delta Y_i}$$

Histogram - 2

Direction for the number of classes: $k \approx \sqrt{n}$

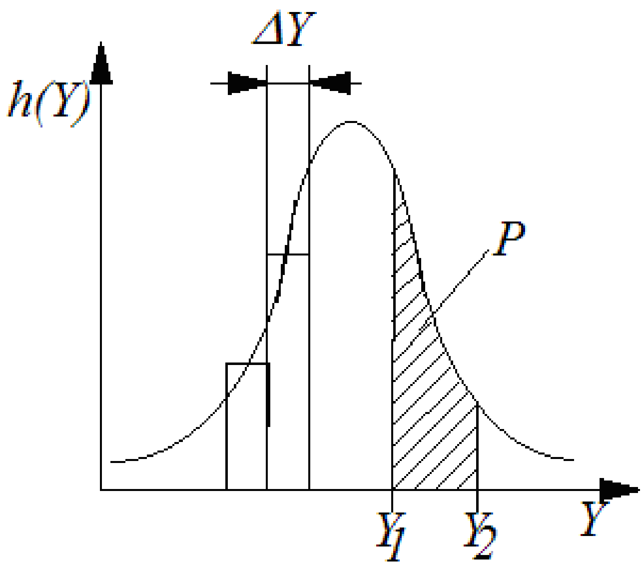
Example:
10.000 measurement values must be presented as an histogram.
The number of classes *k* should
be in maximum round about: $\sqrt{10.000} = 100$



Density Funktion

With increasing number of measurement values the histogram converges towards the density function: $h(Y) = \lim_{\Delta Y \to 0} \left(\lim_{n \to \infty} H(Y) \right)$

For decreasing values of ΔY and increasing number of n would generate the following function:



The number of particles (equal to the possibility P) in the range Y_1 to Y_2 can be calculated:

$$P(Y_1 < Y \leq Y_2) = \int_{y_1}^{y_2} h(Y)dY$$

For decreasing values of ΔY and increasing number of n is the histogram approximately the density function.

Cumulative Histogram

The cumulative histogram enables an easy and fast means to have an answer to the question how many of the measurement results are over or under a limitation (e.g. Quality management)

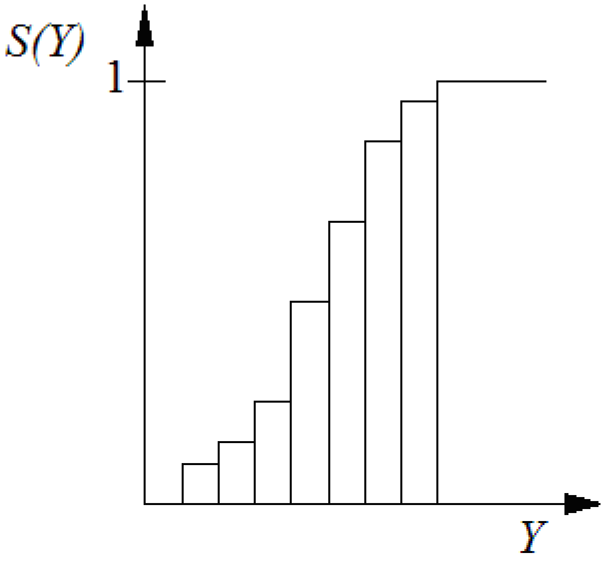
The cumulative histogram will be generated by the cumulating of the relative frequency, starting by the lowest class:

$$S(Y) = \sum_{i=1}^{k} \frac{n_i}{n}$$

that means:

$$S(Y)\big|_{Y \geq Y_{max}} = 1$$

k is the class from *Y*.



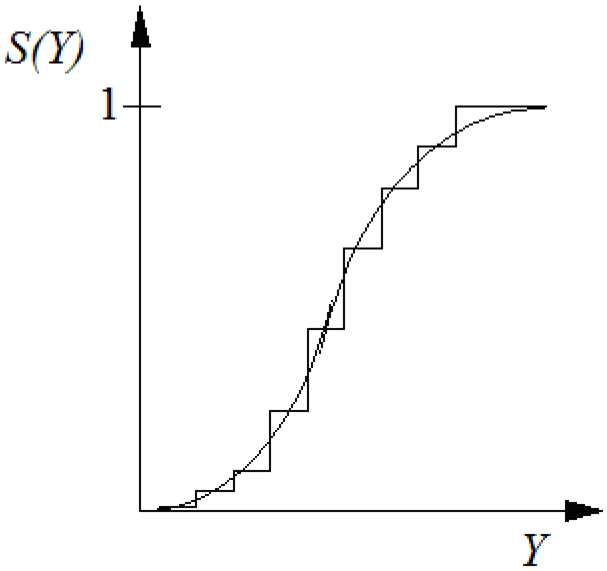
Cumulative density function

With increasing number of measurement values the cumulative histogram converges towards the cumulative density function:

$$s(Y) = \lim_{\Delta Y \to 0} \left(\lim_{n \to \infty} S(Y) \right)$$

For decreasing values of ΔY and increasing number of n would generate the following function:

For decreasing values of ΔY and increasing number of n, the cumulative histogram is approximately the cumulative density function.

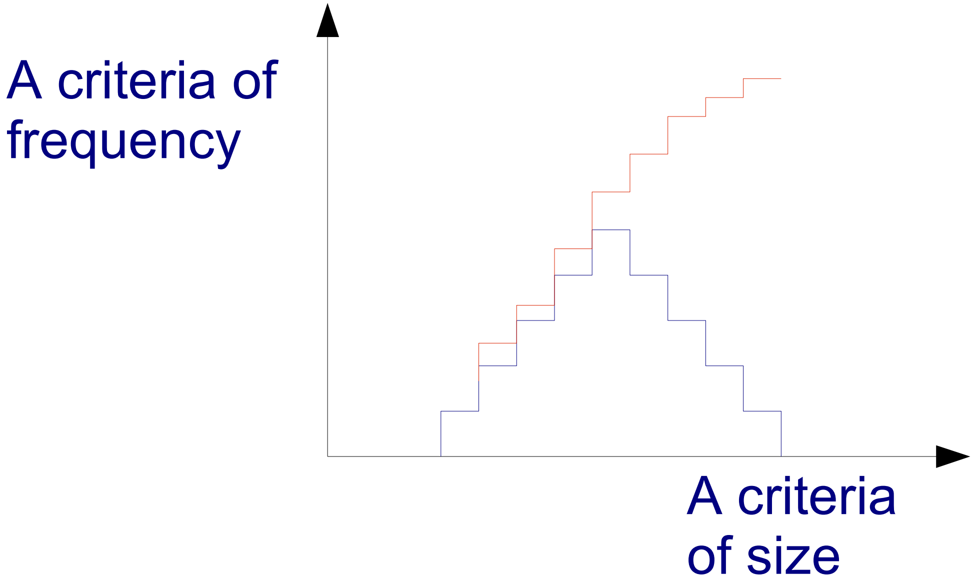


Important parameters

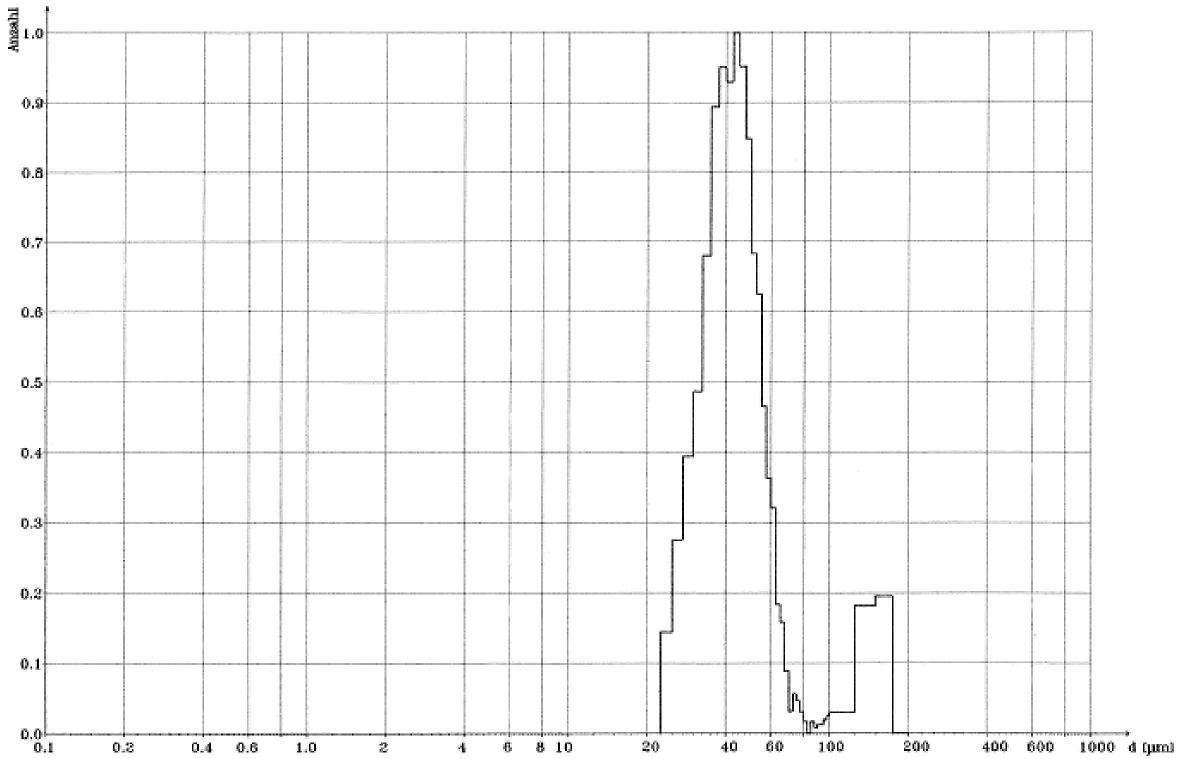
- **Mean value**
- **Standard deviation**
- **Modal value** (most frequently class)
- **Median value** (particle size, where 50% of the particles are smaller and 50% of the particles are larger)

Possibilities of Presentation

- X-Axis: A criteria of size (depend from the method)
- Y-Axis: A criteria of frequency (free choice)
- Presentation as Histogram or Cumulative Histogram (free choice)



Number Distribution



Datum: 6.6.1996 Uhrzeit: 11:16 Meß-Datei: bimcoul.dat Kalibrier-Datei: coulv5.kal Softwareversion: 2

Meßzeit: 120.00 sec
Anzahl der Partikeln: 6907
Schwellwert = 50 mV
kleinstes Partikel = 23.75 µm (0.9792 %)
größtes Partikel = 162.50 µm (13.2164 %)
Kommentar:
bimodal Sand <63µm und 125 – 180µm

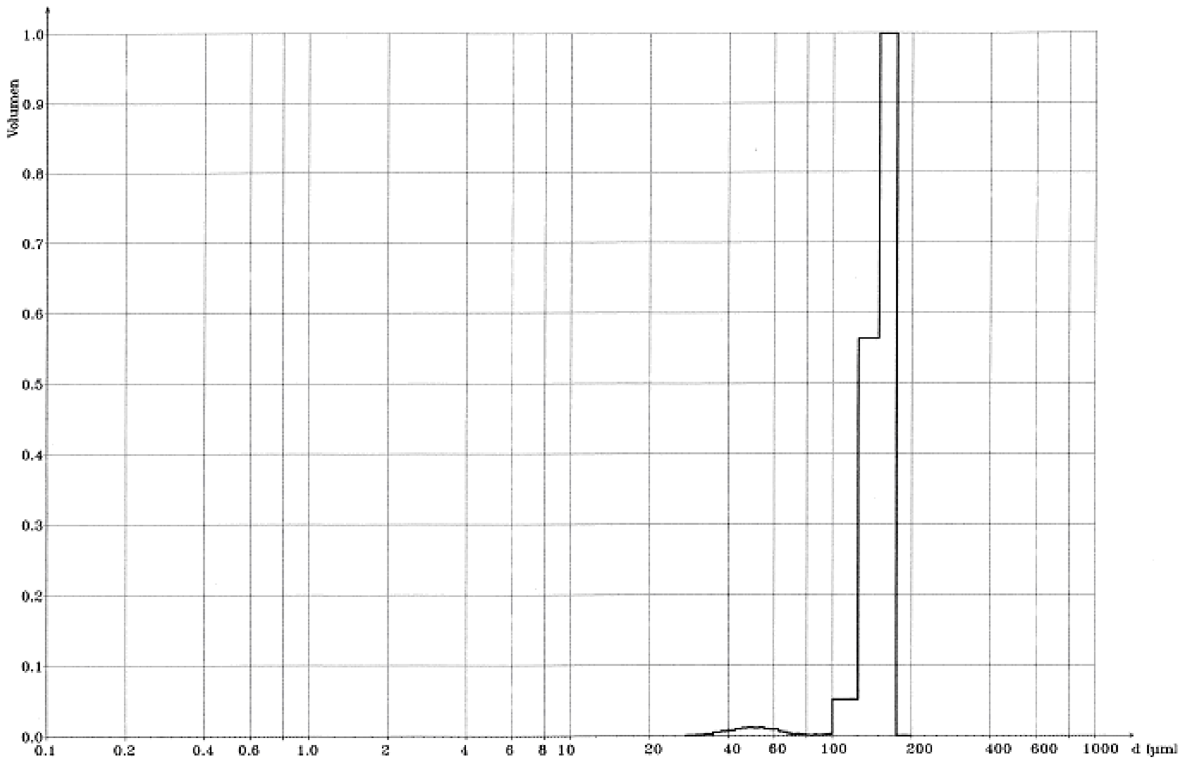
Anzahlverteilung

Modalwert = 43.75 µm (6.75 %)
Mittelwert = 73.38 µm
Standardabweichung = +/- 47.58 µm

Volumenverteilung

Modalwert = 162.50 µm (61.37 %)
Mittelwert = 151.33 µm
Standardabweichung = +/- 16.56 µm

Volume Distribution



Datum: 6.6.1996 Uhrzeit: 11:17 Meß-Datei: bimcoul.dat Kalibrier-Datei: coulv5.kal Softwareversion: 2

Meßzeit: 120.00 sec
Anzahl der Partikeln: 6907
Schwellwert = 50 mV
kleinstes Partikel = 23.75 µm (0.9792 %)
größtes Partikel = 162.50 µm (13.2184 %)
Kommentar:
bimodal Sand <63µm und 125 – 180µm

Anzahlverteilung

Modalwert = 43.75 µm (6.75 %)
Mittelwert = 73.38 µm
Standardabweichung = +/– 47.58 µm

Volumenverteilung

Modalwert = 162.50 µm (61.37 %)
Mittelwert = 151.33 µm
Standardabweichung = +/– 16.56 µm

Results

Tabellarisches Meßergebnis Seite 1

Datum: 6.6.1996 Uhrzeit: 11:18
Meß-Datei: .dat Kalibrier-Datei: coulv5.kal
Softwareversion: 2

Kommentar: bimodal Sand <63µm und 125 – 180µm

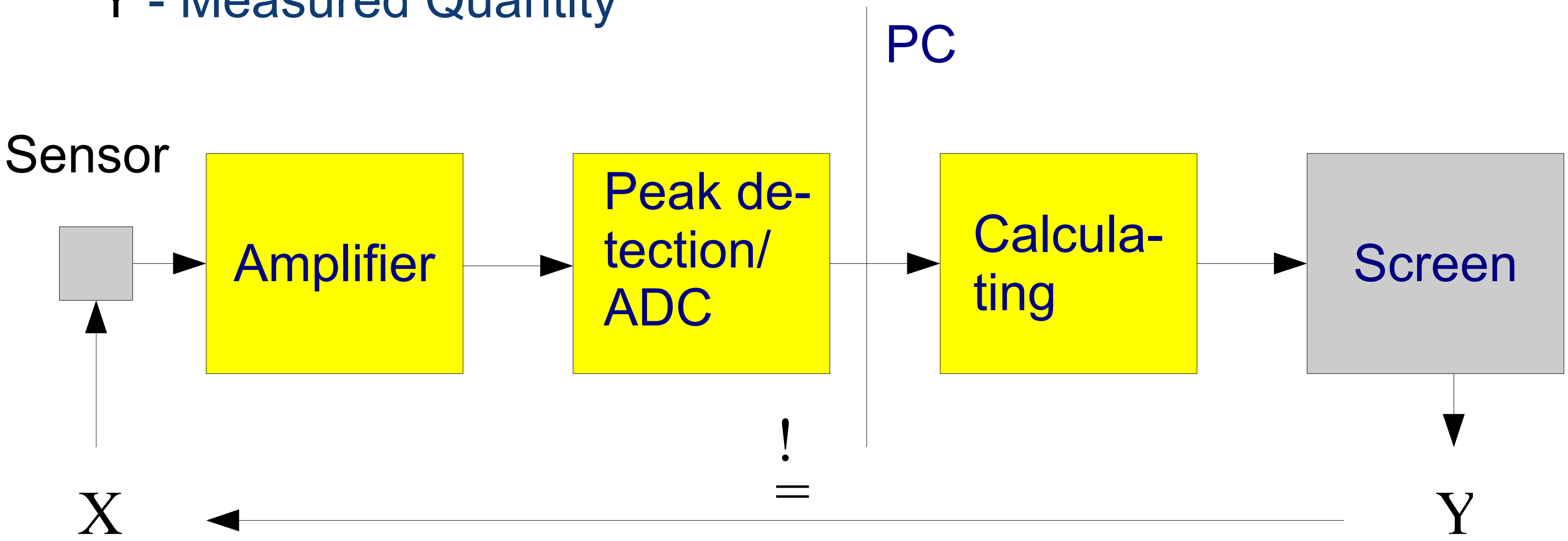
Klassen-obergrenze	Histogramm		Summenhäufigkeit	
	Anzahl	Volumen	Anzahl	Volumen
x0/µm	q0/%	q3/%	Q0/%	Q3/%
17.50	0.00	0.00	0.00	0.00
20.00	0.00	0.00	0.00	0.00
22.50	0.00	0.00	0.00	0.00
25.00	1.30	0.01	1.30	0.01
27.50	2.47	0.03	3.77	0.05
30.00	3.54	0.06	7.31	0.11
32.50	4.37	0.10	11.68	0.21
35.00	6.11	0.18	17.79	0.39
37.50	8.03	0.29	25.82	0.68
40.00	8.54	0.37	34.35	1.05
42.50	8.35	0.44	42.71	1.49
45.00	8.98	0.57	51.69	2.06
47.50	8.55	0.64	60.24	2.70
50.00	7.62	0.67	67.86	3.36
52.50	6.13	0.62	73.99	3.99
55.00	5.61	0.66	79.60	4.64
57.50	4.18	0.56	83.78	5.20
60.00	3.27	0.50	87.05	5.70
62.50	2.88	0.50	89.93	6.20
65.00	1.65	0.32	91.59	6.53
67.50	1.43	0.31	93.01	6.84
70.00	0.80	0.20	93.82	7.04
72.50	0.29	0.08	94.10	7.12
75.00	0.51	0.15	94.61	7.27
77.50	0.42	0.14	95.04	7.41
80.00	0.28	0.10	95.31	7.51
82.50	0.15	0.06	95.47	7.58
85.00	0.00	0.00	95.47	7.58
87.50	0.15	0.07	95.62	7.65
90.00	0.08	0.04	95.70	7.69
92.50	0.12	0.07	95.81	7.76
95.00	0.12	0.07	95.93	7.83
97.50	0.18	0.12	96.11	7.95
100.00	0.23	0.16	96.33	8.11
125.00	0.27	2.93	96.61	11.04
150.00	1.64	32.07	98.24	43.11
175.00	1.76	56.89	100.00	100.00
200.00	0.00	0.00	100.00	100.00
225.00	0.00	0.00	100.00	100.00
250.00	0.00	0.00	100.00	100.00

Computer based calculating of particle size or concentration

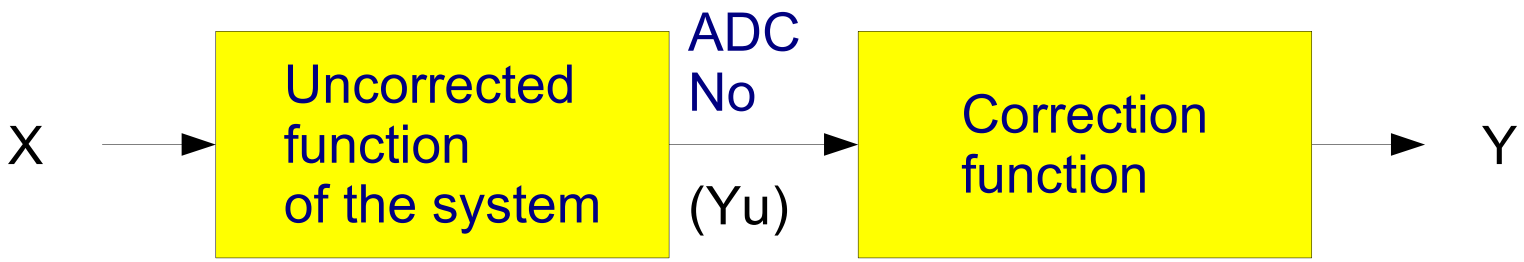
Target: $Y = X$

X - Measure Quantity

Y - Measured Quantity



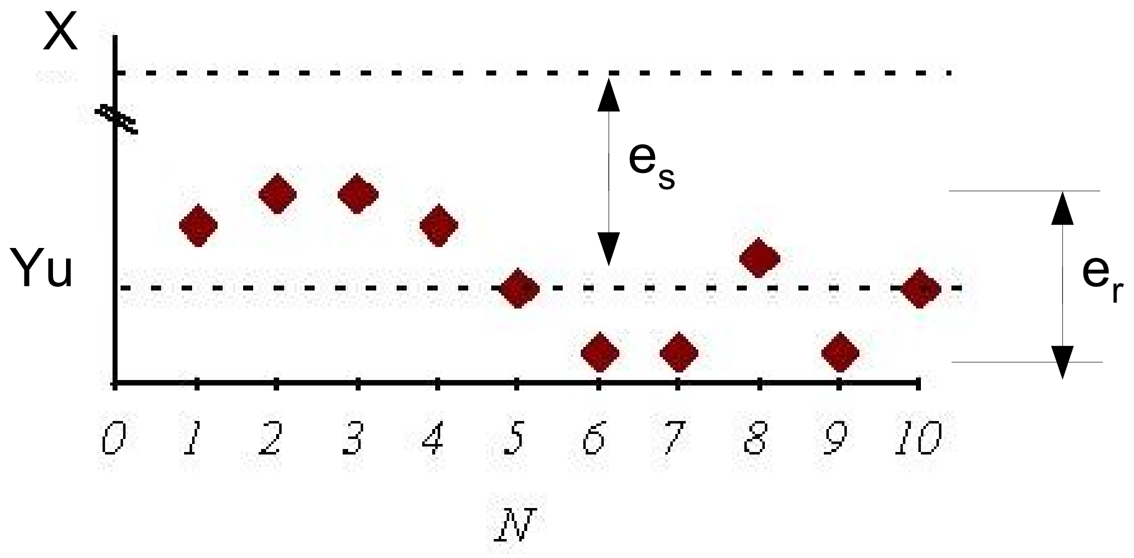
Description of the system



It is measured:

X in µm	Yu
0	4
50	166
100	652
150	1461
200	2595
250	4052

Random Errors



The mean of the scattering values represent the systematic part of the error. From this, Yu is this mean.

Calculation of the results

A) Calculating of the parameter of the uncorrected function between every two points

$$Y_u = mX + n \quad :$$

$$m = \frac{\Delta Y_u}{\Delta X}$$

$$n = Y_u - mX$$

B) Calculating of the parameter of the correction function between every two points

$$Y = K_m (Y_u + K_n) \quad :$$

$$K_m = \frac{1}{m}$$

$$K_n = -n$$

Software to be completed

40 Y_u = *(uncorrected Value)*

50 IF $Y_u < \ldots$ THEN $Y = \ldots * (Y_u + \ldots)$: GOTO 100

60 IF $Y_u < \ldots$ THEN $Y = \ldots * (Y_u + \ldots)$: GOTO 100

70 IF $Y_u < \ldots$ THEN $Y = \ldots * (Y_u + \ldots)$: GOTO 100

80 IF $Y_u < \ldots$ THEN $Y = \ldots * (Y_u + \ldots)$: GOTO 100

90 IF $Y_u \geq \ldots$ THEN $Y = \ldots * (Y_u + \ldots)$: GOTO 100

100 Print Y

Solution

		$Y_u = mX + n$		$Y = K_m (Y_u + K_n)$	
X [µm]	Y_u [N]	m [N/µm]	n [N]	K_m [µm/N]	K_n [N]
0	4				
		3.24	4	0.308642	-4
50	166				
		9.72	-320	0.1028807	320
100	652				
		16.18	-966	0.0618047	966
150	1461				
		22.68	-1941	0.0440917	1941
200	2595				
		29.14	-3233	0.0343171	3233
250	4052				

Software

Please test the Software with a 125 µm particle!

40 Y_u = *(uncorrected Value)*

50 IF $Y_u < 166$ THEN $Y = 0.308642 * (Y_u - 4)$: GOTO 100

60 IF $Y_u < 652$ THEN $Y = 0.1028807 * (Y_u + 320)$: GOTO 100

70 IF $Y_u < 1461$ THEN $Y = 0.0618047 * (Y_u + 966)$: GOTO 100

80 IF $Y_u < 2595$ THEN $Y = 0.0440917 * (Y_u + 1941)$: GOTO 100

90 IF $Y_u \geq 2595$ THEN $Y = 0.0343171 * (Y_u + 3233)$: GOTO 100

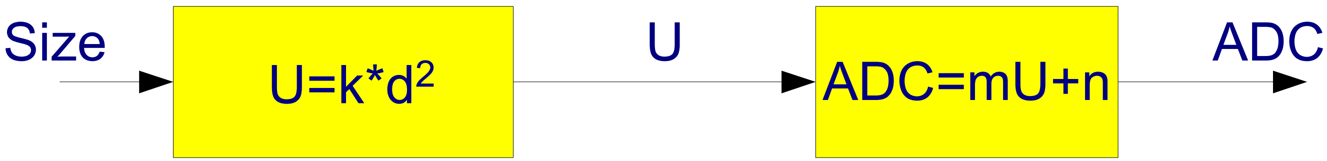
100 Print Y

Number of standards

- Advantage of this method: Every kind of systematic errors can be corrected.
- Disadvantage: For every calibration Point a standard is necessary.

► In many cases exist a known mathematical function and additional to this function nonlinearities, caused by technical reasons. Knowledge about a mathematical function can reduce the number of standards.

Known part of the function



$k=U/d^2$ = <u>0.00008</u>

m= ΔY/ ΔX = 4048/5.00 = <u>809.6</u>

n=ADC-mU = 4 - 809.6 * 0 = <u>4.0</u>

ADC=809.6*U+4.0

X in µm		U		ADC
0		0.000		4
50		0.200		166
100		0.800		652
150		1.800		1461
200		3.200		2595
250		5.000		4052

Pre Calibration

X	Yu	km	kn
0	0		
		$km = \frac{1}{\frac{\Delta Yu}{\Delta X}} = 3.9293413$	0
250	$\sqrt{4048}$		

```
10  Yu = (Number from ADC)
20  Yu = Yu - 4
30  Yu = SQR(Yu)
40  Y = 3.9293413 * (Yu+0)
50  Print Y
```

Please test the Software with a 125 µm particle!

Selected optical principles for particle size and/or concentration measurement

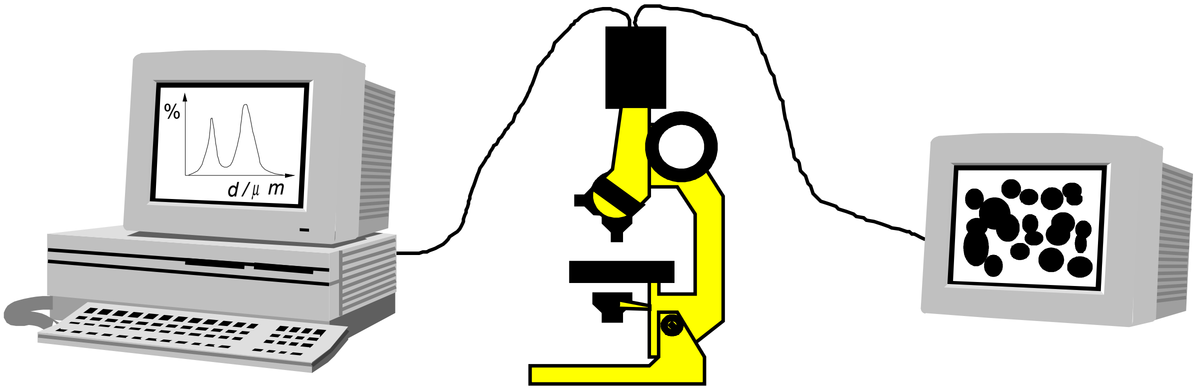
Microscopy

Hot points for microscope analysis:

1. Problem: Preparation of a slide with an equal dispersed sample
2. Problem: Statistical confidence (small number of particles)
3. Errors caused by the size depend sharpness, overlap and so on
4. Slow
5. ”Large” criteria of size

► But: Delivered a good overview about properties and behaviour of the particles.

Microscope analysis system



Computer

Microscope
with CCD - camera

Microscope image

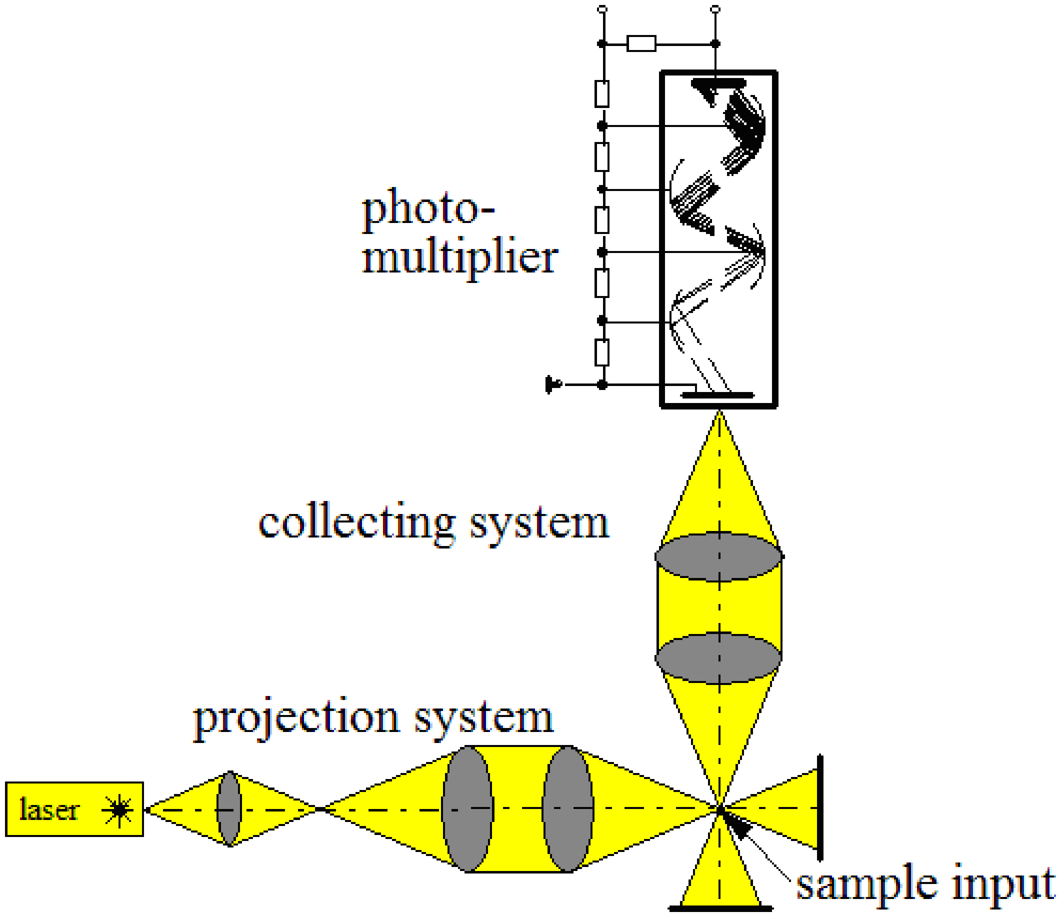
Size determination by Light scattering

Basis: Theory of Mie (1908): The amplitude of the scattering light of a particle is proportional to the size.

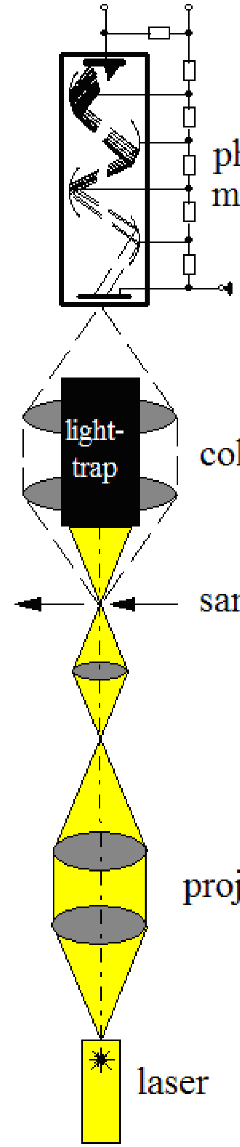
Advantage: Measurement volume are optical limited

Disadvantages: The amplitude of the scattering light is not only dependent on the size. It is also dependent on properties of materials and the used angle for receiving the scattering light.

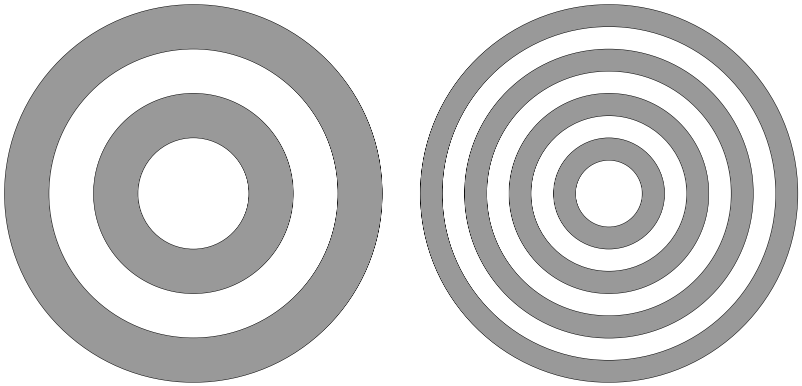
Measurement by 90°



Measurement by 0°



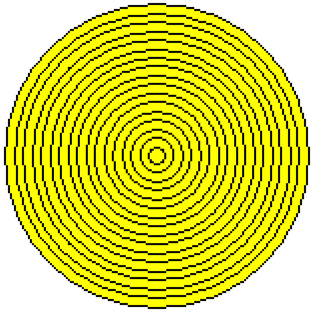
Size determination by Light Diffraction



Small particle large particle

Laser diffraction pattern from a single particle

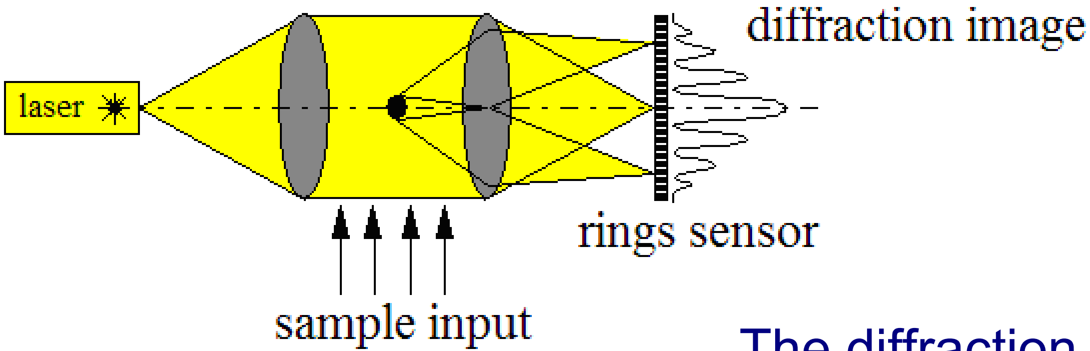
The sensor consist of photo diodes in the form of a ring, currently up to 64 pieces:



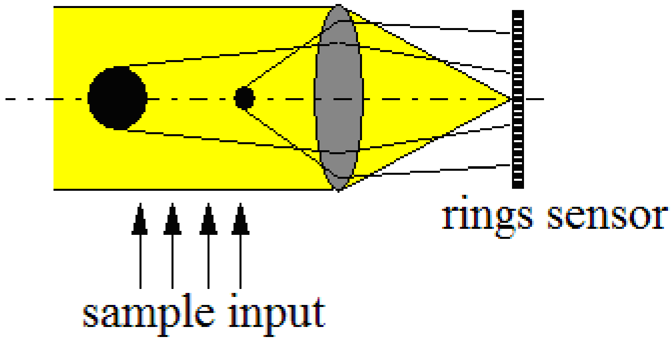
For better connection exist halve-ring designs with lower sensitivity.

- Use of the Fraunhofer diffraction
- Core: pattern sensor
- Strong decrease of the influence of the extinction coefficient
- Faster measurement / higher number of particles

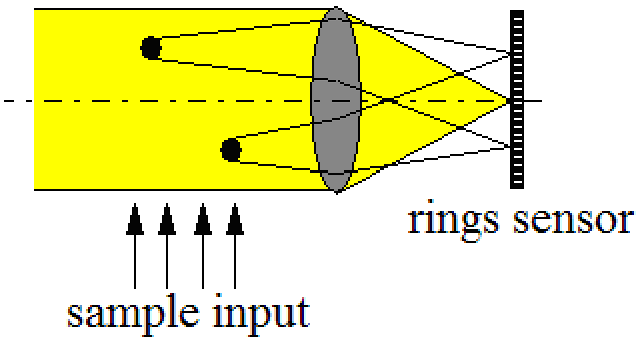
Recieving of the diffraction pattern



Size dependent position of the diffraction patterns:



The diffraction patterns of particles with equal size have an overlap, also when the particles are in motion:



Determination of the size distribution

Caused by the overlap it is possible to calculate from the intensity (as a function of the diameters of the sensor) the frequency of all particle classes. The simultaneous analysis of a large number of particles lead to a shorter measurement time and higher confidence of the results. In this case is required an equal extinction coefficient of all particles. Currently, a total measurement range of 0,1 µm - 3,5 mm is possible.

PCS

The photon correlation spectroscope (PCS) - also known as quasi-elastic light scattering; dynamic light scattering; intensity fluctuation spectroscopy.

Suitable for measurements in a range of about 1 nm to 3 µm.

Condition is, that the basis for changes in particle position is the Brownian motion.

PCS use the dependence of the *fluctuation* of the scattering light intensity from the size depended diffusion coefficient.

The signal, generated by the detector is like a noise signal due to the constantly changing diffraction pattern caused by destructive and constructive interference as the particles change their position.

Additional Methods

- Flight time principle
- Tyndallometer
- LDA and PDA

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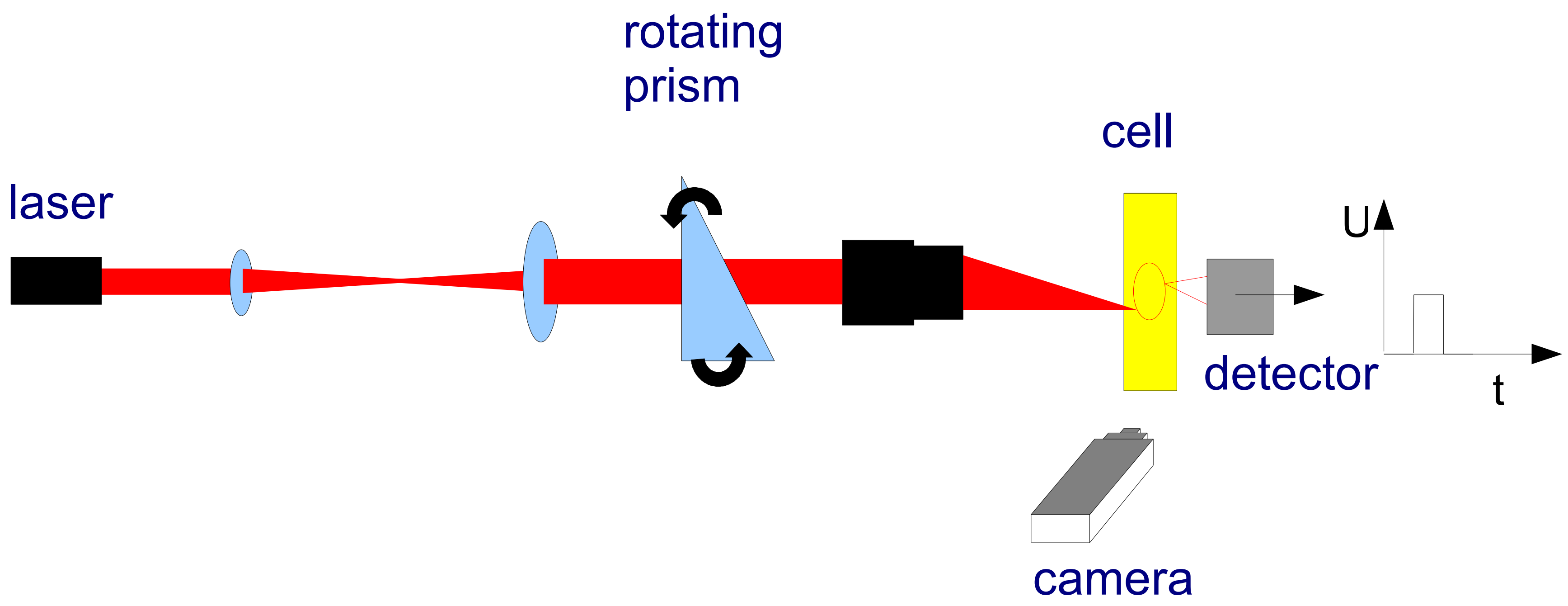
Sample or 100% Check?

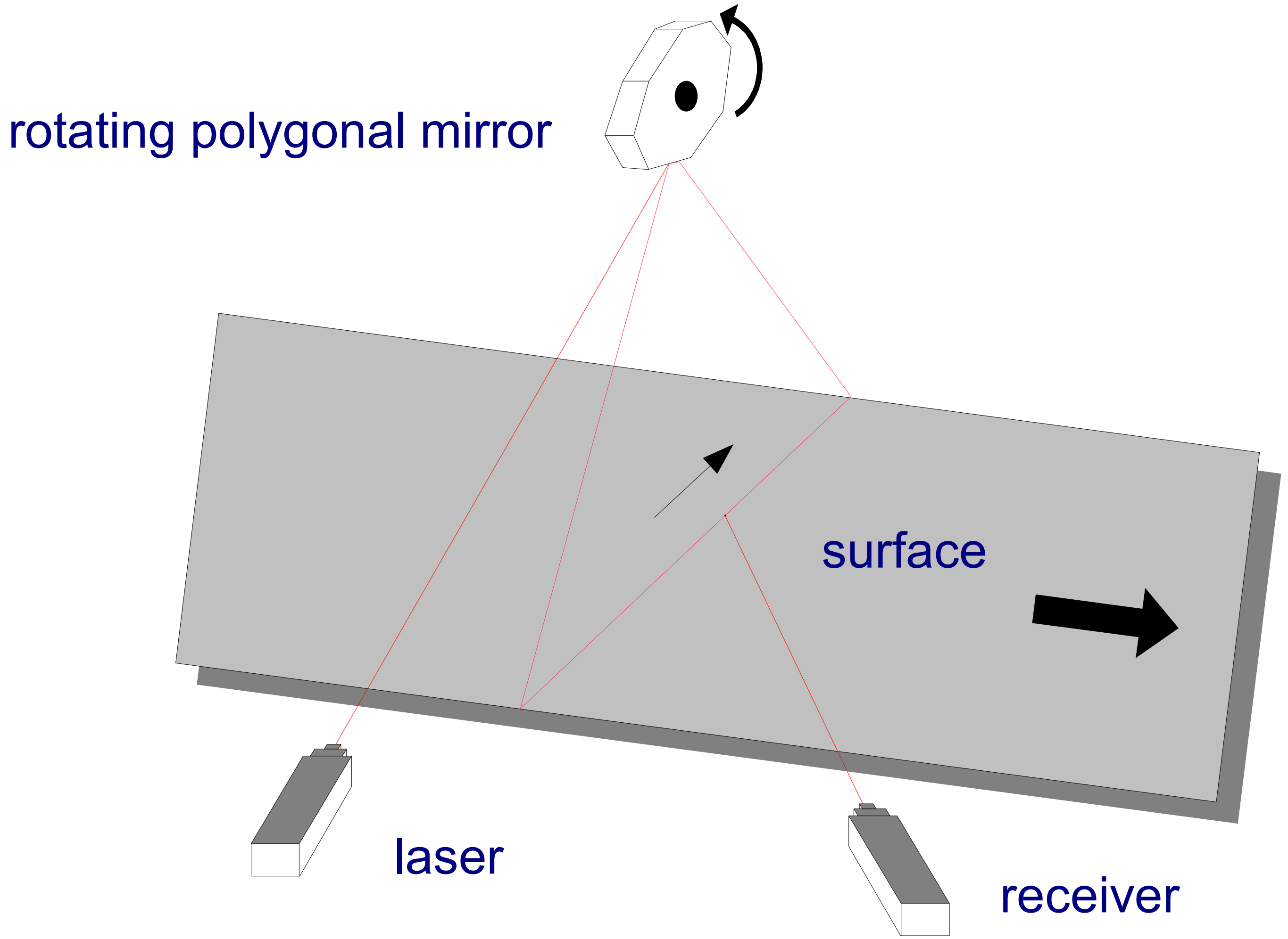
Sample: Determination of size distributions in suspensions, powders and so on.

100% Check: Water pollution for clean room applications, Surface control, determination of particle pollution in cuevettes in medicine and pharmacy.

Volume - Check

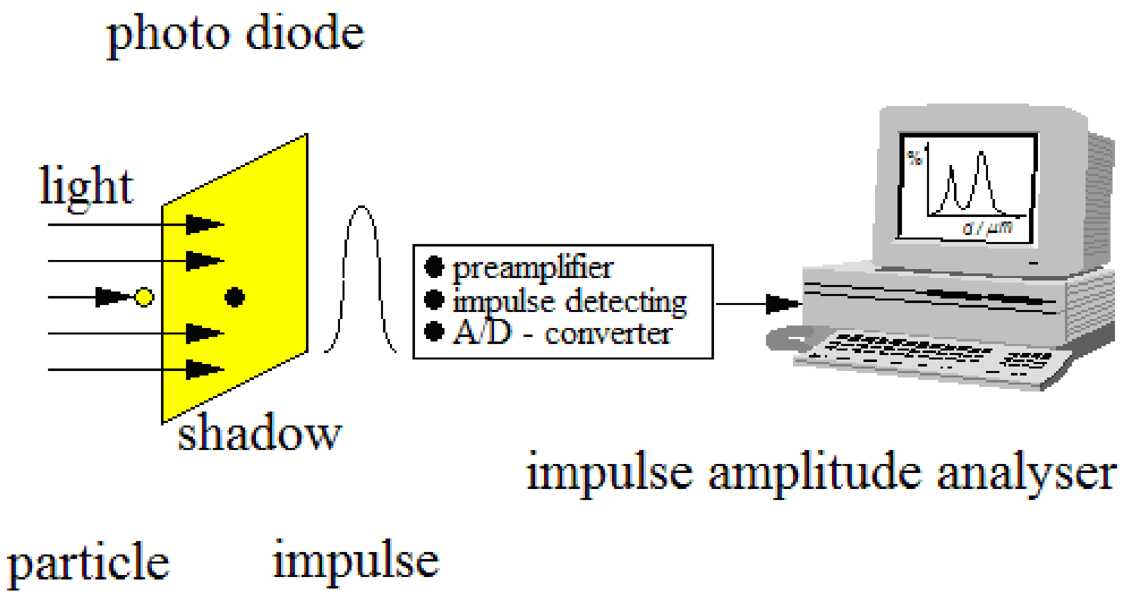


Surface Check

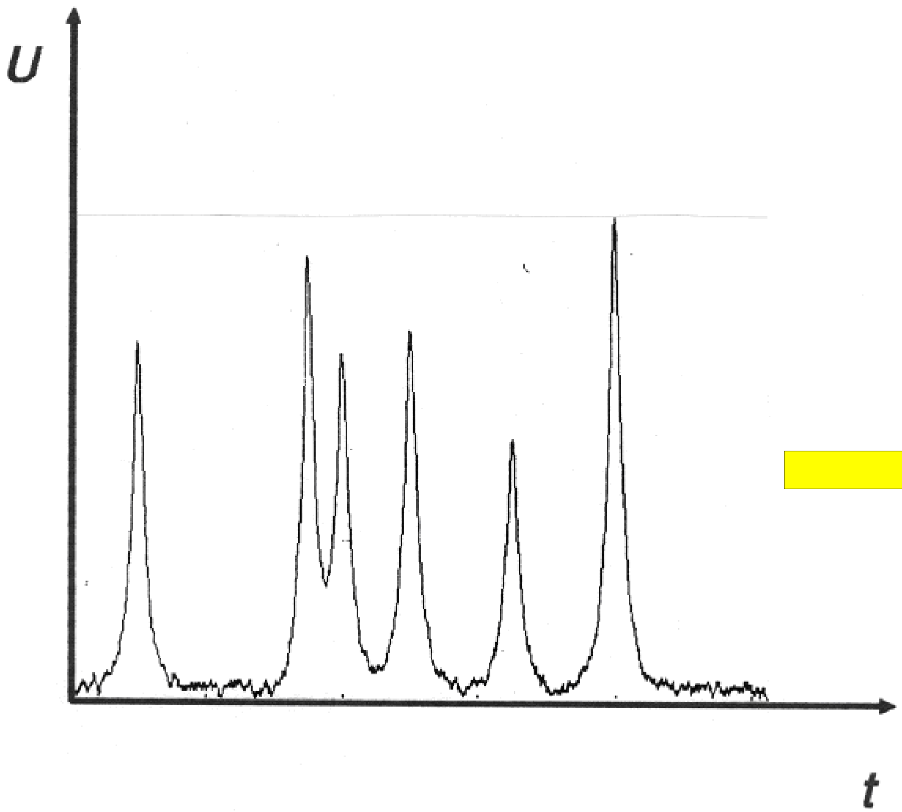


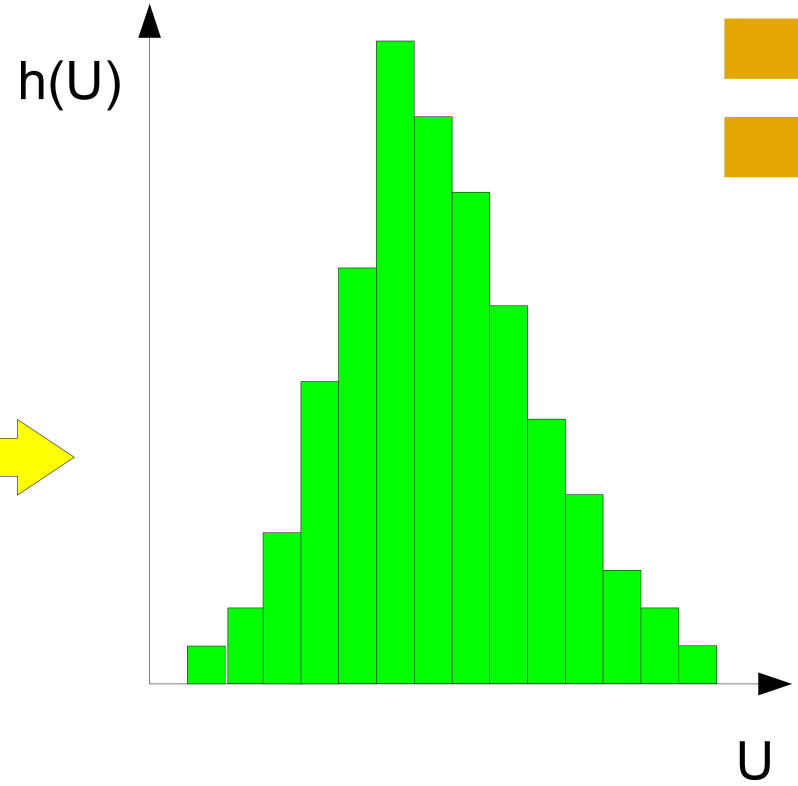
Light Blockade Measurements

The particles going through the light beam and weakens the light which falls on the sensor depending on the particle size. The impulse which comes from the receiver is proportional to the shadow caused by the particle. The impulses are then analysed by an impulse amplitude analyser.



Calculation of the spectrum

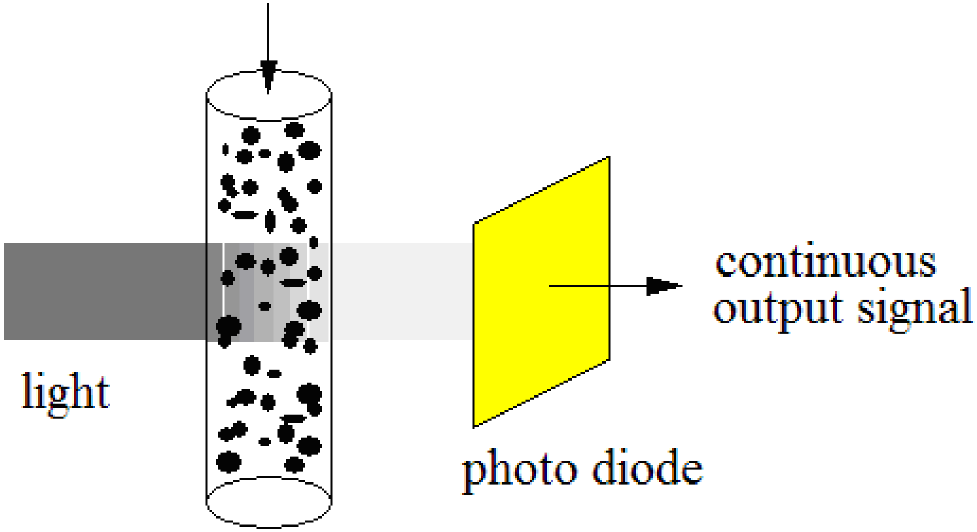




Example: with a 10 bit A/D-converter it is possible to receive a spectrum with 1024 channels. The A/D converted amplitude of the impulses (0-1024) is the number of this channel, to which a one will be added for this impulse.

Turbidity measurement

Target: Determination of the particle concentration. The particles will be not measured separately.



The sensor output signal is a continuous signal. The amplitude is proportional to the concentration of the particles. Calibration is necessary for different materials.

In additional exists solutions which work with reflection techniques (without the ray is going through the sample) and solutions with more then 1 beam.

Lambert-Beer´s Law

$$E = \lg \frac{I_0}{I} = \varepsilon(\lambda, \vartheta)\, c\, d$$

E Extinction
I_0 Input radiation
I Output radiation
ε Extinctioncoefficient
λ Wavelength
ϑ Temperature
c Concentration
d Thickness

Example for calculation

1. Measurement with empty cell

2. Measurement with 100% of the material what is looking for

3. Measurement of the unknown concentration of the material

λ Absorption wavelenght of the material
U Measured intensity of the radiation
d Thickness of the cell

Looking for unknown concentration c.

Solution

Given: λ = absorption wavelength
U = measured intensity of the radiation
d = 3mm
I_0 = 2.4V
$I_{(100\%)}$ = 0.56V
$I_{(c)}$ = 1.34V

Looking for unknown c

Solution:

Calculating of the extinction(at 100%): $E = \lg \frac{I_0}{I}$ → E=0.632

Calculating of the extinction Coefficient: $\varepsilon = \frac{E}{c \cdot d}$ → ε=0.211mm^{-1}

Calculating of the extinction at unknown concentration: $E = \lg \frac{I_0}{I}$ → E=0.253

Calculating of the concentration: $c = \frac{E}{\varepsilon \cdot d}$ → c=0.4 → <u>c=40%</u>

Research Project „Multi Sensor System for the determination of water pollution in industrial washers“

The range of pollutions in industrial washers

Machines for washing medical instruments, injections...

Machines for washing mechanical parts from clocks...

Maschines for washing railway wheels...

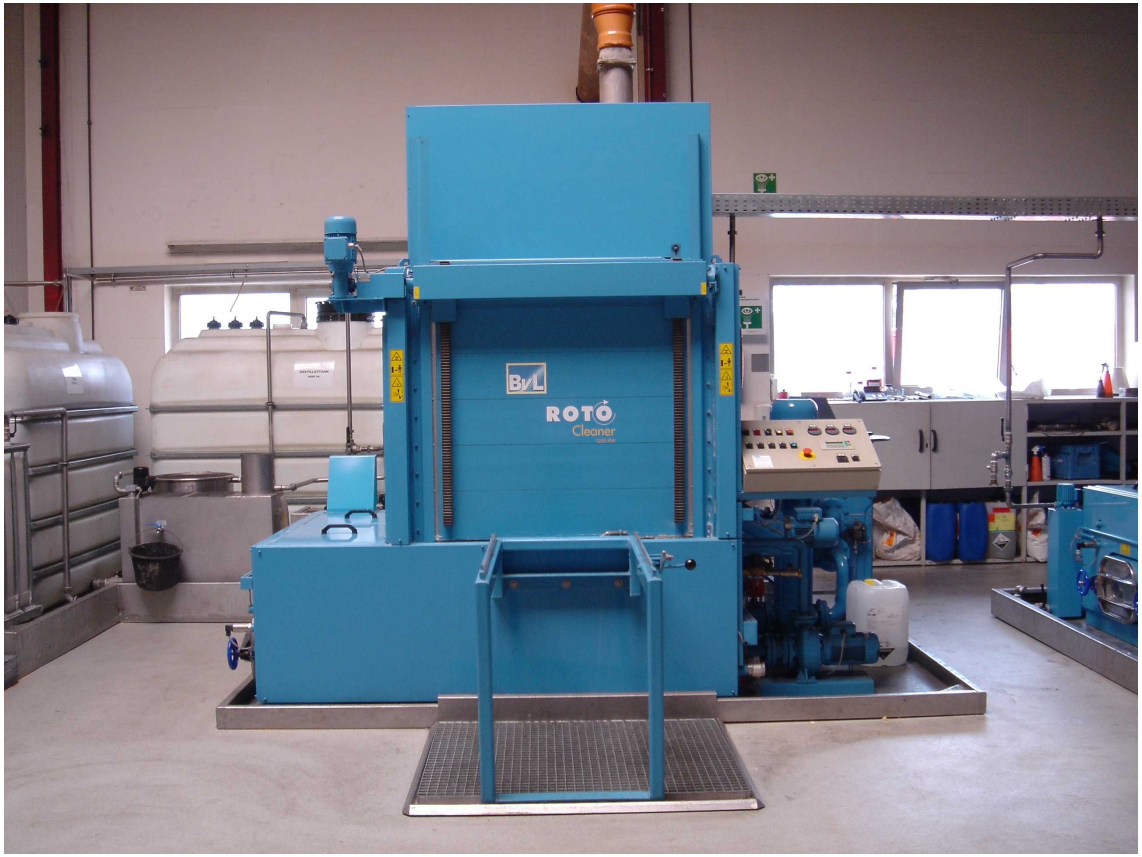
Non visible pollution can lead to the point of wastewater

And all between ←→

Nearly „black“ water can be still to use

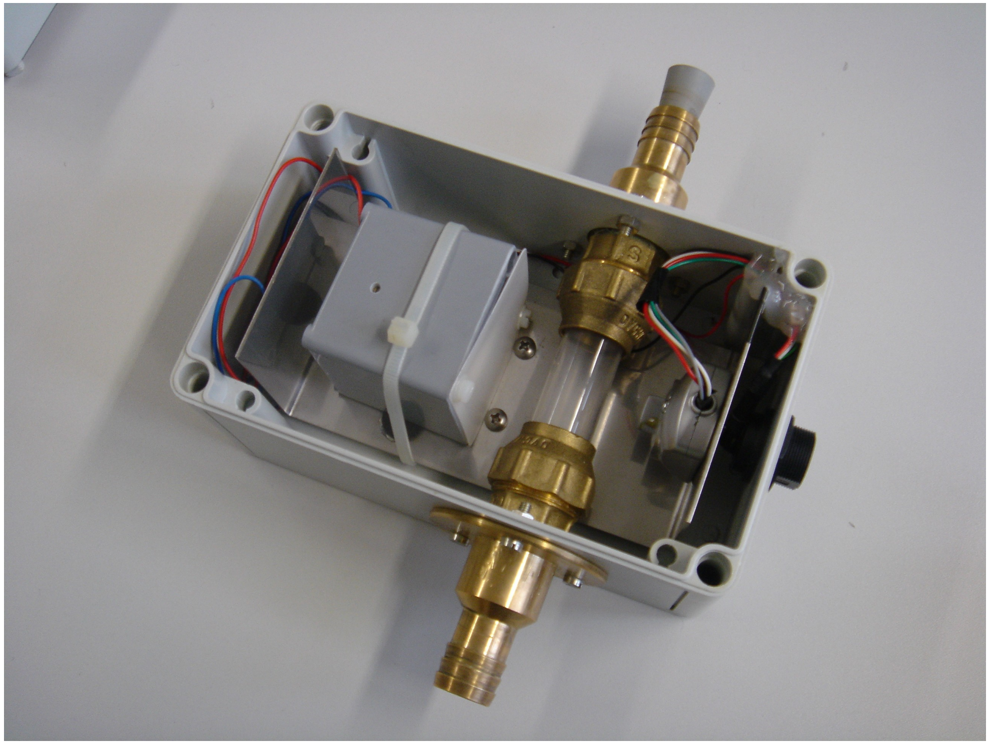
(Mostly oil and biological materials) (Mostly both, solid and oil pollutions)

Visible pollutions



To clean parts from the metal processing industry or to clean used parts before disassembling (washer from BvL GmbH)

Camera and light source



- Webcam instead industrial cam
- light source with diffusion film
- special glass tube

Why do we use a camera?

- Cheap solution including USB-Bus and Software
- large area of measurement (Number of pixels)
- RGB-Signal available to correct the turbidity value

Tasks in reference to the camera

- Switch off of all automatics (brightness, color balances)
- load default Values during installation
- no saturation if liquid is clear, but enough bright if the liquid is almost dark
- additional control of the LEDs is necessary

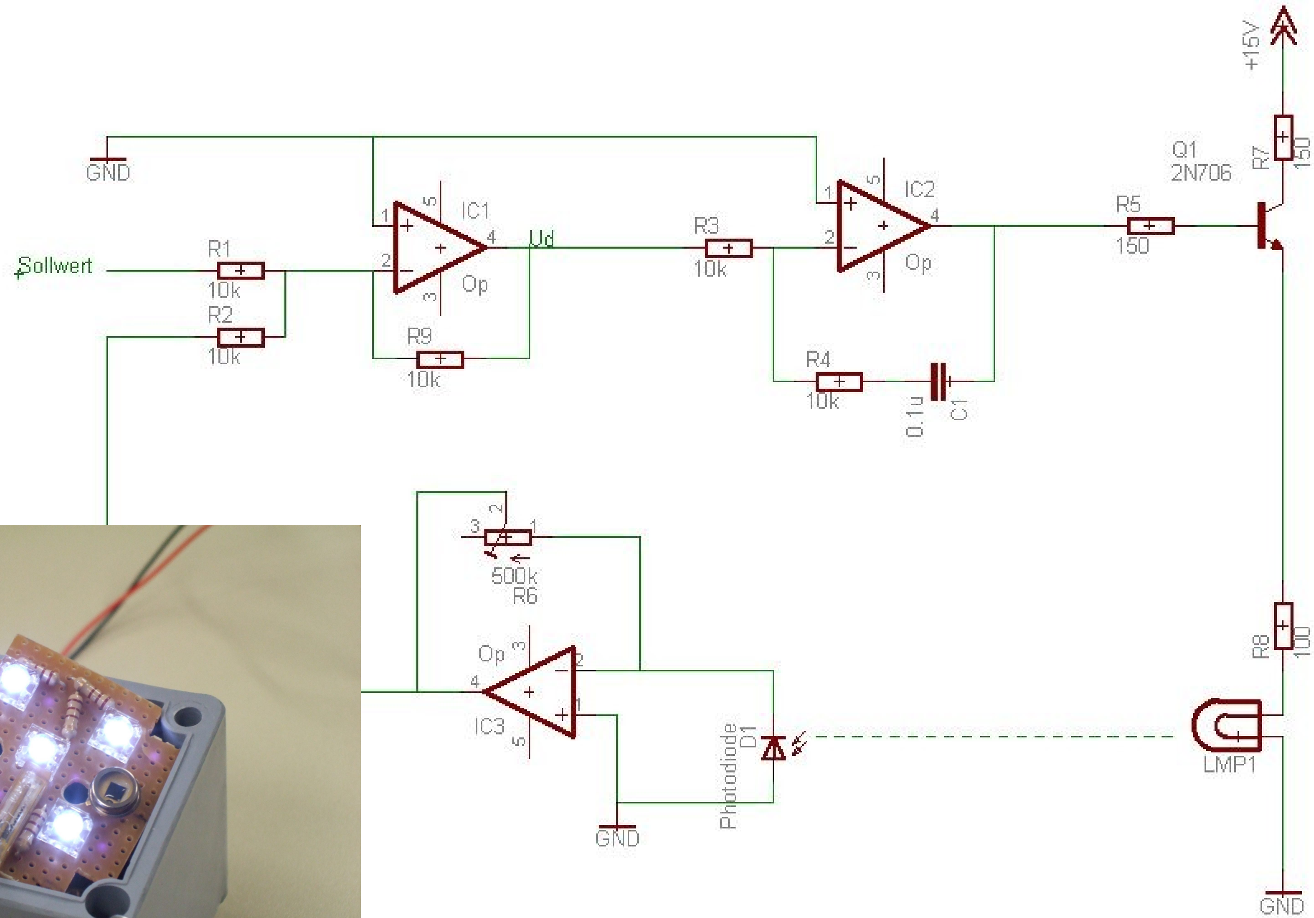
Tasks in reference to the LEDs

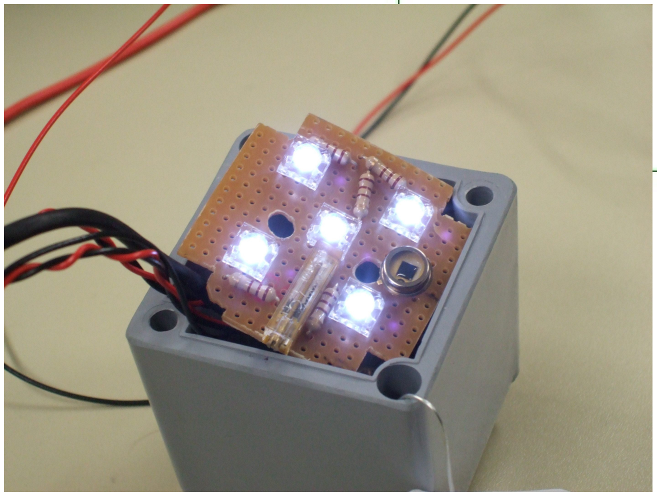
- Equal radiation amplitude on each point of the sensor chip by using of a diffusion film
- constant long term behavior (close loop control of the amplitude of the radiation)

For the extension of the range:

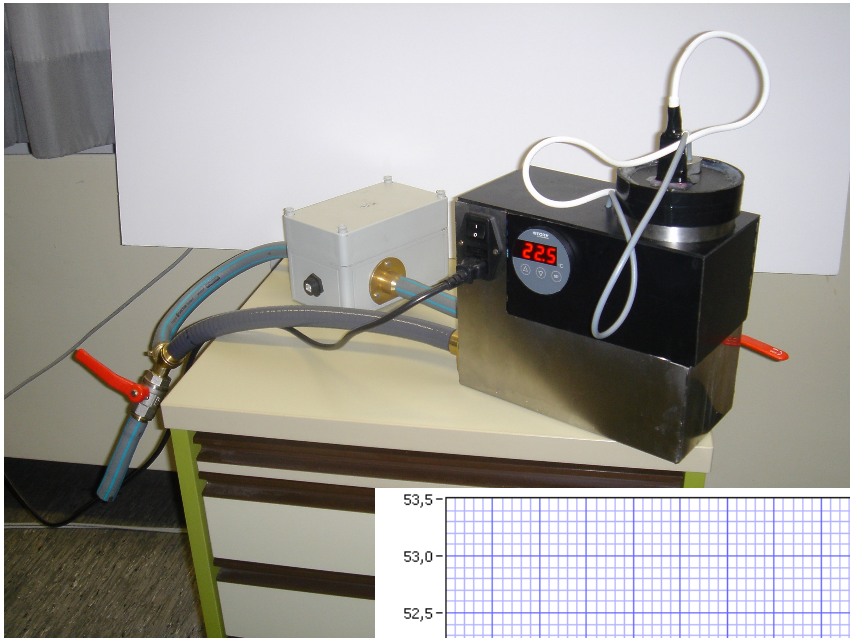
- close lope control with the aim of a constant mean of the gray Value of the camera. The range is extended to the limits of the LEDs current, the resolution in this range is 10Bit (1024 steps)

LEDs and LED control

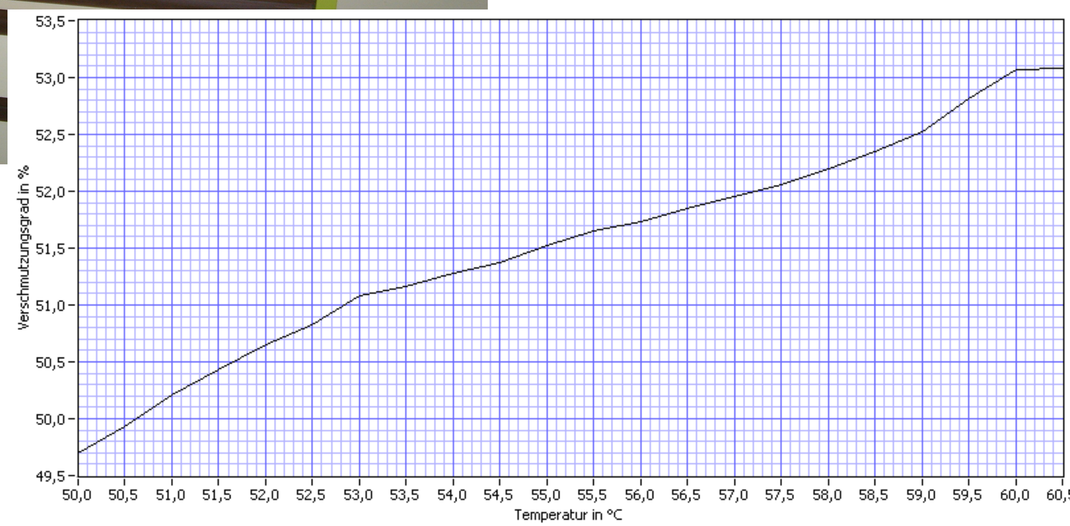




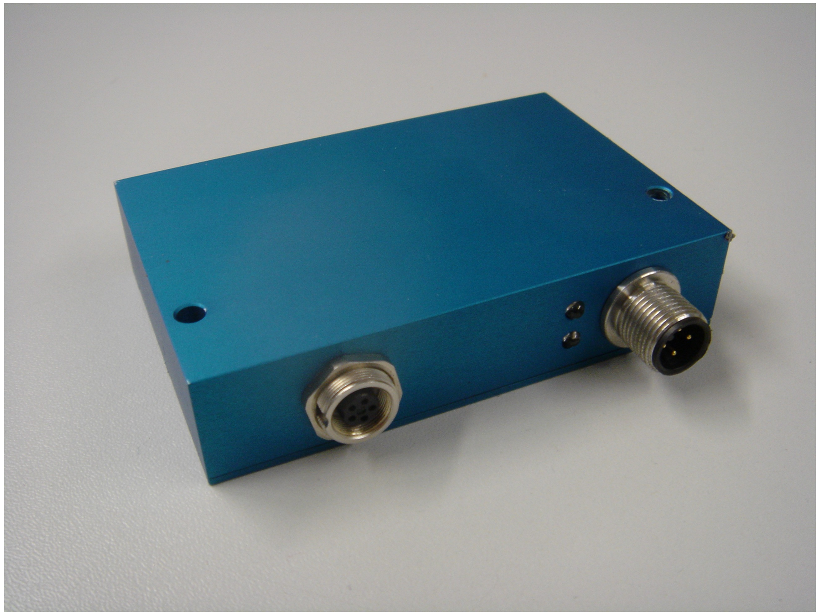
Calibration



Dependence of the turbidity from the temperature

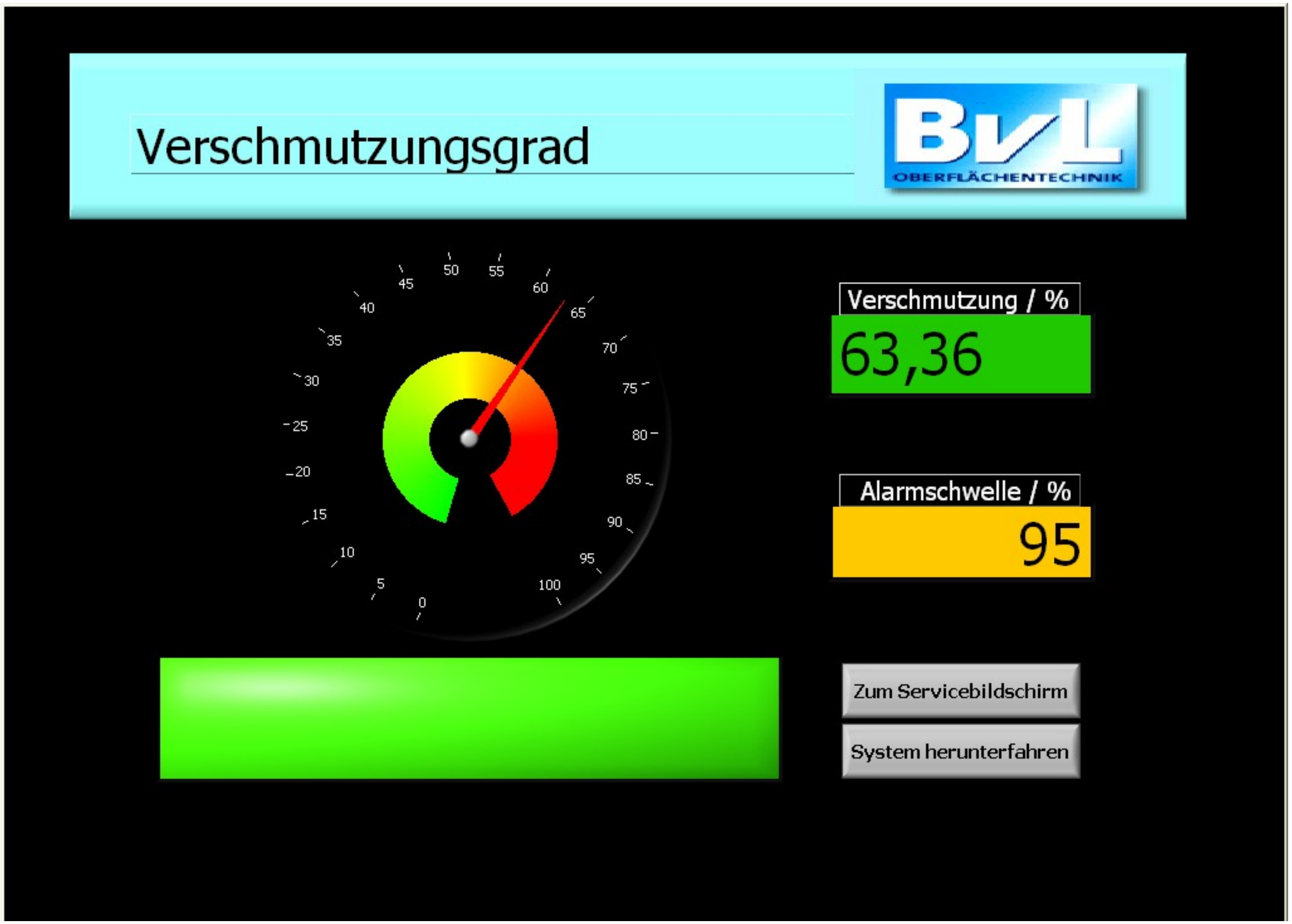


Detection of non visible Pollutions

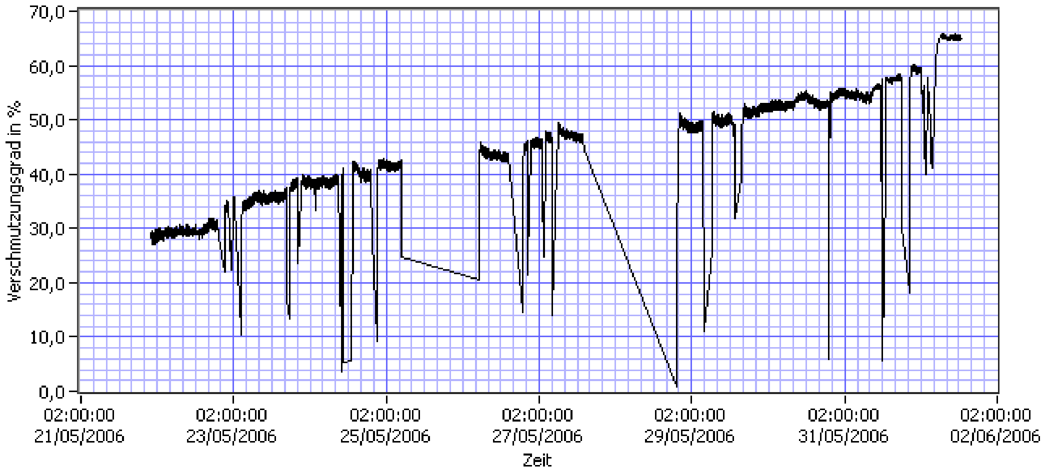


- Available: Sensor for the detection of glue
- For the pollution application necessary:
  - analog output
  - independents from temperature

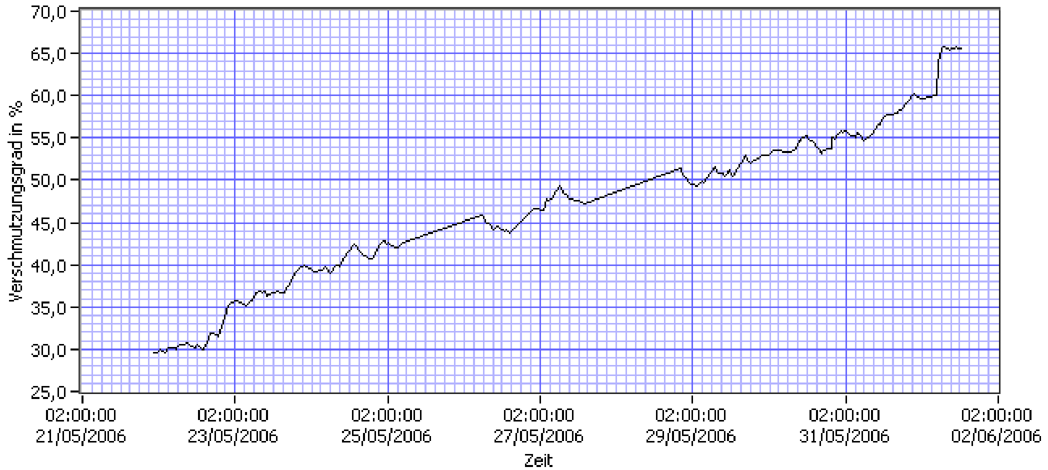
Software


Verschmutzungsgrad
BvL
OBERFLÄCHENTECHNIK
Verschmutzung / %
63,36
Alarmschwelle / %
95
Zum Servicebildschirm
System herunterfahren

Measured pollution over 10 days

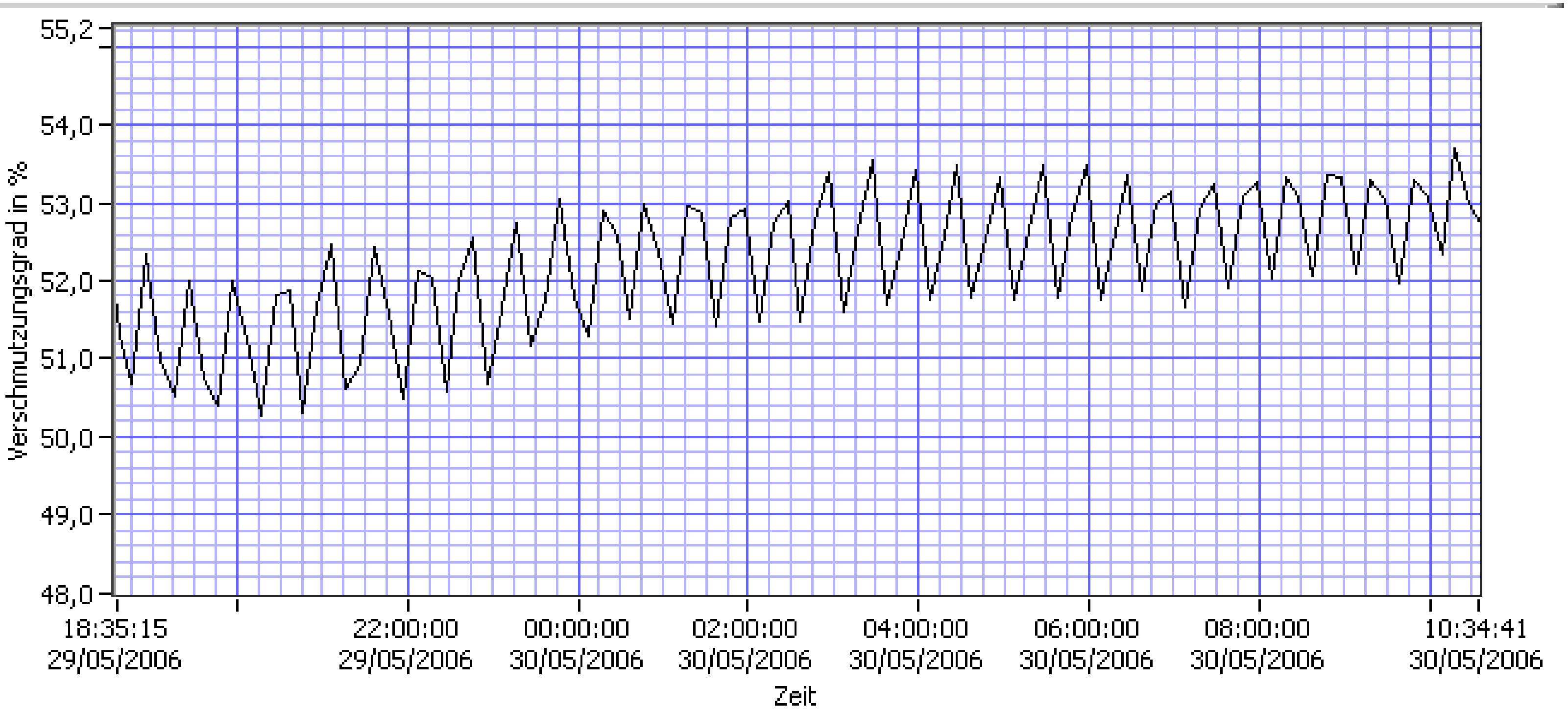


Measured turbidity with disturbances, caused by holidays and other switched off occasions



Calculated development of the water pollution

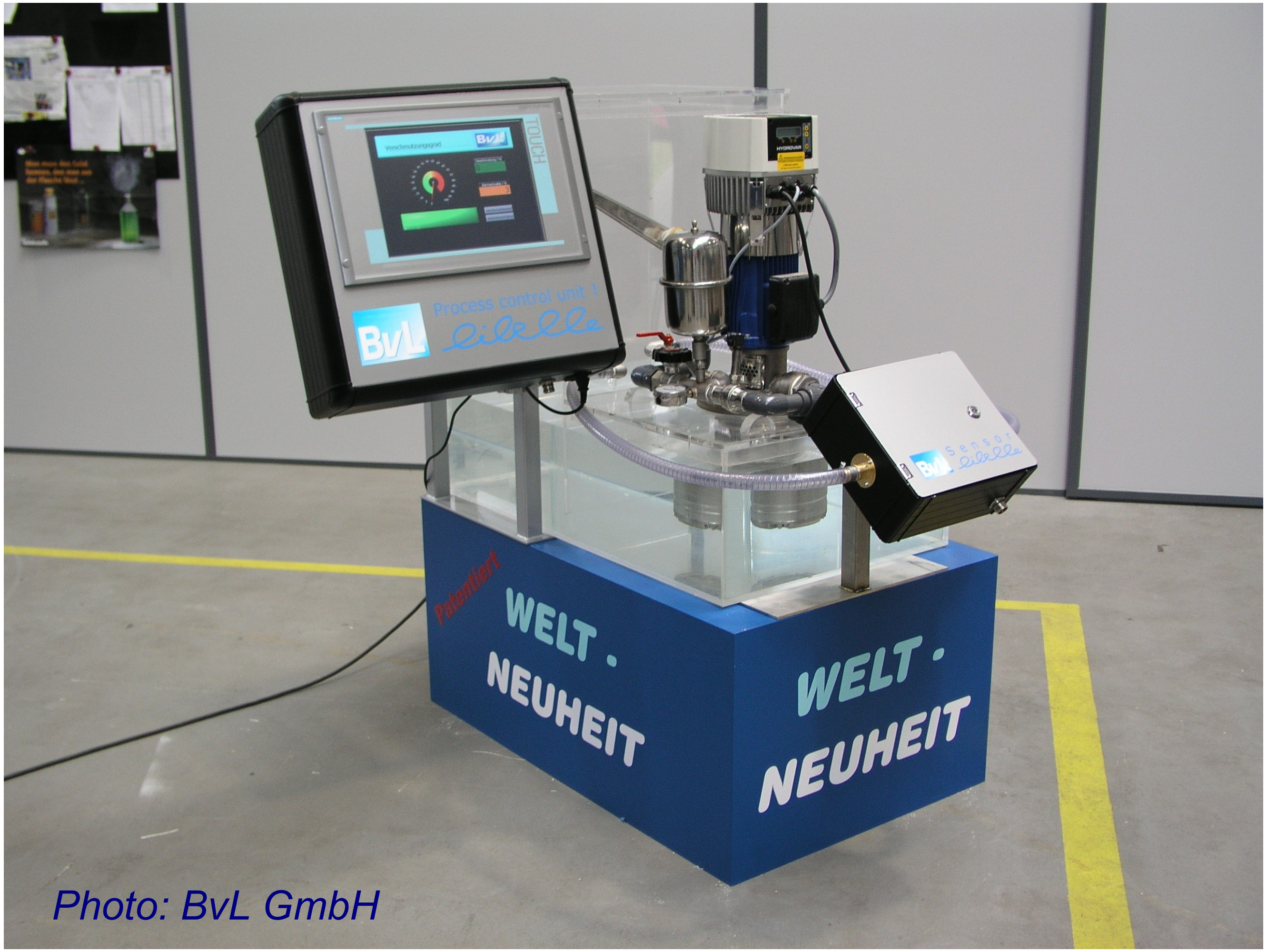
Short term view



- Reason of the periodical: the lost of water by washed parts will be replaced caused by a level sensor.

➡ New possibilities for supervision and diagnostic of the Machines!

On a Fair


BvL
Process control unit 1
Patentiert
WELT -
NEUHEIT
WELT -
NEUHEIT
Photo: BvL GmbH

References

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- *Hoffmann, J. (Hrsg.): **Taschenbuch der Messtechnik**. Fachbuchverlag Leipzig im Carl Hanser Verlag, München, Wien. 4th edition: 2004, 678 pages, ISBN 3-446-22860-8*
- *Hoffmann, J.; Trentmann, W.; Sievering, B.; Düker, Ch.: **Patent Registration** in **Germany**, **Europe** and **US**: "Oberflächenreinigungsvorrichtung". Aktenzeichen 102005049473.0-15, Anmeldetag: 13.10.2005*
- *Hoffmann, J.; Decker,D.; Trentmann, W.; Sievering, B: **Sensor misst Verschmutzung der Reinigungsflüssigkeit**. JOT – Journal für Oberflächentechnik 12/2006, page 2-5*
- *Kirchner, B.; Decker, D.; Hoffmann, J.:* **Sensor zur Bestimmung der Badver-schmutzung an Industriewaschanlagen**. Proceedings, workshop parts2clean 2006, Friedrichshafen, 07.- 09. November 2006.
- *Hoffmann, J.; Trentmann, W.: **Informationsgewinnung an partikularen Prozessen und Materialien.** Sensorik, 2/2006, page 5.*

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