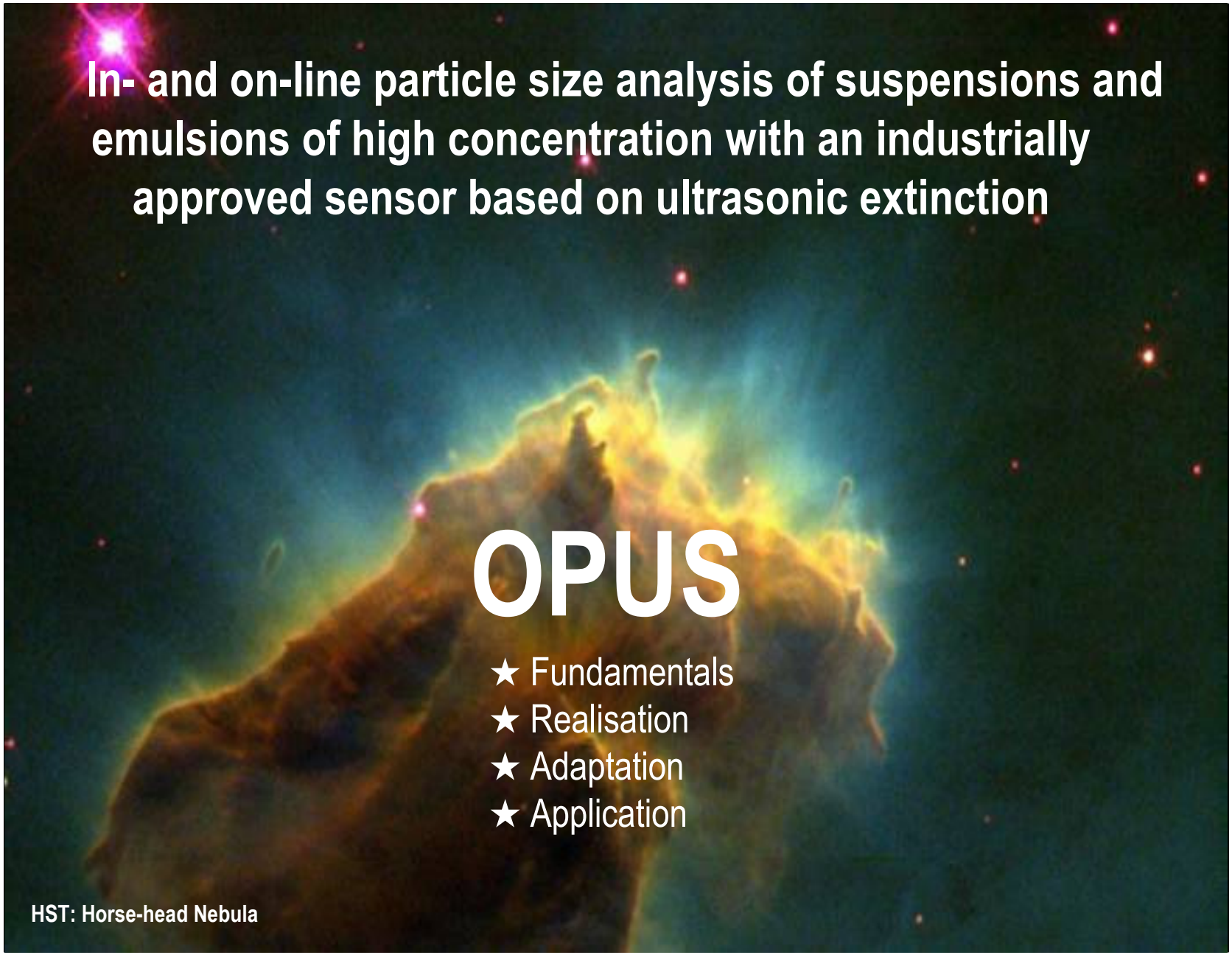
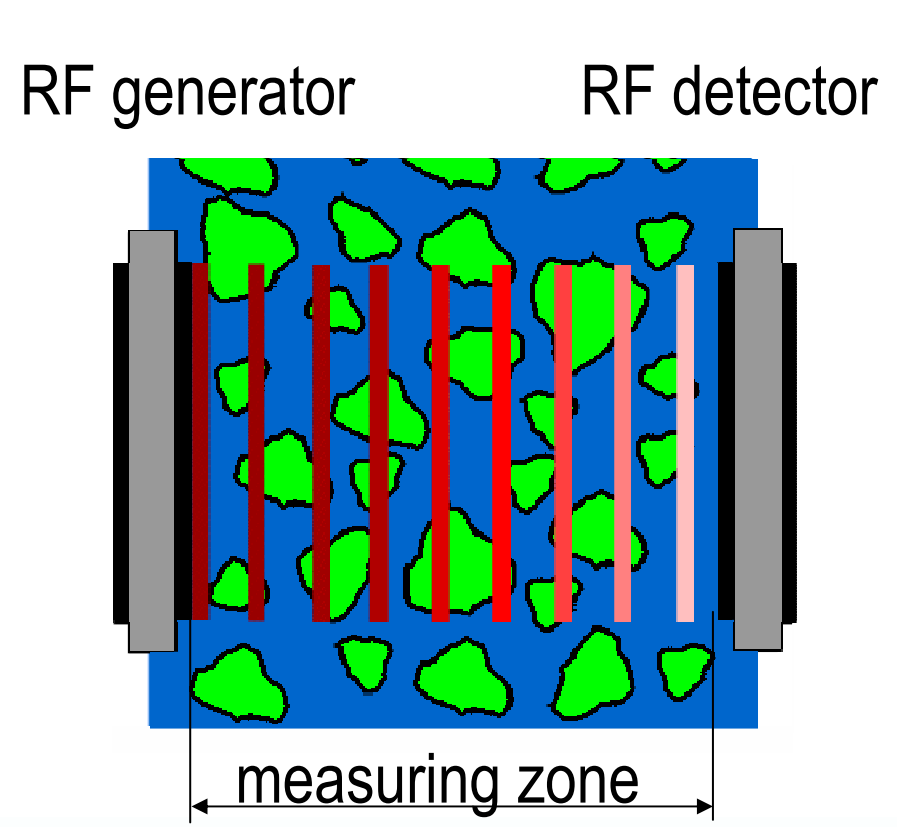
In- and on-line particle size analysis of suspensions and emulsions of high concentration with an industrially approved sensor based on ultrasonic extinction



Fundamentals

Principle



★ Primary signal: Attenuation of sound waves

Ultrasonic Extinction

Fundamentals

Particle-wave Interactions

★ Particle << wave length

↳ Entrainment

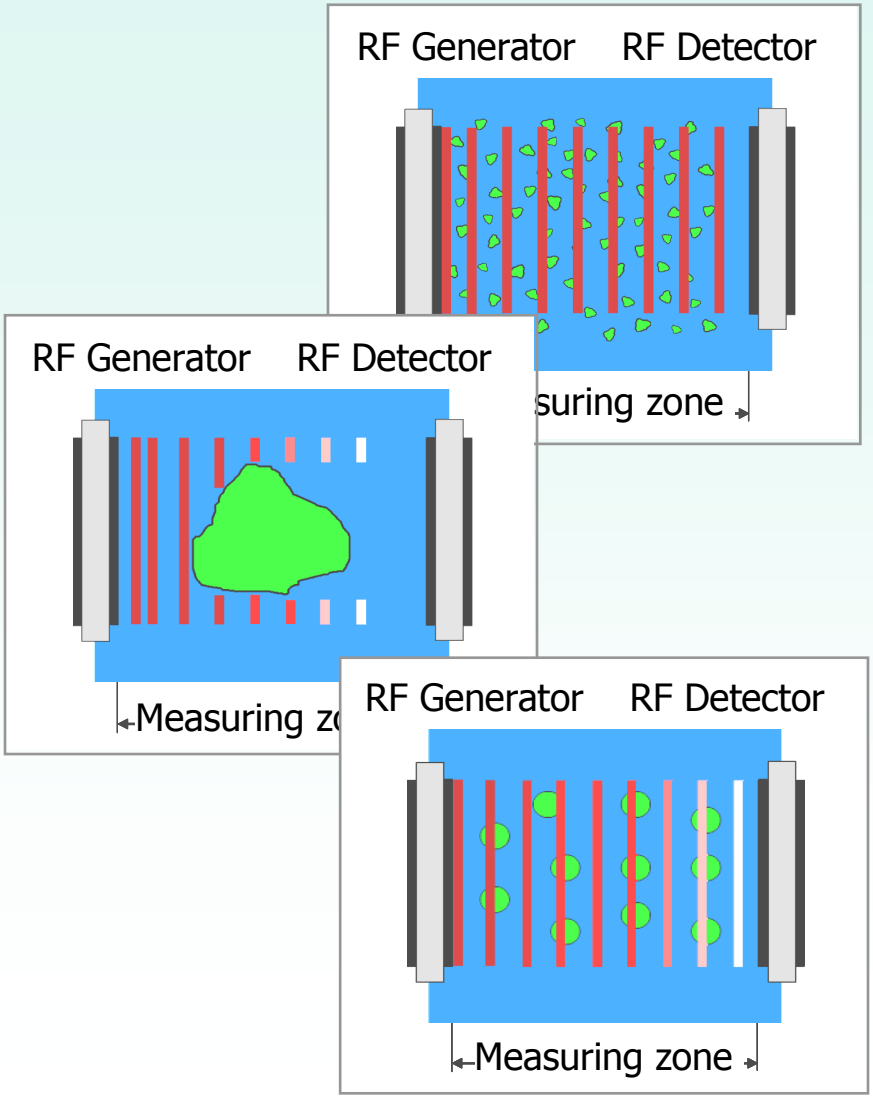
★ Particle >> wave length

↳ Scattering

Special case:

★ Particle ≈ wave length
elastic particles

↳ Resonance

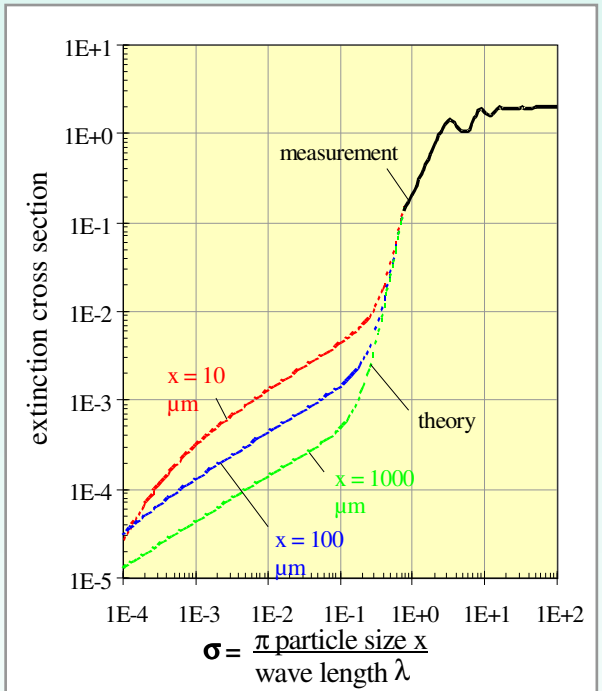


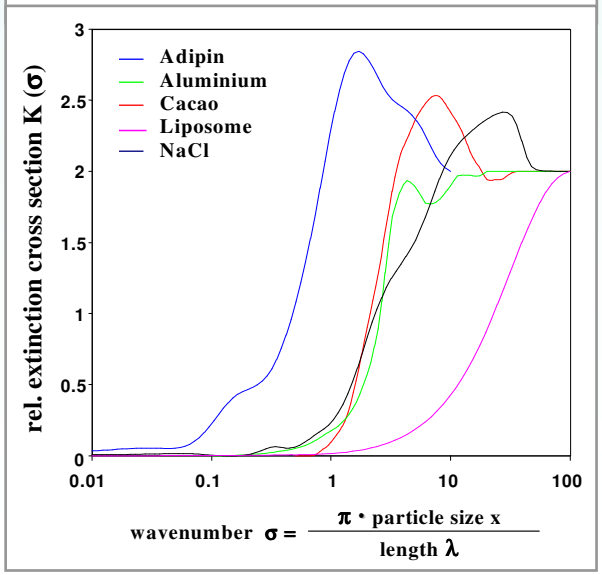
Fundamentals

Calibration

★ Extinction Function K(σ)

- ✔ Depending on material
- ✔ Based on scattering theory
- ✔ Simple to achieve via evaluation software
  - ⇨ WINDOX KSIGMA
- ✔ Broad range of experience e.g. …





Fundamentals

Mathematical Treatment

★ Lambert-Beer's equation

☆ Mono-disperse suspensions

const. values

$$-\ln\left(\frac{I}{I_0}\right)_{f_i} = 1{,}5 \cdot \Delta l \cdot C_v \cdot \frac{1}{x} \cdot K(f_i, x)$$

OPUS Signal

Particle size

Extinction Function

☆ Distributions

$$-\ln\left(\frac{I}{I_0}\right)_{f_i} = 1{,}5 \cdot \Delta l \cdot C_V \cdot \frac{1}{M_{1,2}} \cdot \int_{x_{\min}}^{x_{\max}} K(f_i, x) \cdot q_2(x) dx$$

Integral

$$\cong 1{,}5 \cdot \Delta l \cdot C_V \cdot \frac{1}{M_{1,2}} \cdot \sum_j K(f_i, x_j) \cdot q_2(x_j) \Delta x_j$$

Approx.

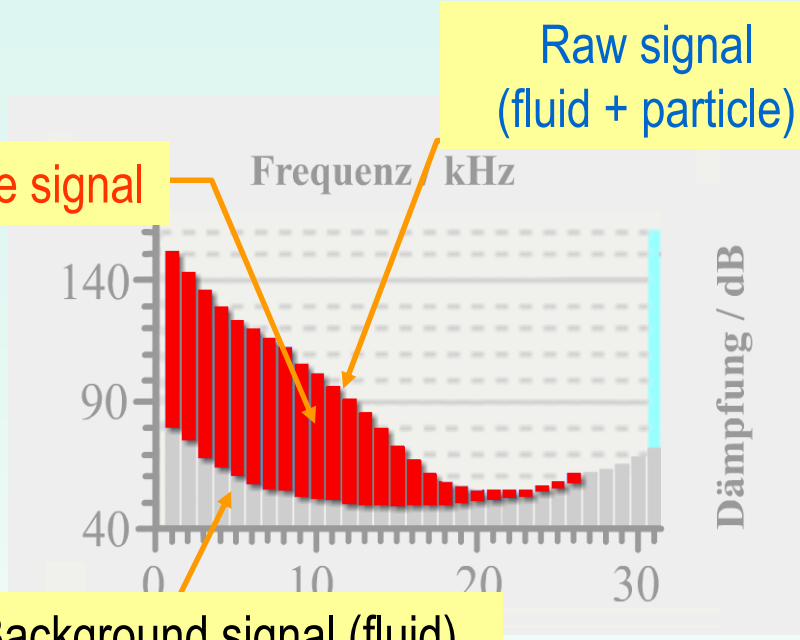
Fundamentals

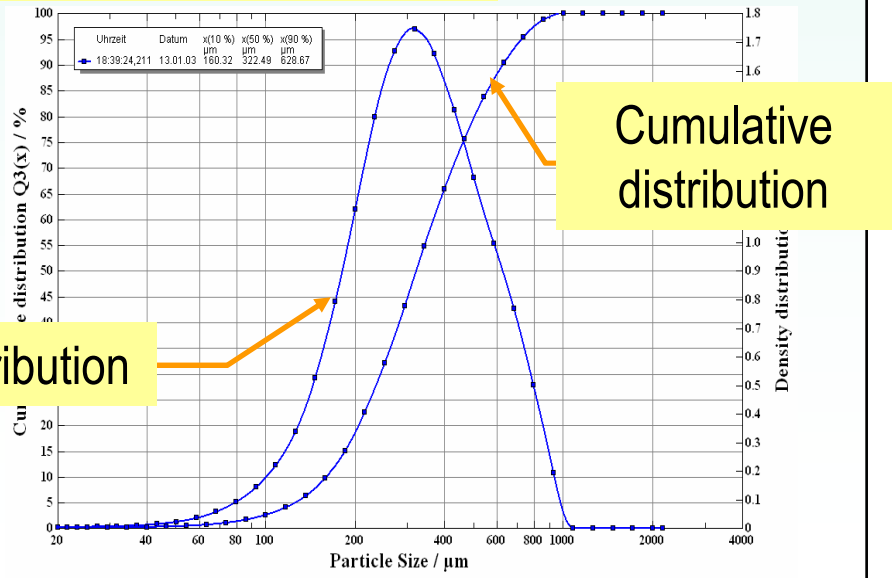
OPUS Measurement

★ Attenuation spectrum

- ✔ 31 frequencies applied
  - ↳ Leads to 31 particle size classes
- ✔ *Fast scan* < 30 sec.
- ✔ High dynamic range (160 dB)

★ Particle size distribution





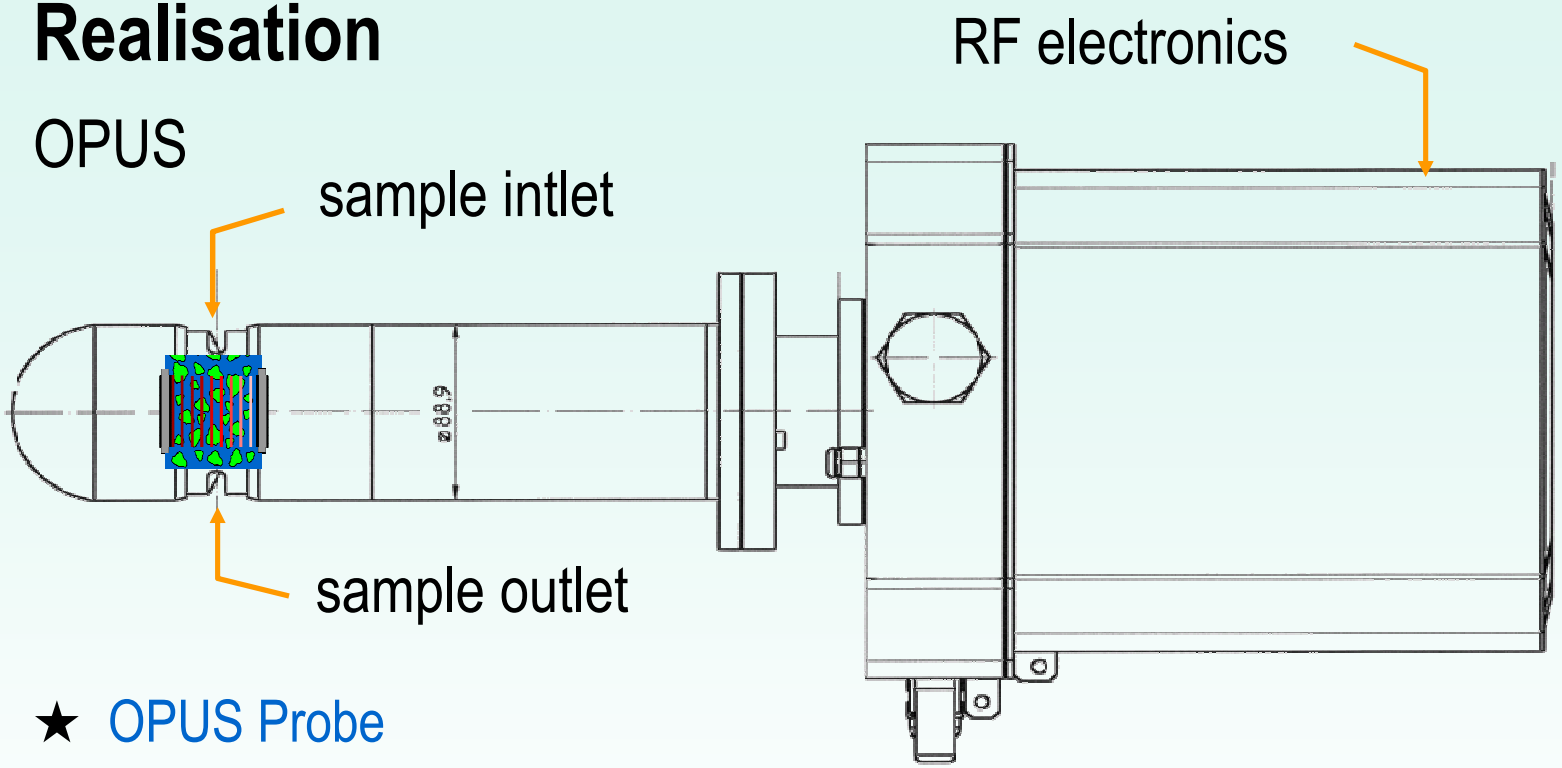
Realisation

Requirements to in-line PSA sensor

- ★ Measuring zone and electronics
- ★ Compact and rugged design
- ★ Applicable for:
  - ↳ Process concentrations (no dilution)
  - ↳ High and variable temperatures
  - ↳ High and variable pressures
  - ↳ Caustic and acidic liquids
- ★ Easily adaptable to different kinds of processes
- ★ Easily accessible

Realisation

OPUS



★ OPUS Probe

- ✔ Measuring gap allows 10 mm intrusion depth
- ✔ Standard materials in contact with suspension (other on request):
  - ↳ Stainless steel (V4A, DIN/ISO 1.4571, SS316)
  - ↳ SIGRADUR ™
  - ↳ Fibre enforced TEFLON ™

Realisation

OPUS specification

★ Process conditions

- ✔ Temperature: 0..120°C (150 °C optional)
- ✔ Pressure: 0..40 bar
- ✔ pH-value: 1 - 14
- ✔ IP65, ATEX II 2G EEx p II T5

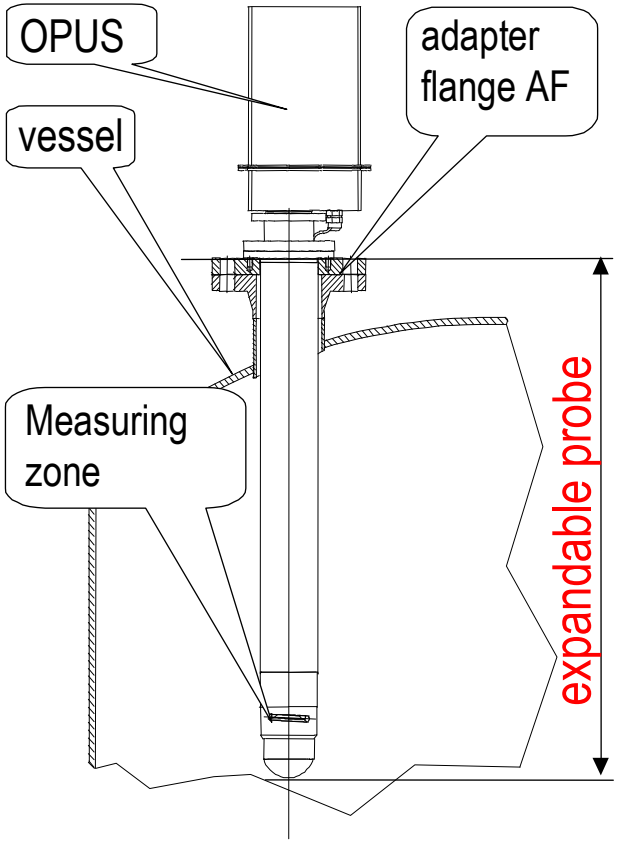
★ Measuring conditions

- ✔ Suspensions & emulsions
- ✔ 0.01 µm - 3000 µm
- ✔ 31 size classes
- ✔ Volume concentration: 1 to 70 % vol.

Process Adaptation

★ Adaptable length

- ✔ OPUS can be manufactured in different lengths (up to 3500 mm)
- ✔ Standard version
  fixed length: 330 mm
  diameter: 89 mm ∅
- ✔ For installation in reactors and pipes > 200 mm
- ✔ Installation via standard flange through the reactor head



Process Adaptation

★ OPUS/FT – Flow-Through Adapter

- ✔ For installation in small pipes Ø < DN 25
- ✔ Entire suspension passes the measuring zone
- ✔ Simple solution for bypass-installations

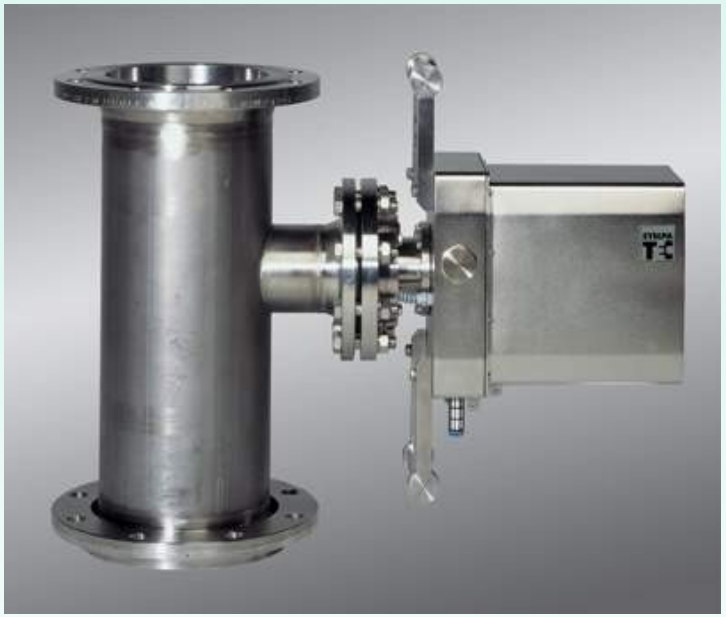
★ OPUS/BP – Bypass-Adapter

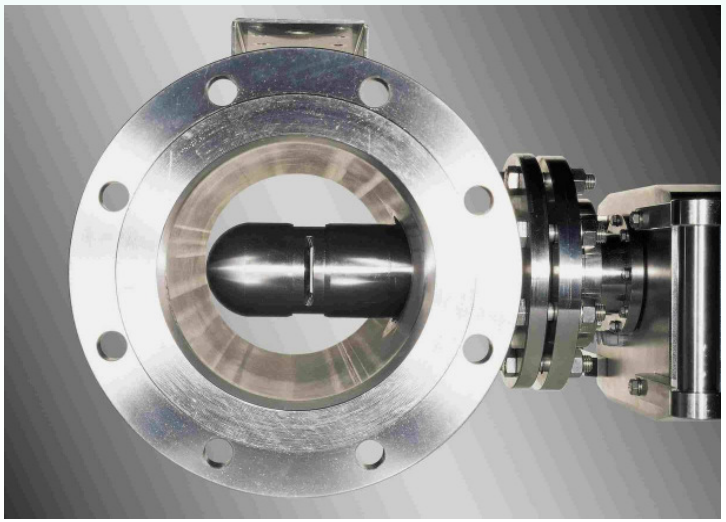
- ✔ For pipes from DN 25 – 200
- ✔ Partial flow passes the measuring zone
  - ↳ Avoids drop of pressure in process pipe

Process Adaptation

★ OPUS/AF – Adapter flange

- ✔ For process pipes Ø > DN 200
- ✔ Simple flange-on solution with OPUS AF-Adapter
- ✔ in-line analysis





Process Adaptation

★ Docking Positioner & Cleaner (DP&C)

- ✔ For installation on vessels or pipes Ø > 200 mm
- ✔ Motorised drive unit for easy access to the OPUS probe without shut down of the process
- ✔ Integrated cleaning station for flushing of the measuring zone

Applications

Typical installations

in-line installation

★ PSA directly in main process flow:

- ↳ High measurement frequency (typ. 2 analyses / min.)
- ↳ No bypass required

on-line installation

★ PSA in bypass-operation e.g. in circulation loop

- ↳ High measurement frequency (typ. 2 analyses / min.)
- ↳ Simple and easy access to the sensor

off-line installation

★ PSA in lab

- ↳ USE available for R&D purposes

Applications

Typical processes and fields of applications

Crystallisation

- ★ Vaporization crystallisation
  - ↳ e.g. NaCl, KCl, K2SO4, ammonium perchlorate
- ★ Precipitation crystallisation
  - ↳ e.g. CaCO3, plastics
- ★ Cooling crystallisation
  - ↳ e.g. dextrose, fructose, lactose, perborate,

Polymerisation

- ↳ e.g. PS, EPS, HIPS, PVAC, EPDMs.

Classifying / Blending

- ↳ e.g. minerals

Grinding

- ★ Ball mill
  - ↳ e.g. minerals, ore, sand, metals, pharm. drugs
- ★ Agitation mill
  - ↳ e.g. pigments, coatings, pharm. drugs

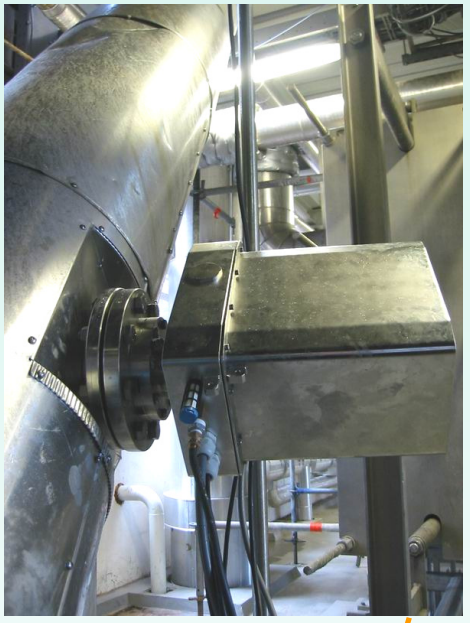
Homogenisation

- ★ Emulsions homogenisation
  - ↳ e.g. beverages, lubrications, polymers, pharm. & cosmet. creams

Installation Example

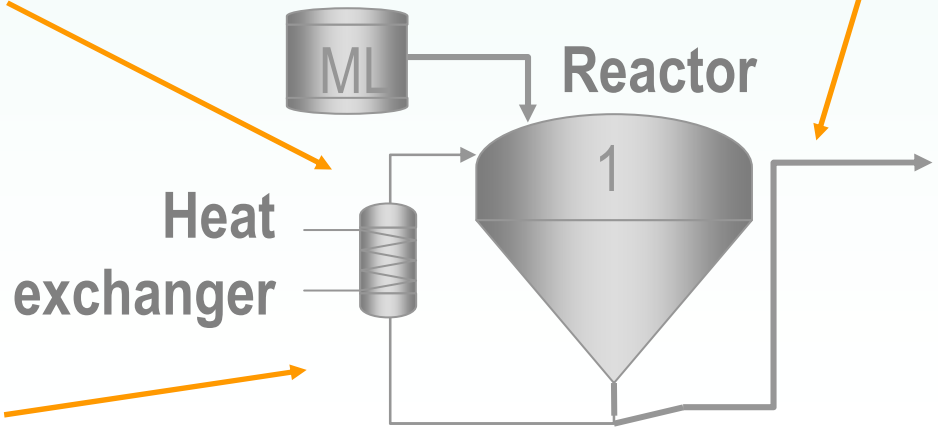
in-line / AF-Adapter / Crystallisation / Food

★ Product: **Sweetening agents**



★ 3 OPUS in on one crystalliser

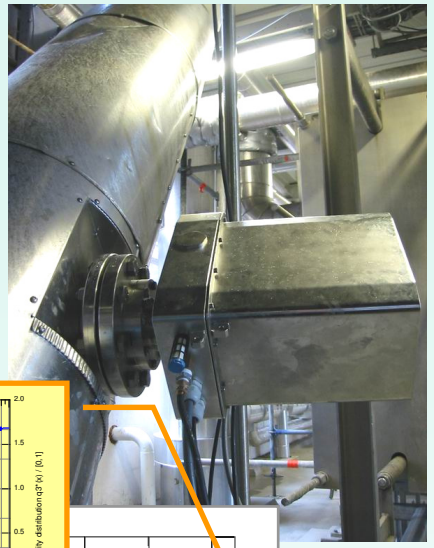
★ Goal: modelling of crystallisation dynamics

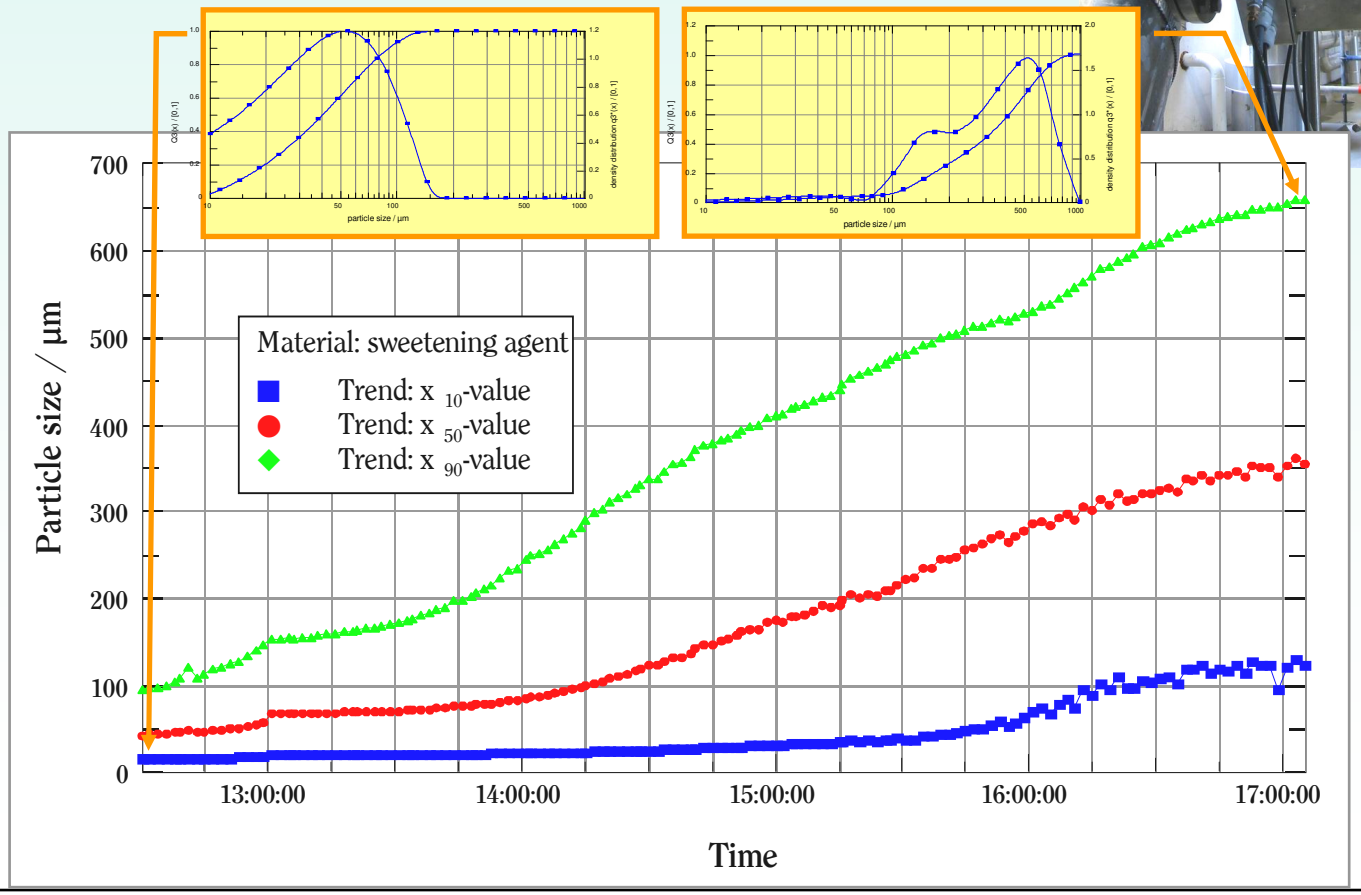


Installation Example

in-line / AF-Adapter / Crystallisation / Food

★ Results: **Sweetening agent**

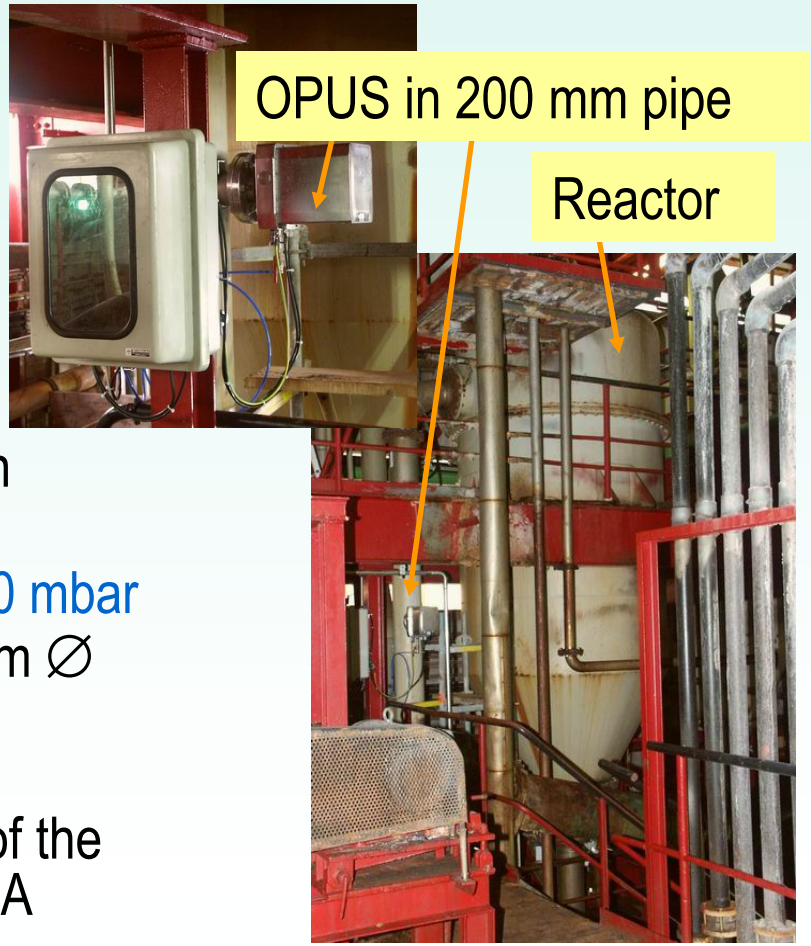




Installation Example

in-line / AF-Adapter / Crystallisation / Pharmaceuticals

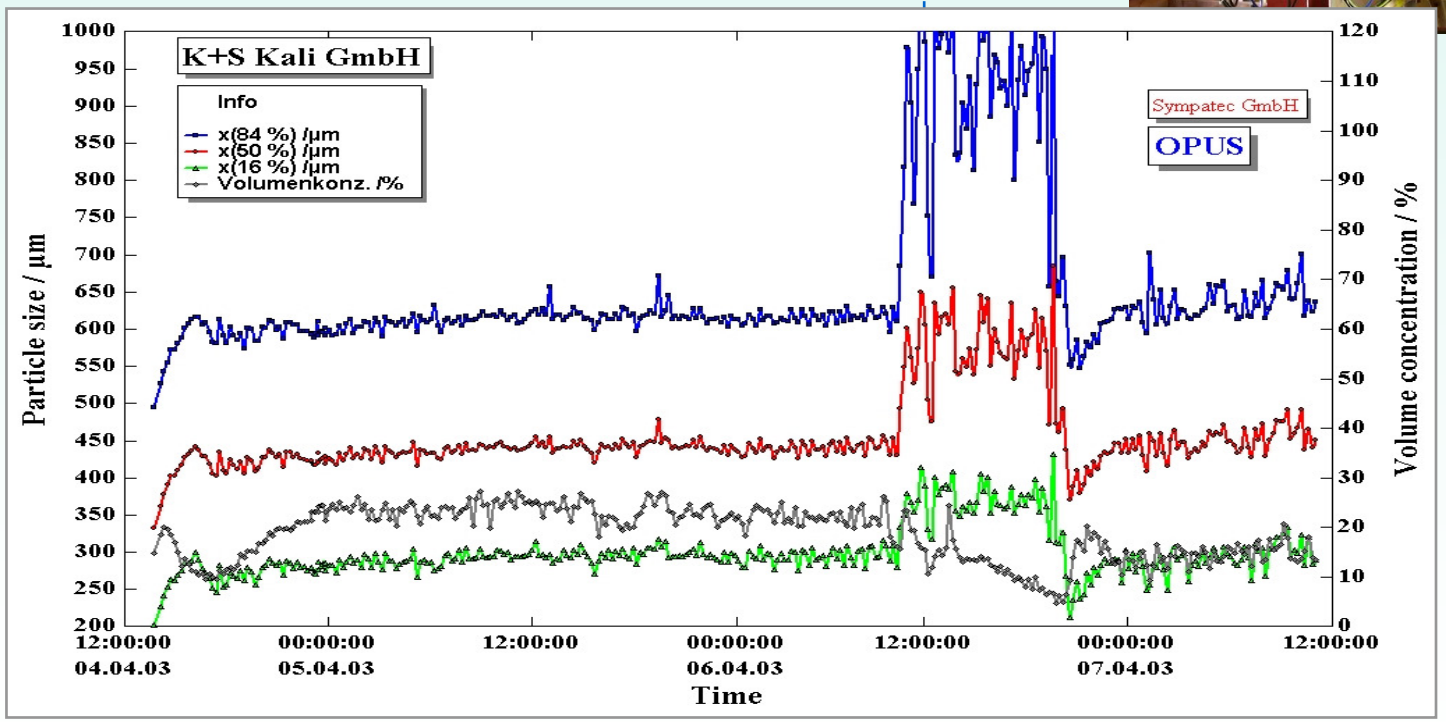
- ★ Products
  - ↳ **Ultra refined NaCl, KCl and K2SO4** for pharmaceutical use
- ★ Process
  - ↳ Vaporisation crystallisation
  - ↳ Temperature: 70 °C
  - ↳ Vacuum: ca. 100 mbar
  - ↳ Process pipe: 200 mm ∅
- ★ Goal
  - ↳ Significant enhancement of the reactor utilising on-line PSA



Installation Example

in-line / AF-Adapter / Crystallisation / Pharma

★ Results: **Ultra refined NaCl, KCl + K2SO4**



✔ Particle size analysis throughout the entire process

✔ Process regimes are clearly detected

Installation Example

in-line / Docking Positioner & Cleaner / Crystallisation / Chemicals

★ Product: **Ammonium sulphate**

OPUS 1400 & DP&C

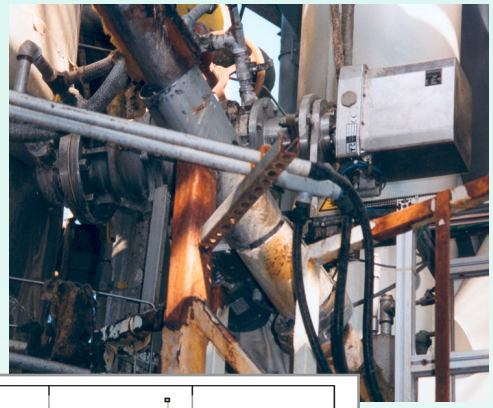
Circulation pipe 28 m

★ Process

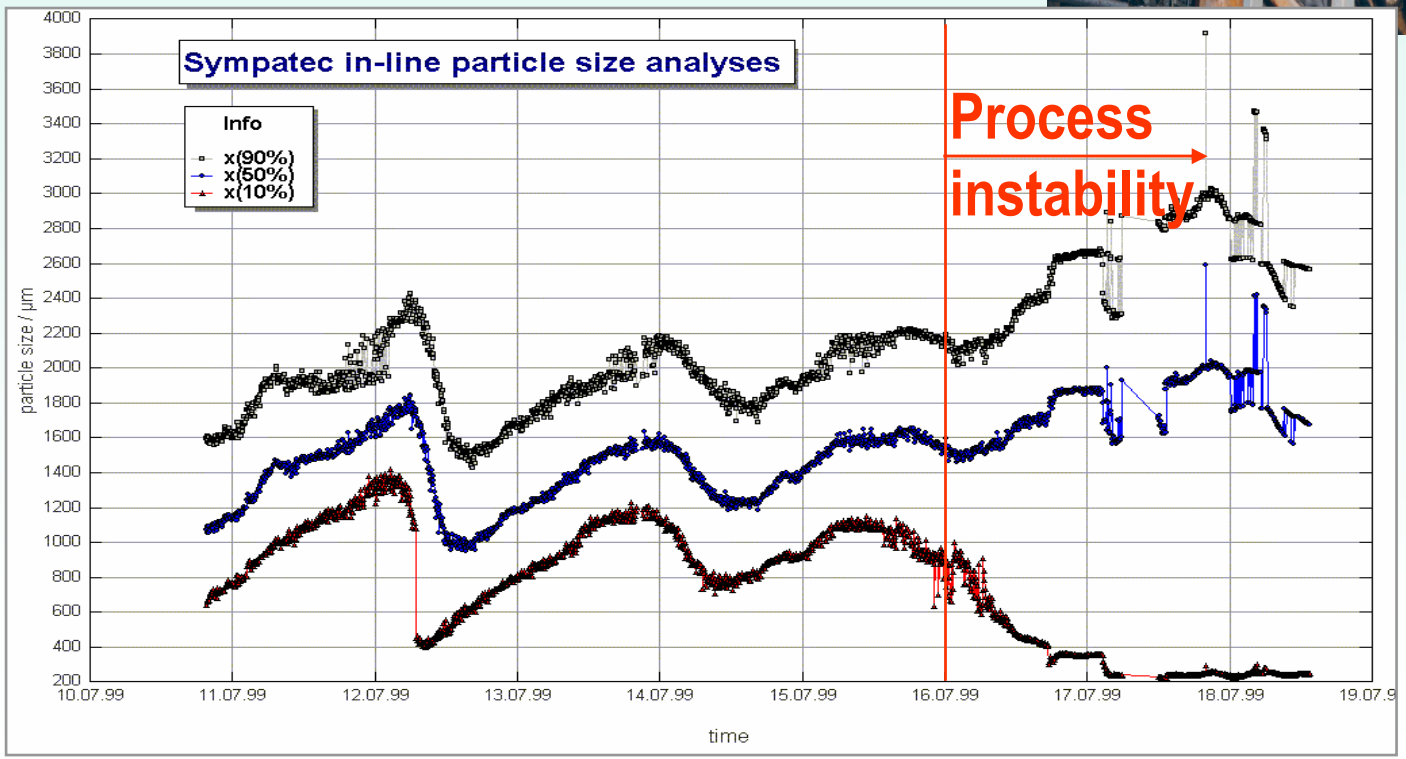
- Cont. crystallisation
- 115 °C
- 3 bar
- Pipe: 1100 mm ∅
- Operation: 24/7

Installation Example

in-line / DP&C / Crystallisation / Chemicals



★ Results: **Ammonium sulphate**



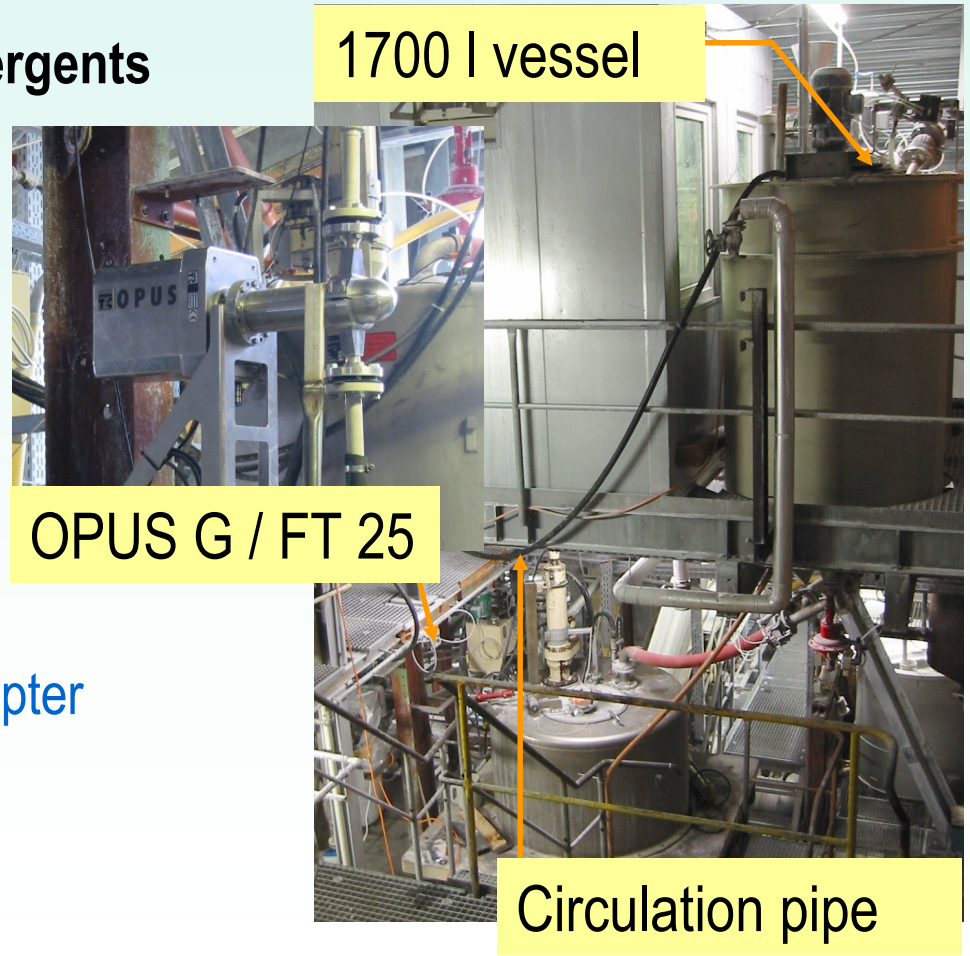
✔ Detection of known oscillation of cont. operating crystallisers

✔ Instabilities are recognised and allow for fast reaction

Installation Examples

on-line / FT-Adapter / Crystallisation / Detergents

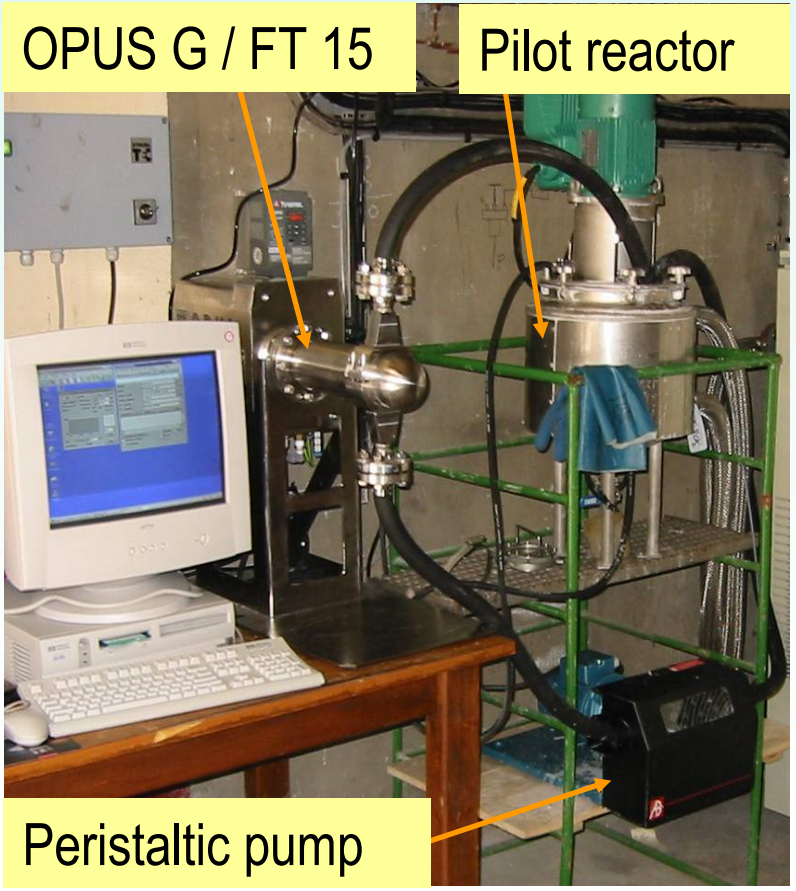
- ★ Product: **Various detergents**
- ★ Process conditions
  - ↳ Batch operation
  - ↳ 1700 l reactor
  - ↳ 35 °C
  - ↳ 1 bar
  - ↳ 30 – 60 % vol.
- ★ Adaptation
  - ↳ Using a FT 25 Adapter
  - ↳ In circulation loop



Installation Example

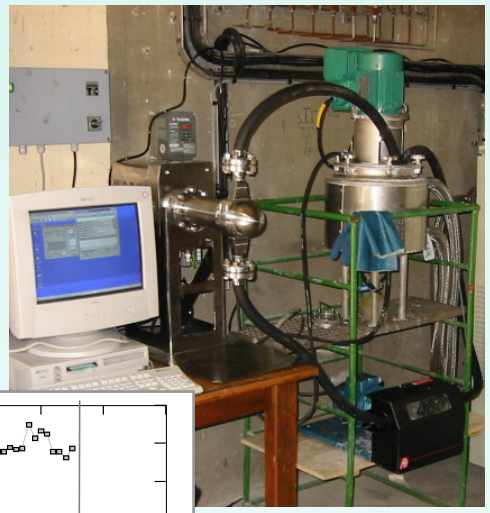
on-line / FT-Adapter / Crystallisation / R&D

- ★ Product: **Ammonium per-chlorate** (for solid propellant missile)
- ★ Process
  - ↳ Batch operation
  - ↳ Cooling crystallisation
  - ↳ 14 l reactor
  - ↳ 70 - 20 °C
  - ↳ 1 bar
  - ↳ 0 - 60 % vol.
- ★ Adaptation
  - ↳ FT-25 Adapter

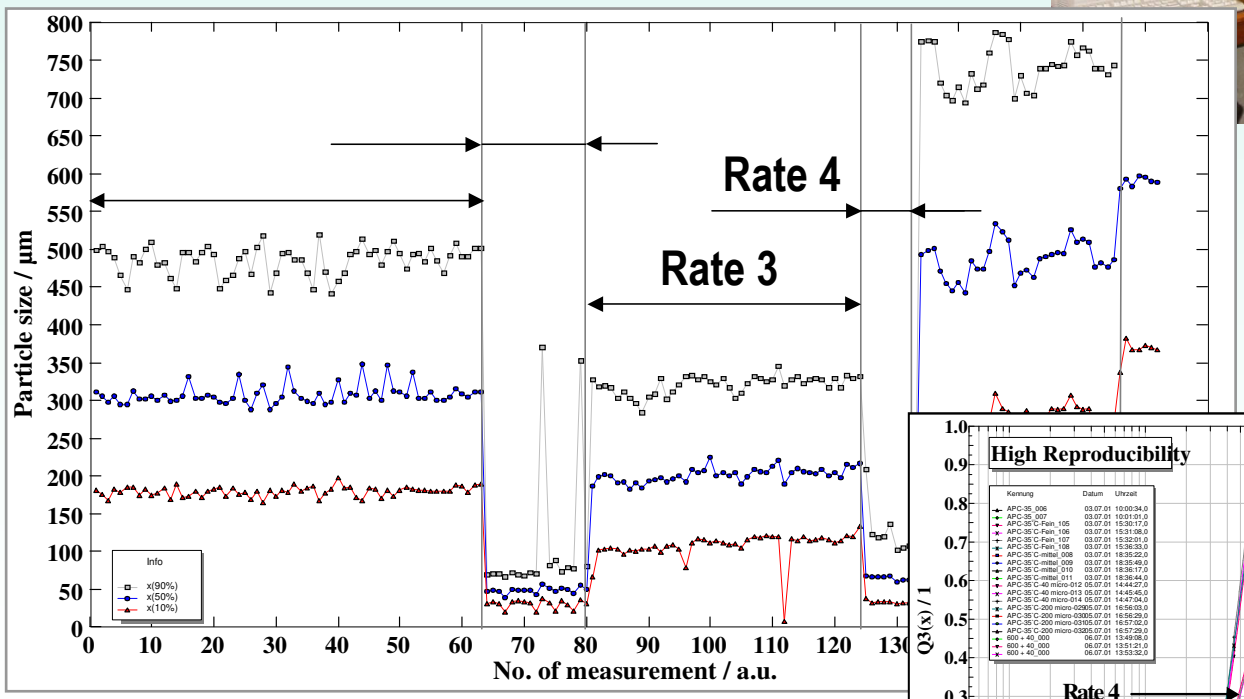


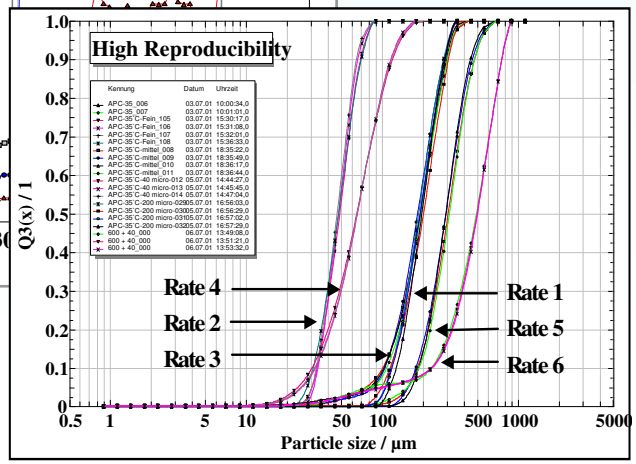
Installation Example

on-line / FT-Adapter / Crystallisation / R&D



★ Results: **Ammonium perchlorate**





✔ OPUS detects varying particle sizes depending on the cooling rate

Installation Examples

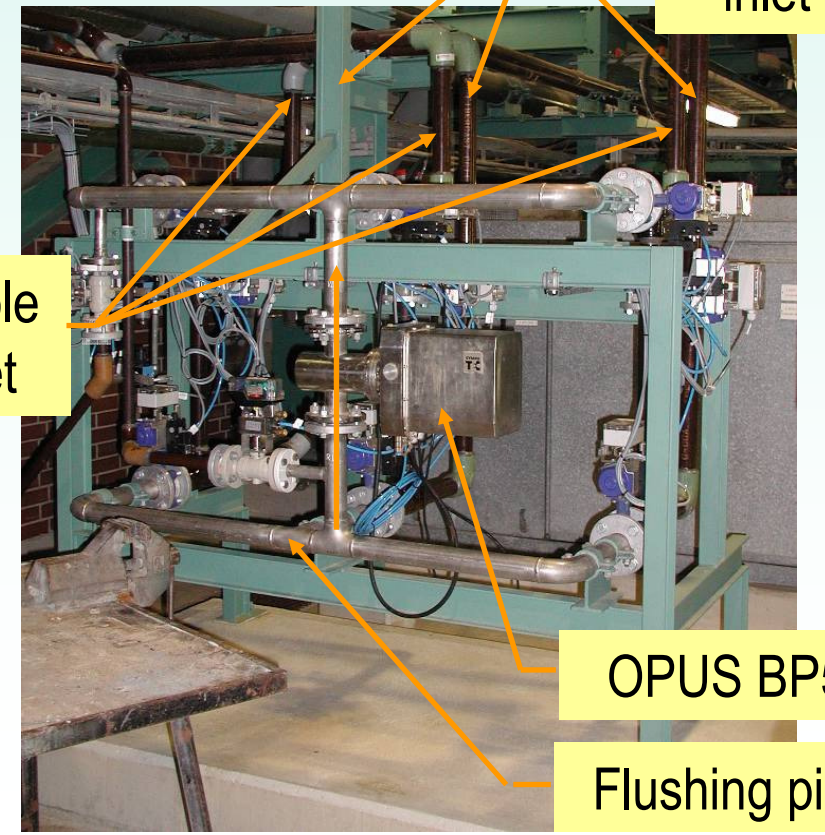
on-line / BP-Adapter / Crystallisation / building materials

- ★ Product: **Gypsum slurry (native)**
- ★ Process
  - ↳ Temperature: 120 °C
  - ↳ Pressure: Approx. 4 bar
  - ↳ Conc.: Approx. 20 % vol.
- ★ Adaption
  - ↳ BP50-Adapter
  - ↳ Between two digesters
- ★ Background
  - ↳ PSD is relevant with respect to the drying stage
  - ↳ PSD decisive for the strength of gypsum

Installation Examples

on-line / BP-Adapter / Crystallisation / Building materials

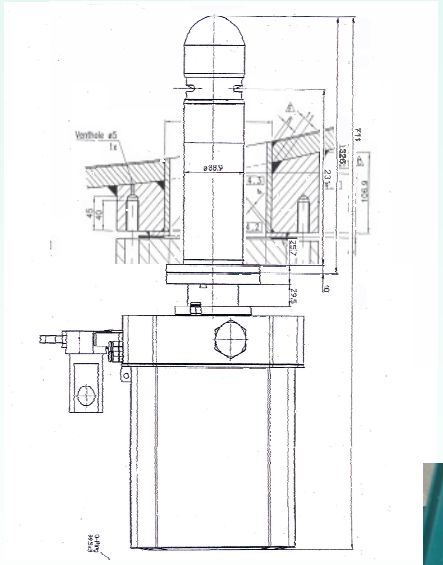
- ★ Product: **Gypsum slurry** (flue gas desulphurisation/FGD)
- ★ Central measuring stage
  - ↳ Alternating sampling from 3 lines
  - ↳ Measuring frequency 1 M / 15 min
  - ↳ Flushing between each sampling
- ★ Primary goal: Solids concentration analysis

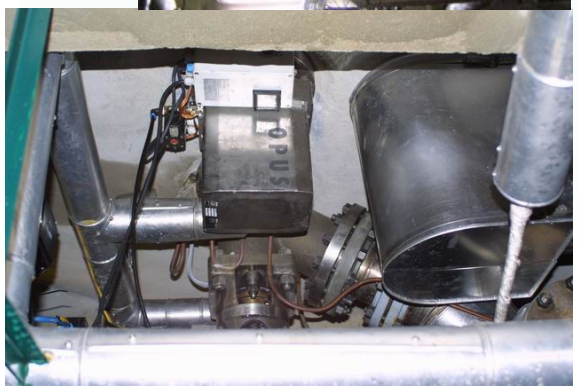


Installation Example

in-line / AF-Adapter / Polymerisation / EPS

- ★ Product: **Expandable polystyrene (EPS)**
- ★ Process
  - ↳ 12 h batch
  - ↳ 90 - 120 °C
  - ↳ 2 - 7 bar
- ★ Installation
  - ↳ Through the reactor bottom
- ★ Goal
  - ↳ Visualisation of EPS-particle growth in order to stop the process “just-in-time”



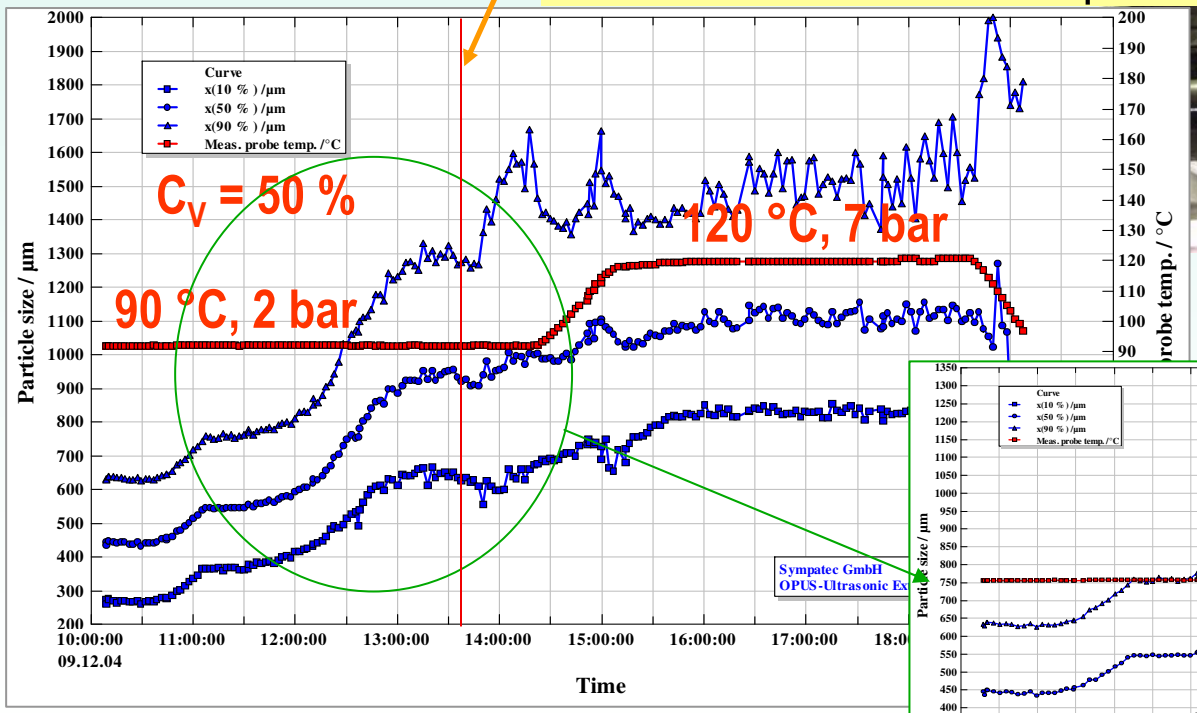


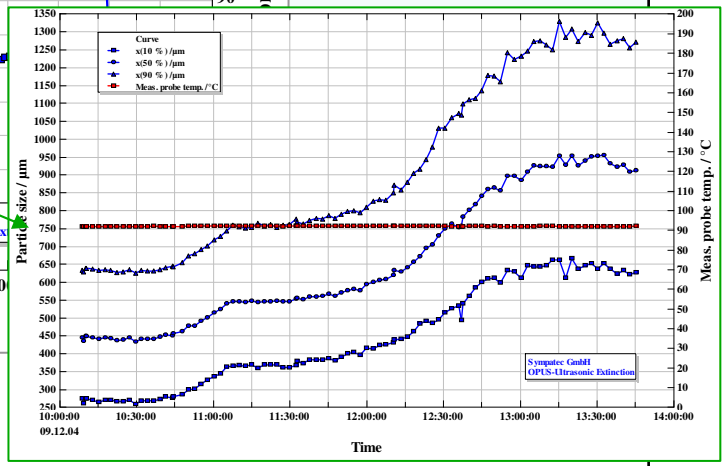
Installation Examples

in-line / AF-Adapter / Polymerisation / EPS

★ Results: **EPS**

addition of inhibitors and pentane



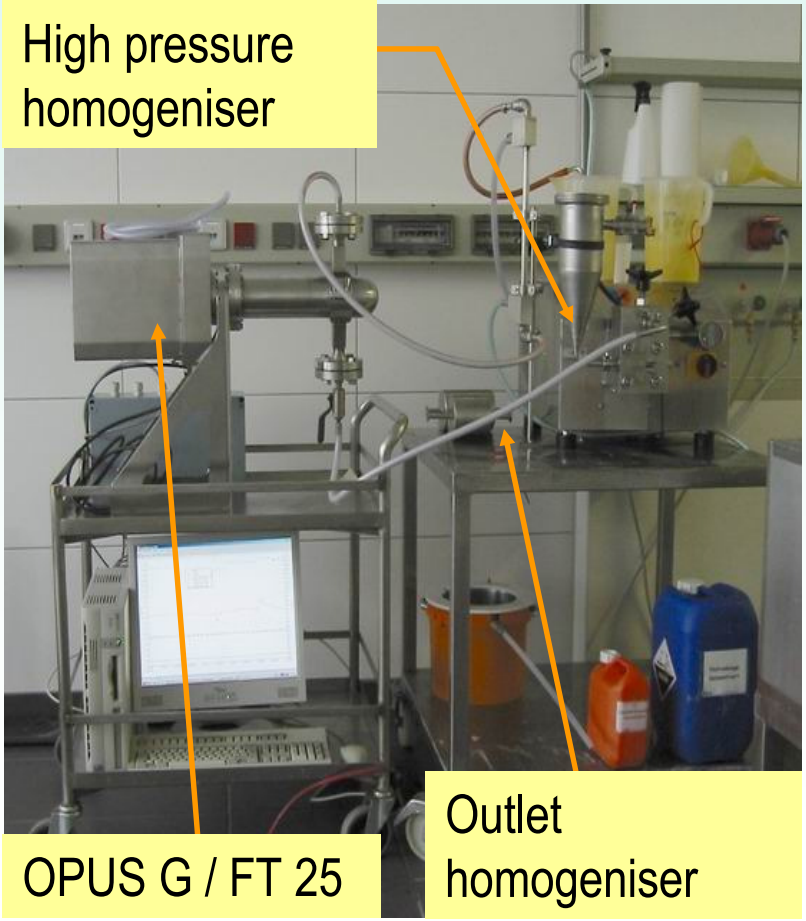


✔ Bead growth is precisely detected

Installation Example

in-line / FT-Adapter / Homogenisation / Food

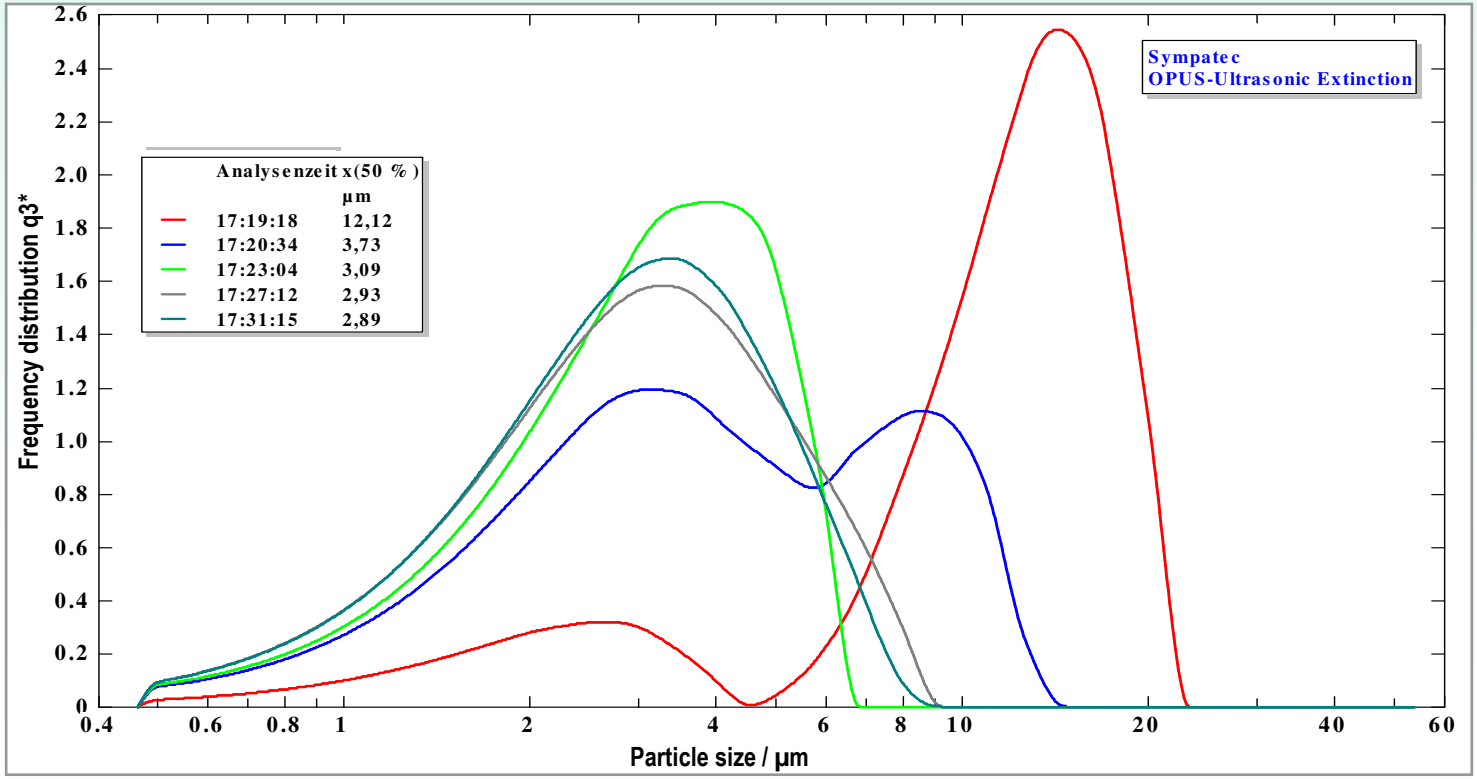
- ★ Product:
  Beverage emulsions
- ★ Process conditions:
  - ↳ 7 – 10 min.
    batch operation
  - ↳ High pressure
    homogeniser
  - ↳ Conc. 30 % vol.
- ★ OPUS adaptation
  - ↳ With FT 25 Adapter
  - ↳ In sample outlet



Installation Example

in-line / FT-Adapter / Homogenisation / Food

★ Results: **Beverage emulsions**

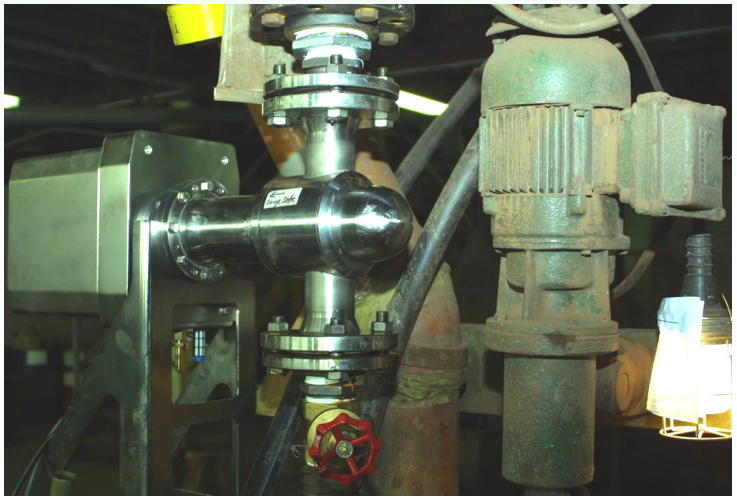


✔ Progress of homogenisation is clearly detected in real-time

Installation Example

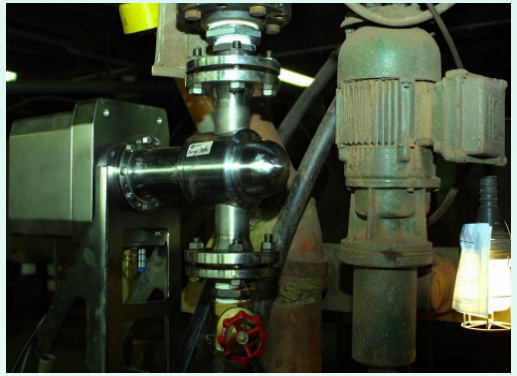
in-line / BP-Adapter / Grinding / Minerals

- ★ Product: **Iron ore**
- ★ Process
  - ↳ Ball mill 10 m³/h
- ★ Installation
  - ↳ Main process flow
    sieve under run
  - ↳ Behind cyclone
- ★ Adaption
  - ↳ BP 50 Adapter

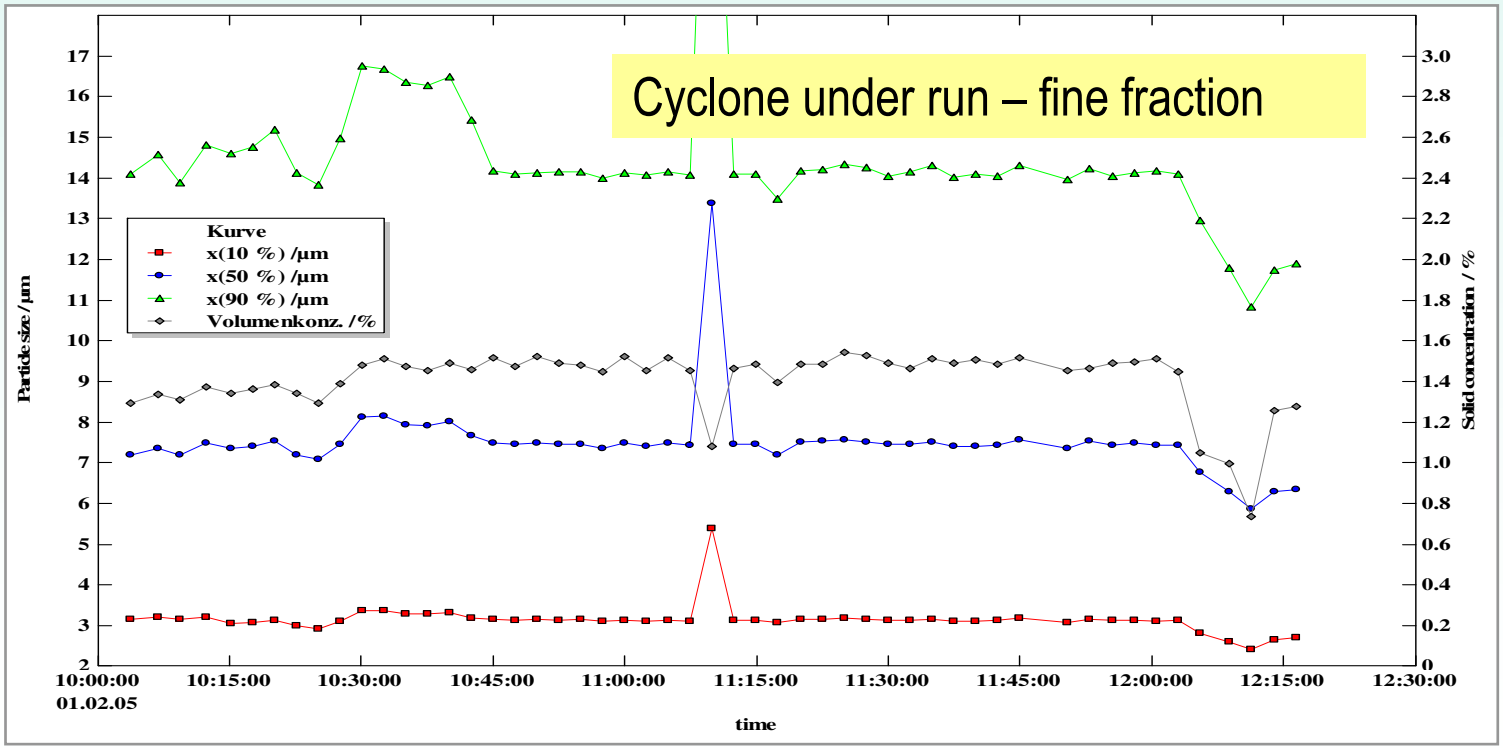


Installation Example

in-line / BP-Adapter / Grinding / Minerals



★ Results: **Iron ore**

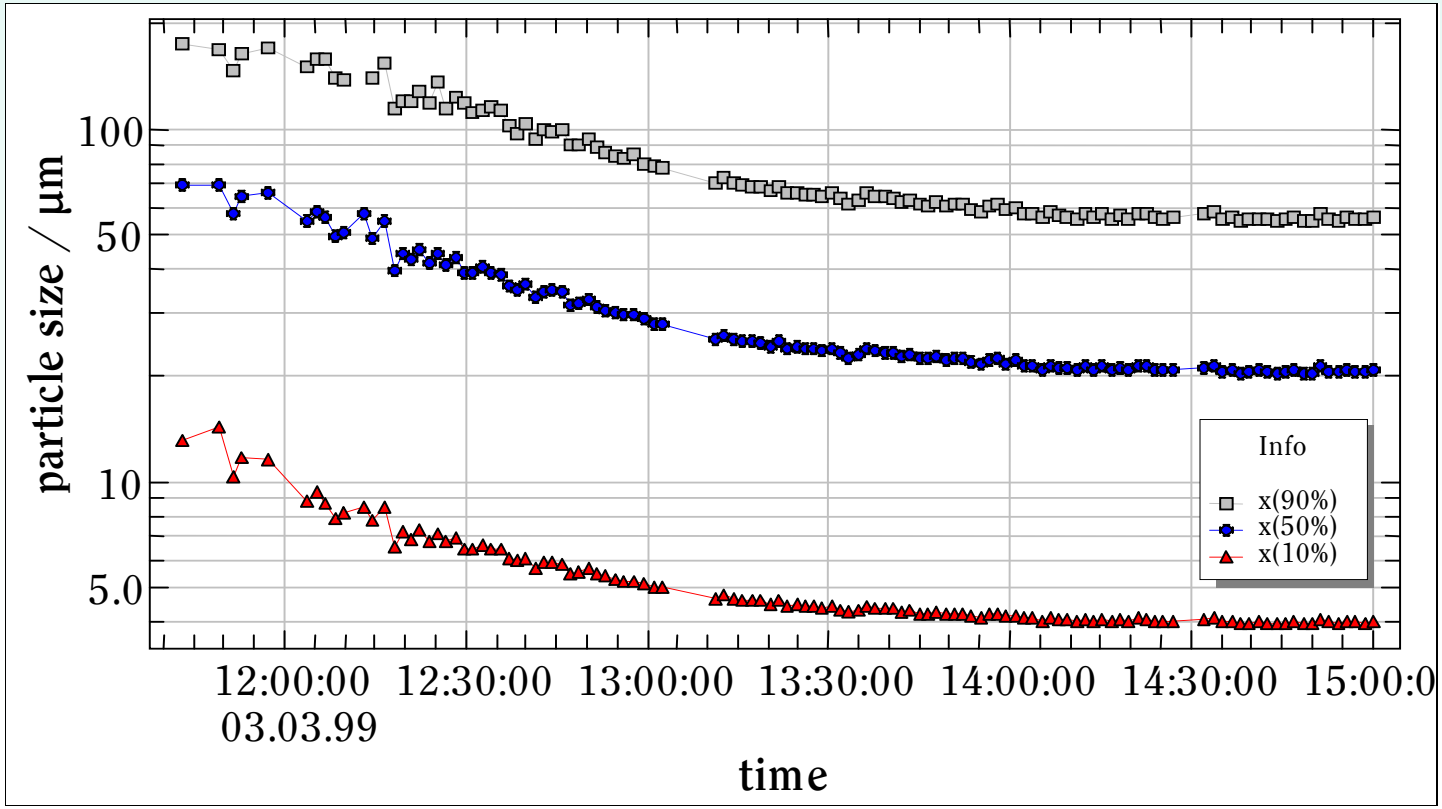


✔ Process interactions are monitored immediately

Installation Example

in-line / FT-Adapter / Grinding / Pigments

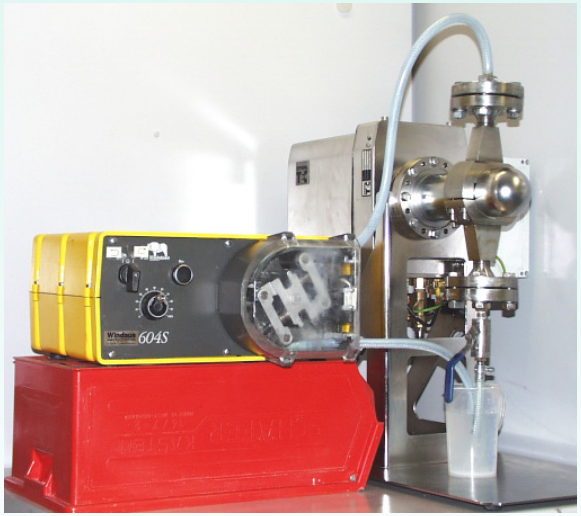
★ Results: **Metal pigments in benzene**



Installation Example

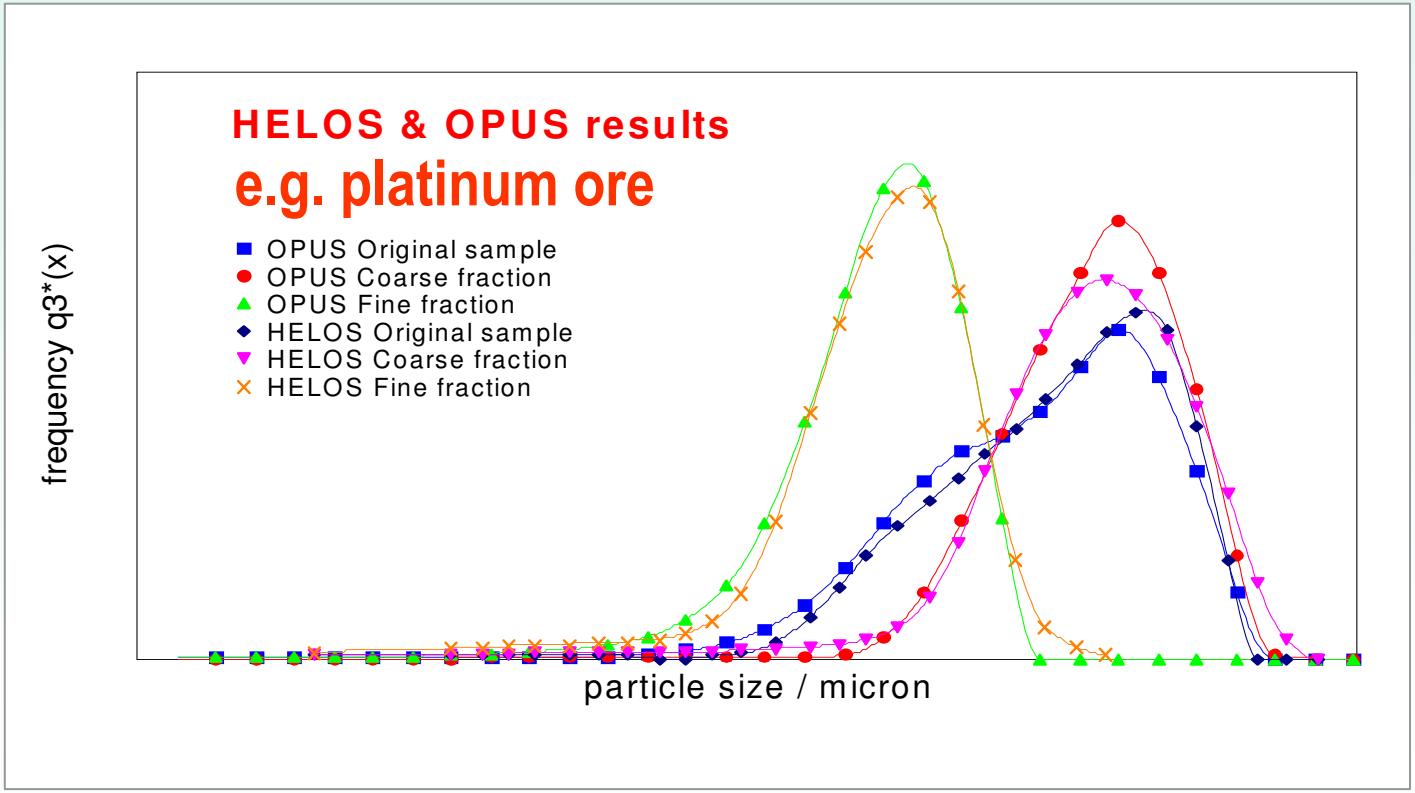
off-line / FT-Adapter / various products / R&D

- ★ OPUS in 0,5 – 1 l
  re-circulation loop
- ★ Static measurement (no circulation)
  - ↳ Sedimentation during analysis has to be avoided
  - ↳ Required sample approx. 100 ml
- ★ Product: Various
- ★ Calibration: WINDOX KSIGMA
- ★ Lab-version of USE:
  NIMBUS = "OPUS" +
  integrated circulation loop



Coherence of OPUS Measurements

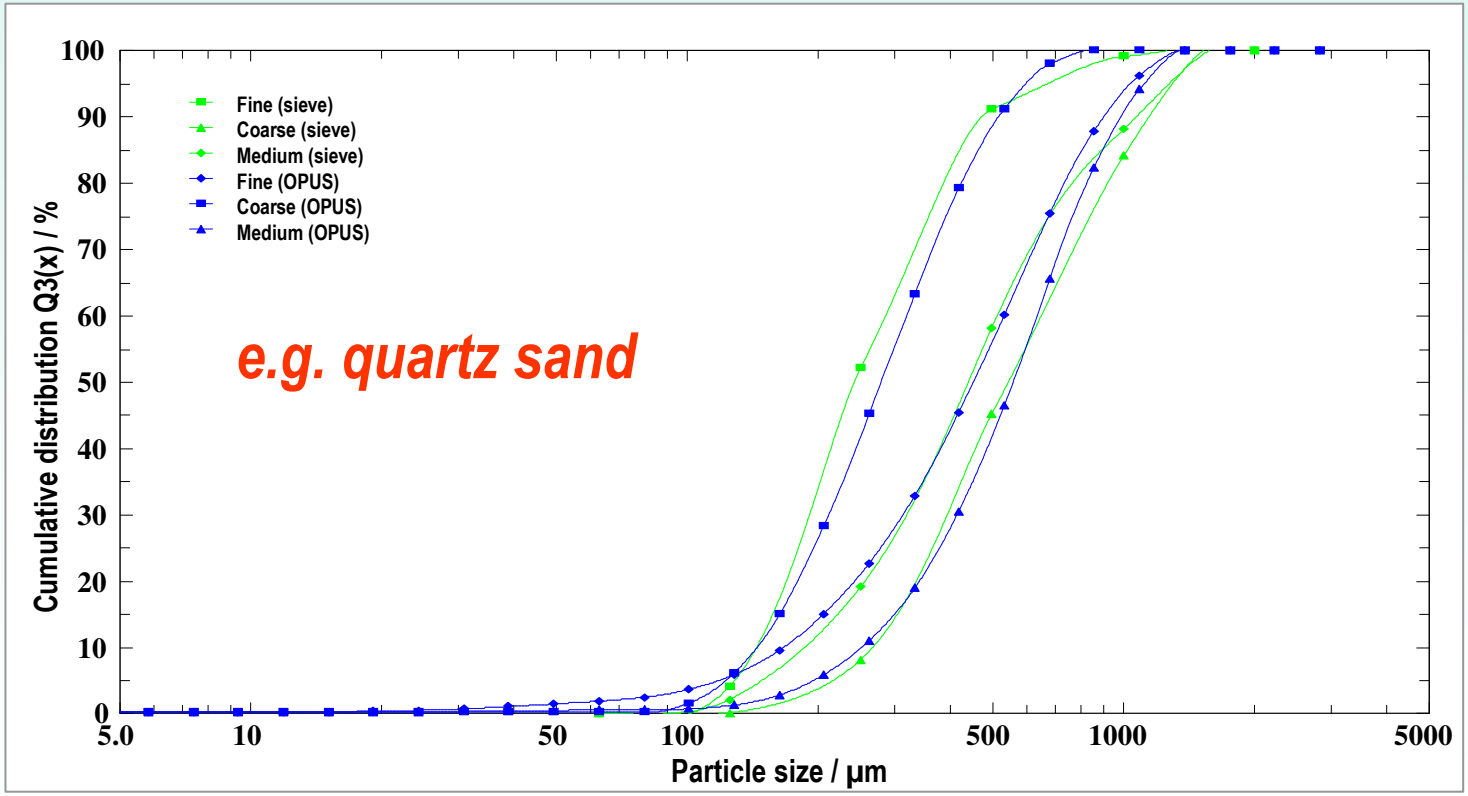
to HELOS Laser Diffraction



✔ Very good agreement between ultrasonic extinction OPUS and laser diffraction HELOS

Coherence of OPUS Measurements

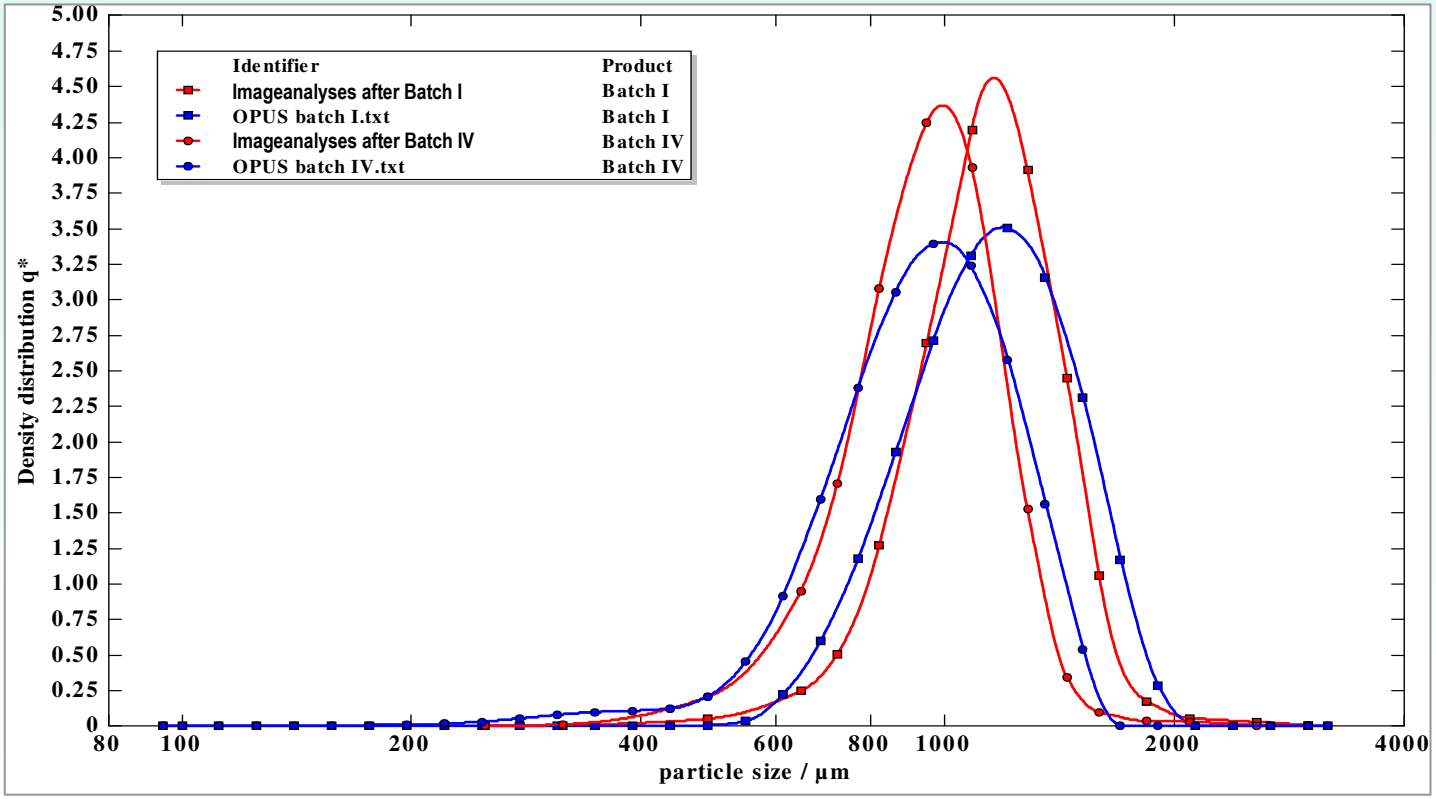
to sieve analysis



✔ Very good agreement between ultrasonic extinction OPUS and sieve analysis

Coherence of OPUS Measurements

to image analysis



✔ Very good agreement between ultrasonic extinction OPUS and image analysis

Experience (Excerpt)

- ✔ ABS polymers
- ✔ Adipic acid
- ✔ Aluminium
- ✔ Aluminium hydroxide
- ✔ Aluminium oxide
- ✔ Ammonium wolframate
- ✔ Ammonium perchlorate
- ✔ Ammonium poly vanadate
- ✔ Ammonium sulphate
- ✔ Barium sulphate
- ✔ Calcium carbonate
- ✔ Carbon black
- ✔ Chocolate
- ✔ Chrome
- ✔ Coca mass
- ✔ Colour pigments
- ✔ Dextrose
- ✔ Food colour
- ✔ Glass beads
- ✔ Gypsum
- ✔ Harbour mud
- ✔ Iron powder
- ✔ Isodecanole in water
- ✔ Kaolin slurry
- ✔ Lactitole
- ✔ Limestone
- ✔ Lithium hydrate
- ✔ Magnetic suspensions
- ✔ Mayonnaise
- ✔ Melamine
- ✔ Offset paint
- ✔ Oil in water
- ✔ Ore (generally)
- ✔ Paraffin oil in water
- ✔ Precipitate
- ✔ Petroleum in water
- ✔ Polymer systems
- ✔ Polystyrene beads
- ✔ PVC suspensions
- ✔ Quartz sand
- ✔ Sepharose
- ✔ Silicon oil / water
- ✔ Sodium chlorate
- ✔ Sodium chloride
- ✔ Sodium nitrate
- ✔ Sodium perborate
- ✔ Sodium percarbonate
- ✔ Sodium water glas
- ✔ Soft rinser
- ✔ Soot suspensions
- ✔ Suggar slurry
- ✔ Tenside suspensions
- ✔ Terephtalic acid
- ✔ Titanium dioxide
- ✔ Zeolite
- ✔ Zinc carbonate
- ✔ Zinc oxide ...