

Live Particle Sizer

Permanent information on size progression in disperse phase systems



INSITU PAT
SENSOR
TECHNOLOGY



실시간-온라인 입도 분석기 (On-Line Particle Size Analyzer)

특징 및 장점

1. Live Particle Sizer - Insitu PAT Sensors

독일 S&E GmbH 사의 Unique한 특허 PAT sensor technology를 기초로 제작된 실시간 온-라인 입도 안정성 유화도 변화의 24시간 영상 모니터링. 연구용 및 공정용으로 사용 가능하여 현재 SHELL, SIEMENS, BASF 등의 생산라인에 설치 하여 사용 중. 건식 및 습식 형태의 미세 입자 생산공정의 최적화 및 입도 균일화를 통한 품질향상 및 제조공정 자동화를 통한 품질향상과 원가절감, 공정 안정화, 불량 최소화를 동시에 가능하게하는 공정용 입도분석기로서 모든 분말 파우더 생산공정에 적용이 가능.

2. Emulsion Characterisation Analyser(ECA)

이물질 입도, 안정도 및 유화도의 변화 과정을 실시간으로 모니터링을 할 수 있는 첨단 공정용 온-라인 영상 분석 장치.



Live Particle Sizer



PAT Sensor

유지 보수 및 적용 분야

독일 S&E GmbH 사의 Live Particle Sizer 및 ECA 장치는 365 일 24 시간 무인 입도 측정이 가능하고 유지보수가 거의 필요 없이 시료 채취에서부터 분석결과 전송까지를 전자동으로 수행할 수 있는 실시간 공정입도 분석으로 응용 및 적용이 가능



적용분야

- 페인트/잉크 생산공정 (Paint, Pigment)
- 금속 및 금속산화물 분말생산 공정 (전자재료, Dielectric, MLCC, TiO2, Catalyst)
- Dry products: Aluminum oxide, microcapsules, potassium chlorides
- 흑백 및 컬러 토너 생산 공정 (Toner)
- 고분자 분말생산 공정 (PTA, PIA, PMMA, HDPE)
- 세라믹 / 유리분말 생산 공정 (HEMC, Alumina, Silica, Glass, Cement)
- Dry products: Cements
- Organic products: Organic flocculating, Carotene, Brewer's yeasts etc.
- 제약 / Bio 생산공정 (Pharmaceutical solid, Emulsion, Colloid, cell)
- Spray / Sol / Gel 생산공정 (Spray, Aerosol, Droplet)
- 화장품 및 식품용 Emulsion
- 크리스탈 형성 과정 고찰



Live Particle Sizer Insitu PAT-Sensors

Insitu PAT-Sensors for detection of particulate changes under process conditions

Sequip PAT-Sensor Series provide:

- Highest selectivity and sensitivity under original production conditions
- Optimisation of quality and purity of solids
- Increase of productivity with the formulation of new products
- Increase of production safety
- High saving of time within product development
- Production quality control 24h/365 days a year
- Ex-protection versions for operational application



Insitu PAT-Sensors measure all particles and droplets with optical back reflection properties.

Absolutely transparent products cannot be measured yet!

Clear products without large diffraction differences like CO₂ in CH₄ or polystyrol can be measured with special laser components!

As the particles tend to agglomerate with concentrations from <1 Vol% , the particle collectives are normally present as Particle Systems (PSyA).

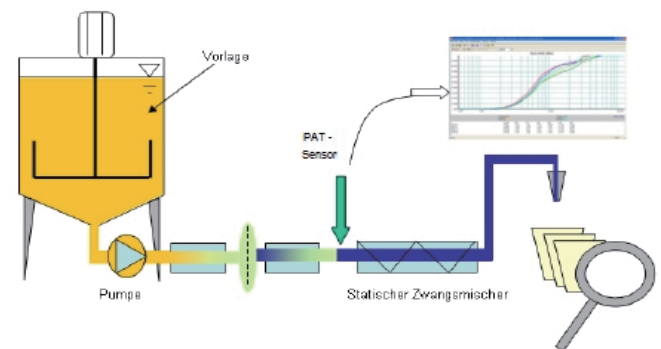
Therefore, the resultant particulate Fingerprints of the Particle SYSTEM Analysis represent reproducible indices for PAT control. Conditional to acquisition properties of agglomeration structures you can evaluate broader particulate distributions than with laser diffraction spectrometres (PSD) and get displayed the total spectrum as size index over the time. Furthermore, the roughness of the examined Particle SYSTEMS can be determined by Multi Capture Technique.

Therefore, the measurements with PAT-Sensors in original concentrated products show fingerprints of the actual Particle System Distributions under process conditions.

The changes are detected and displayed by the PAT-Sensors under original conditions at any time during production.

When the products to be measured are diluted, the sensors detect the size distribution of discrete present particles continuously as under laboratory conditions.

Thus, an optical shading is impossible.



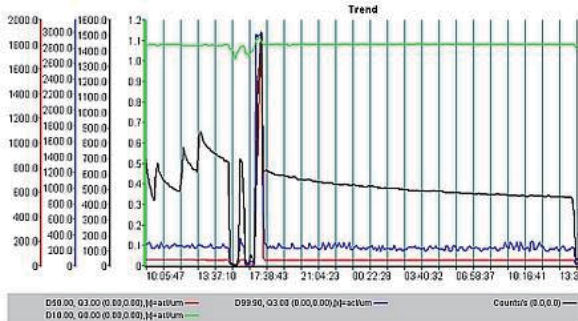
Benefits:

- All particles are counted and presented as trend value over time, based on counting results in the fraction sizes, as quantity distribution, length distribution, surface distribution or volume distribution
- Measuring periods from 1 - 300 sec
- Temperatures from minus 120°C to 300°C
- Even the laboratory PAT-Sensors are insitu sterilizable up to 165° when switched-off



Presentation of results

The following presentation demonstrates a measurement of PVC 100-125 μm (sieved on validated Rhewum sieving machine) in dependance of the concentration.



Within the area from 10:05 to 13:37 the picture shows the addition of 5g PVC each time to the total source of 10 g to 400 g water at start. Then, the mixer was turned of twice and reactivated after a short break. The measurement very quickly shows the sedimentation of the particles. At the second mixing process the rotational speed was shortly doubled, by what the count rate in counts/sec strongly increases. After reducing the rotation speed for the mixer to an experimental value of 450 rpm, the decreasing count rate demonstrates the presence of a light sedimentation effect. The measurement results still remain within the limits and are reproducible.

PAT - Sensor Benefits

Sequip Live Particle Sizer PAT-Series provide:

- Sequip Live Particle Sizer PAT-Series provide:
- Insitu control of stability of dispersphase systems
- Inline process control of particle and droplet sizes in real time
- Online quality control
- Highest selectivity and sensitivity under original production conditions
- Optimisation of quality and purity of solids
- Increase of productivity
- Increase of production safety
- High saving of time within product development

- Production quality control 24h/365 days a year
- Operation control of the sensor by use of supplied reticles
- A reliable real-time measurement system for insitu operation for counting, sizing etc. of particles within the focus
- A broad modular sensor plate allows adjustment to your requirements
- All your requirements regarding materials and physical and chemical basic conditions as far as Ex-protection can be accomplished
- Sensors with more than 70,000 operating hours are no rarity
- Self selective dynamic multi-focus-depth scanning system assures measurements of droplet and particle sizes
- Special sensors for ATEX Ex-protection or heatable sensors for crystallisation
- Quality and design "Made in Germany"

PAT - Particle Analysis

PAT Analysis of particulate systems - from beaker to tube line and the reactor, inside of the process

The Insitu PAT-Sensor is placed in the product, this may be within a laboratory beaker or directly within a supply line in pilot plant stations or process. The PAT-Sensor System ist started and the distribution is presented on screen within seconds. Changes during measurement phases are continuously displayed. The data are displayed as trend graph over time. Changes in production process can be detected at once and appropriate action can be taken accordingly. The Insitu PAT-Sensor Systems provide the opportunity to transmit data digitally and/or analogically.

Insitu PAT-Sensor Systems - Measuring Ranges and Concentration

Possible measuring ranges <0.5 to 4000 μm

Volume concentration in Vol%	Measurement of
< 5%	Particle Distributions
5 -> 40%	Particle System Distributions
above 60%	Fingerprints



Applications

- Homogenization
- Polymerisation under EX-conditions
- Dispersion
- Granulation
- Fermentation
- Disaggregation
- Flocculation
- Particle and droplet size dependent crystallization



PAT - Sensor Technology

Technology

We provide two measurement techniques for your applications:

- undiluted measurement in original concentrations
- diluted measurement



Live Particle Sizer insitu PAT-Sensors provide size distributions from diluted and original concentrated suspensions and emulsions as well as measurement results from disperse present dry products.

Process flow in practice

Practice	Particle Change	Result
1 Droplet formation	Droplets of different compounds are homogenized	Distinct distribution shift
2 Blending of pigments in emulsions	Distribution of pigments in the emulsion	After the emulsion is homogen, the emulsion raw data are subtracted from the pigments+emulsion data
3 Distribution of droplet formation during polymerisation	Droplets get larger and stabilize	Fine fraction is reduced in favour of coarse fraction
4 Dry or wet grinding	Particles getting smaller	Distribution change - higher fine fraction
5 Nucleation at precipitation	Particles grow out of nano scale	Distribution change - higher fine fraction increasing
6 Particle breakage or splitting	Particles getting smaller	Distribution change - higher fine fraction - lower coarse fraction
7 Growth of particles	Particles getting larger	Distribution change - higher coarse fraction
8 Agglomeration and flocculation processes	Particles form particle systems	Distribution change - higher coarse fraction - lower fine fraction
9 Deaggregation	Particles dissolve	Particles dissolve
10 Change of shape at crystallisation	Particles deformate	Change of distribution function

We supply you with the Live Particle Sizer insitu PAT-Sensor Systems in line with usage licences, completely configured with hard- and software. The easy handling allows you an autonomous usage. If required, our consulting service is also available.

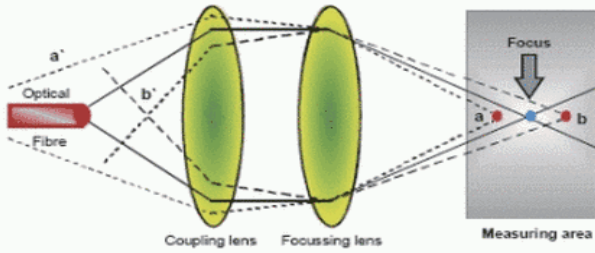
Your benefit at a glance:

TOR + ORM - Technology	The <i>Insitu PAT-sensor</i> continuously determines the „particulate Fingerprint“ of your particle systems in situ under process conditions and you increase your product quality by continuous quality control over 24 hours. The <i>unique dynamic focussing</i> provides a broad measurement range from 0.5 to 4000 µm because of continuous changes of the focus depth. By continuous acquisition of the particulate changes, deviations of size, number and shape can be detected at once, representing a major advantage for production control. The Laser Time of Reflection analysis (TOR) added by Sequip in combination with the Sequip Software allows permanent monitoring of possible production variations. The hereby achieved constancy of your products serves the safety of your company 365 days a year.
Application in process	<i>Insitu PAT-sensors</i> are mounted directly in the production plant and measure the product in its original state and in realtime. Thus, processes like e.g. crystallization processes, flocculation processes, granulation and polymerisation can be monitored and controlled accurately. A time-consuming sample taking is not necessary.
Return of Investment (ROI)	The ROI normally is a few weeks per PAT-Sensor
Time saving in product development	The <i>Insitu PAT-sensors</i> measures in original concentrations and in realtime. So, the influence of changes in particle size can be evaluated at each step of development.

Whether in beaker or pipeline - you always get an "undiluted information" under insitu conditions to reach your target.



Patented PAT-Sensor Technology - measures only particles in the focus



Multiple scatterings are not detected by the opto-mechanical construction design.

Through an only 4 µm sized single mode fiber a laser beam is dynamically focussed through a measurement probe and the inner optics into the product to be analyzed. An optical system rotating with 2 m/s inserts the laser beam spirally.

The patented dynamical focus is to be synchronized and moved with the same speed into the medium to be measured. Thus, measuring of particles in the focus only and blinding out of the surrounding area is warranted.

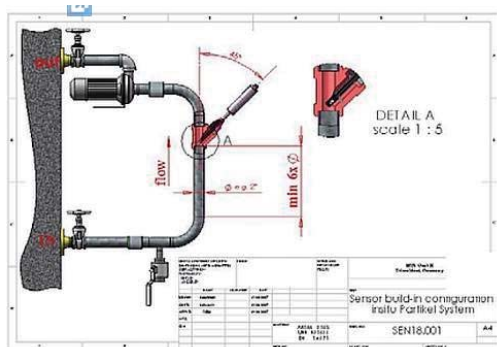
The spiral dynamical focus is the decisive advantage to conventional turbidity measurement and FBRM! (*TM by Mettler Toledo Lasentec)

Installation examples

Installation examples in practice

The installation of Live Particle Sizer In situ PAT-Sensors in industrial instruments, tanks or pipelines is carried out with appropriate mounting sockets.

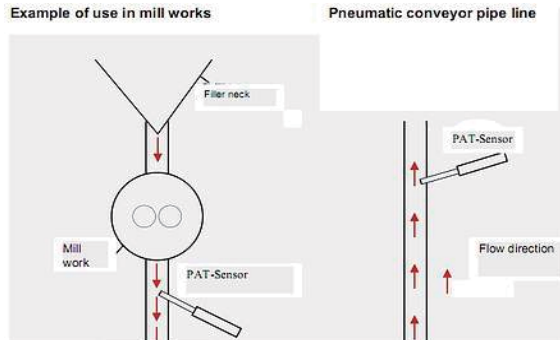
The following figures show two typical inline installation configurations of the sensor. Due to the existing flow conditions, certain installation guidelines have to be observed to achieve optimal measurement conditions.



PAT-Sensor Installation in a bypass

Optimisation of milling processes

By continuous monitoring of the milling process a deviation of the milling material from the set-point parameters is detected directly. Then, you can interfere immediately with the milling process. The Insitu PAT-Sensor detects smallest changes. Thus, the milling time is optimized and the quantity of milling cycles is reduced.



The PAT Sensors - were developed for product optimisation under process conditions

Configuration of the system:

The system consists of a patented Sensor, which is manufactured for Sequip in Hasteloy C 276 by default and the modified Sequip evaluation software (TOR), which is connected with a single mode fibre optic RJ 45 Hub cable to the Computer and the Sequip Trend Software.

Standard models and options:

Diameter of sensor at measurement window	18 mm laboratory model or 25 mm process model
Total length of the sensor at site of measurement window	240 mm medium touched site
Total length of the sensor	500 mm in standard version; extendable up to max. 3 metres
Measurement speed	Up to 30 m/s with adding of particle velocity into evaluation software
Number of measurement channels	1024 internal and 128 external
Particle sizes	In measurement ranges from <0.5 µm to 4000 µm
Pressure range	Up to 6 bar standard, optional up to 16 bar
Temperature range	From +5°C to 125 °C or 165 °C
Optimal physical requirements	possible on demand in R&D version
Measurement period per measurement	1 second to 300 seconds
Long time evaluation	6 trends as particulate ratios over elapsed time over 24 hours
Distribution	Internal sieving curve as base for trend display
Modelling / Validation	possible
Calibration:	PVC and CaOH calibration standards
Output alternative	6 x 4-20 mA; 6 x digital outputs
Shape Analysis:	Shape factors can be considered by evaluation software



Application areas

Application areas of Live Particle Sizer

PAT-Sensors	Measuring range (µm)	Cv max
125 PAT	< 0.5 to < 125	80%
250 PAT	< 0.8 to < 250	60%
500 PAT	< 1 to < 500	50%
1000 PAT	< 2 to < 1000	40%
2000 PAT	< 5 to < 2000	30%
3000 PAT	< 10 to < 3000	25%
4000 PAT	< 20 to < 4000	20%
Sensor diameter: 18 mm; Sensor length 255/478 mm		
Pressure range optional: 6 - 16 bar Cryogen sensor: < 200 bar		
Temperature range optional: 5 to 165°C or -90 to 165°C Cryogen sensor: -120°C to 20°C		

Mini Plant Sensors

MP-Sensors	Measuring range (µm)	Cv max
125 MP	< 1 to < 125	< 80%
250 MP	< 2 to < 250	< 60%
500 MP	< 5 to < 500	< 50%
Sensor diameter: 12 mm; Sensor length 160 mm		

Measurable Products from A - Z

You wonder, what, for example, is measurable of particulate product properties?

Measurable Products from A to Z

A limited selection from measured products: state 2007

aluminium oxide	gold overburden	printing colour
baby lotion	granulates 100 - 500µm	paste < 2µm
bio flocks	gypsum	raw gypsum
Bio-ointment < 10µm	hair dying products	road metal
bitter salt	hand lotion	salt 20 - 3500µm
bitter salt / magnesium sulphate	high speed granulating 20 - 850µm	seeds
bitumen emulsion 5 - 125µm	Hostaform powder < 2 - 25µm	sewer mud 0,5 - 250µm
breadcrumbs	ice creme	shampoo
brewer's yeast	Inert-gas distribution in O/W ink	silica gel
butter	ink jet slurry < 4µm	silicic gel / 2 - 250µm
calcium chloride	insuline	silicone emulsions
precipitation crystals	keto-glucol acid < 500µm	silver pigment paste < 1µm
cat litter	kidney stone	skin lotion
cellulose	lactose < 50 - 500µm	soda
cellulose slurry	latex	sorbic acid
cement < 45µm	latex paint	β-carotene eye drops
ceramic slurries < 2µm	magnesium chloride precipitation crystals	steel / water in a counter-flow
cereals	Malt products with shape recognition	styrene
chocolate	margarine	sugar
coal slurry after the grinding	mayonnaise	sugar < 800 µm
coarse meal granulate	megapearls	sulphur (polymer)
coating paint	metal powder	Sus-emulsions < 5µm
cocoa	MgCl ₂ × 6 H ₂ O	Terephthalaldehyde
coffee	micro capsules 2 - 15µm	Titanium dioxide 0.5 to 5 µm
colour pigments < 2µm	milk of lime	titanium dioxide in emulsions
coolant emulsions	multi-material emulsions	Toothpaste
copper slurries	natural cosmetics < 20µm	Uranium particles
cream	paper additives < 3µm	vaccination sugar < 150 µm
crystals in emulsions < 125µm	PCC	vitamin flocks
defrothing agent	peeling lotion	w - o - w emulsion
deodorant lotion	penta-erythrite	washing liquid
dextrose	phosphate gravel 100 - 800µm	washing powder
dextrose monohydrate	photo emulsion	washing powder pearls
diamond slurry	pigments: yellow, red, black < 4µm	water lacquer 15 Vol. %
emulsions + micro capsules	polyethylene (PE)	waxes / < 4µm
eye drops	Polymer + defrothing agent	wood shavings
feed	polymer granulate	yeast
fermentation	shape recognition	yoghurt
fertilizer	polystyrene	o-w-o emulsion
fodder	polyvinyl chloride (PVC)	
gas bubbles in vegetable fat	potassium chloride	
Gas pockets in margarine (PIA)	powdered paint	
glucose and other saccharine	precipitated gypsum	
	primary matter for washing agent	



Droplet Size Analysis - Cryogen Sensor

In situ measurement of particle and droplet size in Cryogen-application in a real time measurement with the Sequip-Sensor

Droplet size and number of particle measurement in liquid pressured gas with a sensor for insitu operation

Application In situ measurement of particle and droplet size distribution the reaction of precipitation in Cryogen-application



Figures 1 and 2: Sequip-Sensor and evaluation-electronic

Cryogen- Applications with Live Particle Sizer:

1. Monitoring of droplets in liquid gas
2. Measuring of particle and droplet size inside liquid gas
3. Number and size of precipitation particles inside liquid mediums
4. Control of extractive cleaning processes of gas
5. Display of the reaction of precipitation and crystallisation at temperatures from minus 120°C up to 300°C and vacuum pressure up to 300 bar
6. Ex-proof versions available

Inline Particle in Cryogen applications under real time production conditions

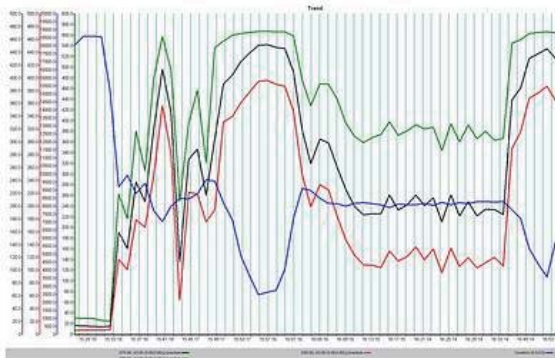


Fig. 3: shows the reaction of precipitation of particles based on different temp and pressure in a liquid gas application as a function over time

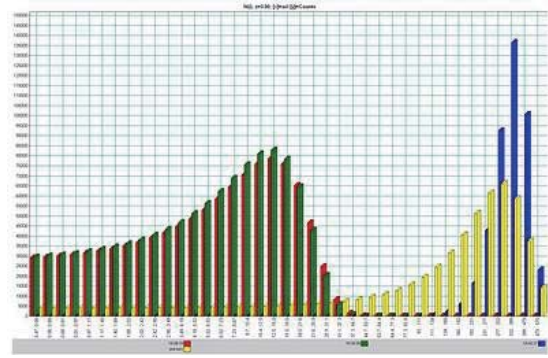


Fig. 4: Histogram shows the reaction of precipitation of particles based on different temperatures and pressure in a liquid gas application during different time and pressures with temperature up to minus 120 °C

Benefits of the Sequip Live Particle Sizer System with an insitu PAT- Cryogen Sensor:

- Inline measurement of particles and droplets inside a liquid gas at cryogen temperature
- Different versions of sensor configuration are available to create a modular concept of combined technologies of ORM and ToF in combination of other linear scanner systems .

Live Particle Sizer is using several versions of in situ PAT Cryogen-Sensor technology under real time conditions in this Cryogen high-pressured process condition, which has international patents.

Sequip has developed several special sensor systems, which allows the customer to work in low temperature and high-pressure Cryogen-applications under in line and on line process conditions in real time.

These Sequip cryogen -sensors can directly be mounted in line the pipe work at the customer side, also under explosion proof conditions.

Sequip is taking over the exclusive distribution of PAT- Cryogen- technology by MTS within a cooperation since January 2008.



Fig. 5: Cryogen-sensor with a bypass measurement cell
Fig. 6: Cryogen-construction of medium touched side



PAT - Sensors Technical Data

Live Particle Sizer

PAT Sensor for lab and insitu process application and pilot plant and mini plant operations

Application:

The PAT- Sensor facilitates measurement of particle sizes and droplet sizes in original suspensions and emulsions under insitu conditions. Thus, the development of particle sizes in flowing dispersions can be observed in real-time. For this, the sensor of the PAT- Sensor is placed insitu directly into applications with undiluted dispersions, e.g. directly into beakers or mini-reactors at formulation of new products. Observe development of crystals and crystal growth, agglomerations of particle systems, stability of disperse phase systems, dissolution processes of substances with the PAT- Sensor

The time-consuming sample taking and sample preparation is unnecessary.



Mode of Operation:

The PAT- Sensor -Technique is based on the Time of Reflection (TOR) and the Optical Back-Reflexion Measurement (ORM). A laser beam scans the dispersed particles and detects their geometric expansion when passing the laser beam. The detected lengths are displayed as a statistical graph. The statistics classify the measured sizes in 128 logarithmic arranged size classes. The measurement period for acquisition of the statistical data can be selected between 1 second till 5 minutes. As the TOR / ORM measurement method depends on the optical visibility of the particles in the dispersion, it is advisable for economical reasons to perform a test measurement with critical substances (transparent particles, strongly absorbing homogenous phases of the dispersion) with a rental system.

Live Particle Sizer

Specification

Dynamic insitu PAT - Sensor

Hardware	
Electronics	Desktop Case, IP 54 Data Interface RJ 45 Bus operating voltage 230V , 110 V on request
LWL- cable	5 m cable between sensor and electronics
Data cable	350 m cable between electronics and PC
Automation	
Software	Sequip ORM-Software is part of delivery content Microsoft Windows 2000 and XP compatible PC is not included in delivery content but can be provided completely configured on request.
Installation and requirements	
Installation and Training	1 day software training after setting-up operation by Sequip and one day training on site Within the first year we allow a rebate of 10% on application related consulting fees.
Documentation	The user manual is supplied as book or CD in English or German language.
Requirements	Installation of the in situ PAT Sensor: In line sensor configuration for use in reactors and pipelines: The process has not to be interrupted to install a flange and the system. (By Pass Installation see figure below) Electrical supply 100 – 240 Volt AC; 5 AmaiPS, ground cable is required. PC should be installed and connected in a dust free surrounding with controlled temperature.

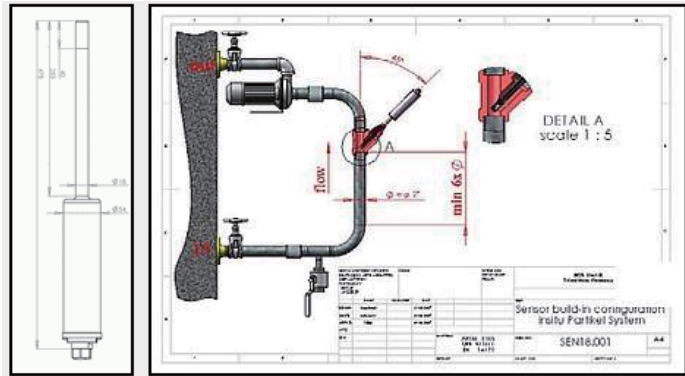
Type	Measuring range in micron	Concentration vol%
125 PAT	< 0.5 to < 125	80
250 PAT	< 0.8 to < 250	60
500 PAT	< 1 to < 500	50
1000 PAT	< 2 to < 1000	40
2000 PAT	< 5 to < 2000	30
3000 PAT	< 10 to < 3000	25
4000 PAT	< 20 to < 4000	20
Sensor diameter: 18 mm; Sensor length 255/478 mm		
Pressure range optional: 6 - 16 bar Cryogen sensor: < 200 bar		
Temperature range optional: 5 to 165°C or -90 to 165°C Cryogen sensor: -120°C bis 20°C		



Mini Plant Sensor Dimension: diameter: 14 mm; length: 160 mm

Mini Plant-Sensors	Measuring range micron	Concentration vol.%
125 MP	< 1 to < 125	< 80
250 MP	< 2 to < 250	< 60
500 MP	< 5 to < 500	< 50

Specifications	
Sensor Material	1,4571 (SS 316) for all metal parts touched by medium to be measured – Special materials on request electro / chemical polished sapphire window at measuring head with optical quality class MIL- PRF-1383B 10-5 Viton – O-rings (other material on request)
Max. operation pressure	16 bar
Sensor operation temperature	5°C to 85°C or if requested -20°C to 165°C
Industrial protection	Dust and water proof. measuring head IP65, casing IP54
Installation	3000 m – max. distance between measurement device and PC Max. amount of devices to be connected with one PC: 1
Validation	Validation and 21 CFR part 11 - <i>Option on delivery 2009</i>
Software	SequipORM
Dimensions	as per drawing
Weight	15 kg (Sensor + electronics)



PAT-Sensor
Dimensions

Bypass Installation of Sensors