



Insitu Particle System Measurement in Lab and Process

Insitu PAT Sensor







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Insitu PAT Sensors

The patent-registered PAT Sensor technology quantifies size and number of particles in originally concentrated dispersion based on the optic measurement of back reflection in connection with ToF (Time of Flight) technology. The influence of process parameters on the product will be identified fast and effectively. In contrast to normal particle analyzers PAT Sensors work with a *Self Selecting Focus* which guarantees the highest resolution of particles in the focal point.

Insitu PAT Sensors identify the particle size distribution of thinned and originally concentrated suspensions and emulsions as well as results of disperse present dry products. By the transparent signal analysis based on the newest developments of laser-based analysis technology by Sequip the operator is able to see his actual results under insitu conditions.

Size, the profile of the signal and the surface finish of the particle will be identified and analyzed to result in a real image of the particle system.

Advantages

- $\sqrt{10}$ Based on facts statistical analysis because of more than 100.000 signal pictures
- $\sqrt{10}$ Fitting of the sensor directly in reactor or in a pipeline possible
- \sqrt{No} sample taken
- $\sqrt{1}$ Insitu operation from petri dish up to beaker and reactor

Thereby:

- \Rightarrow Stability control under process conditions
- \Rightarrow Faster development of new formulas
- \Rightarrow Quality saving 24h a day



Basics of Technology

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Basis of success: ORM Technology

Patented ORM technology (Optical Back Reflection Measurement) allows an analysis of the whole particle system because of operation of selective working focus system.

In short the laser beam leaves the optical fibre (shown as a solid line). It passes through the coupling lens and the focussing lens, rejoins at the focal point scanning the sample in an elliptical orbit. If the light of the laser beam hits a particle or droplet at the focal point, the reflected light is then sent back to the optical fibre on the exact same path.



Moving of the focus into the medium:

- 1D -> static focus
- 2D -> circular moving focus
- 3D -> spiral vertical moving focus

An optical mechanical system conducts the laser beam spiralled. The patent-registered dynamic focus is synchronized and moved with higher speed into the medium to be measured. It is assured that the particles are only measured in the focus and the surrounding with the other particles is faded out.

Multiple scattering won't be registered because of the patent-registered opt mechanical design of the sensor.

The spiral dynamic focus in connection with the patent-registered opt mechanical technology is decisive advantage for the normal haze measurement and the Laser Scattering Radar Analyzer (LSRA) (see p.8).

The product to be measured has to be optical back-reflecting and have enough difference between trigger medium and product.



Basics of Technology

Multi Capture Signal Shape Technology Analysis

Based on the previously described patented 3D ORM technology Sequip developed **Multi Capture Signal Shape Technology Analysis (MCSSTA™)**.

The factor of the signal form and profile-dependent signals is captured and evaluated by a high performance electronic system. Information on the surface finish and profile of the particle are necessary to identify the real size and the change of profile.

All signal images will be saved in the Multi Capture Signal Analysis and are available to be evaluated by the electronic system.

This high-developed technology is applied by the systems **IPAS** (Insitu Particle Analyzing System), **IMAS** (Insitu Morphology Analyzing System) and **APAS** (Advanced Particle Analyzing System) by Sequip.







Modular customer-oriented product control with Insitu particle analysis

LSRA—Laser Scattering Ra- dar Analyser Utilization: Product control and improvement	Displays of fingerprints – changes inside the particle system are detected. Function: extended laser measurements with optical back reflection
PMS—Particle Monitoring System	Control in definitely adjusted size range of the particle sys- tems under production condi-
Utilization: Process control and improvement, 10-<300µm, 30-<600µm	tions. Function: 1D ORM technology
ECA—Emulsion Characteris- tic Analyser Utilization: measurement of drop size in emulsions and	Undiluted measurements in laboratory and insitu process in real-time.
slurry, 0.5 – 125µm	Function: 3D ORM technology
	·······
PAT Sensor	Undiluted measurements in laboratory and insitu process
Utilization: insitu Particle	of dispersed phase systems in

Utilization: insitu Particle analysis with different metering ranges, 2-2000µm

real-time. Function: 3D ORM with a moving depth selective focus

Advanced Highly Developed

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Product Range

Real particle through multi capture analysis

IPAS—Insitu Particle Analysing System

Utilization: Collection and analysis of signal forms to define morphologically crystals and particles; 0.5-2000µm

IMAS—Insitu Morphology Analysing System

Utilization: insitu particle analysis in dynamic measurement range, crystallization and polymerization processes, identification of product specification and form inhomogenity; 0.5-2000µm

APAS—Advanced Particle Analysing System

Utilization: insitu particle analysis with dynamic measurement range and analysis of form signals—open signal tap by an oszilloscope exit for research applications 0,5—4000 µm Undiluted measurements in highest concentration in laboratory and insitu process in real time, dispersion of the signal form.

Function: deep, variable and selective focus with integrated signal form analysis

Undiluted measurements in highest concentration in laboratory and insitu process in real time.

Function: Multi Capture Signal Analysis plus identification of geometrical signal form functions and roughness and size of crystals and particles.

Undiluted measurements in pumpable concentration in laboratory and insitu process in real time.

Function: Multi Capture Signal Analysis plus identification of geometrical form factors, roughness and size of crystals and particles with open signal structure.

End Advanced Technology

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Laser Scattering Radar Analyser

The principle

To capture the homogeneity of products an analysis electronic systems seizes and counts different particle sizes which produce different multi scattering effects, and sorts them in different length categories by their signal length.

The signals are capture by a wide, launched angle and a lens system inside the sensor and presented as a signal length distribution. This distribution of all signal length during a period of time is an advanced signal distribution of the Multi Scattering range. The signals proportional to the size are captured and presented from a particulate system of disperse phase systems, which develop under process conditions because of the Multi Scattering effects.

The raw data is saved and can be relayed to the Process Control System of the operator.

Applied characteristics

On the basis of previously set tolerances for the "fingerprint" of the measured product you can influence and control the quality of the process.

The system calculates the characteristics of the current trend. If it is possible that the limit of the tolerance is over travelled, the unit will alarm the operator and calculate the time until the frontier crossing. If the trend changes, the alarm is deactivated and the process keeps running (see diagram).



Laser Scattering Radar Analyser

Application-benefits for the operator

- Optimized control and capture of crystallization processes, capture of the starting point of germination and control of the fine and roughness fraction rates by optical density allocation and detecting of the limit point of crystallization.
- Functionality for Lasentrack[™] and FBRM[™] customers is saved.
- PAT control as a fingerprint of batch and continuous processes to constant product quality
- Control of automatically sampling to reduce time in laboratory
- Capture of agglomeration and deagglomeration processes

Benefits for the operator by sensor technology

- Static lens system inside the sensor, therefore you have maintenance rates of 5 years
- Design of the medium-touching parts according to customer's specification, for example C 276
- Polymer-free seal system therefore prevention to crystallization inside the seal chamber
- Norm sensor diameters of 14, 18, 25 mm and mini plant sensors
- Sensor length of 150 up to 4000 mm therefore even existing reactors can be equipped through manhole
- Sensor pressure range: Vacuum up to 16 bar
- Medium temperature: -40° to +165°C
- Optional extension regarding the application conditions for OEM customers possible
- Best price/ gain proportion
- Multivendor-capability, cost-efficient installation by the customer!
- Logical structure of the analysis software.



Process control: 365 Days / 24Hours

Sleep well, the sensor is working for you!

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INSITU PAT SENSOR TECHNOLOGY

PMS Particle Monitoring System

The principle

To control continuously a defined fraction of a particle distribution a one-dimensional selective focus captures all particles which are in the focus with a factory-provided narrow static size-range.



Particle size parameters are controlled continuously in line, in real time and 24h. Sampling are made, when the PMS sensor collect irregularities. Therewith, the laboratory released from controlling the samples which are in the specific range.

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Technical Datas

Measurement range: Concentration:	<10 –300 µm and < 30 – 600 µm < 40 %
Temperature:	-120° up to 220 °C
Pressure:	Up to 16 bar
0	14 mana 10 mana 04 mana

Sensors: 14 mm, 18 mm, 24 mm

Options possible !

Application range:

Suspensions:

E.g. aluminum oxide, sulphur (polymers), water-based varnish, lactose, glucose, sugar, calcium chloride precipitation crystals, microcapsule, potassium chloride

Dry products:

E.g. cement, pigments

Organic products:

E.g. organic flocculating, putrid slimes, fermentations, vitamin flocculating, yeasts, insulin, carotene, brewer's yeasts





ECA Emulsion Characteristic Analyser

The principle

The ECA sensor is used to analyze drop sizes in original concentrated emulsions. The ECA sensor technology is based on the Time of Flight (ToF) technology and the optical back reflection measurements (ORM). A high-energy laser beam scans with 10mW the dispersed particles and drops and their structure. The beam captures their geometric extension, when they cross it in the dynamic selective focus. The recorded times in microsecond range are displayed as a distribution.

Utilization:

The ECA sensor enables to measure the change in size in original formulations of suspensions and emulsions, when you have insitu conditions with minimum distance of 1µsec to the next measurement between to drops.



Graphic display of the signal formation

Technical data

Measurement range: Concentration:	≥0,5 –<60 µm and < 2 – <125 µm < 70 % (oil / water)
Temperature:	5° - 85 °C
Pressure:	Up to 6 bar
Sensors:	18 mm

Interpretation with the WIN ORM—Software

Example of an instable emulsion



Example of a stabile emulsion





ECA Emulsion Characteristic Analyser

Watch with the ECA-Sensor :

Agglomeration of material systems, Stability of dispersphased systems Dissolution process of substances





Inline Fitting between mixer and homogenisation

Sensor in undiluted emulsion

Advantages for user:

No sample taken Development of new products in shorter time Real results because of measuring in original concentration Sensor is sterilisable—not working Continuously measurement 24h / 365 days a year

Insitu Labor Particle Sizer

Lab device in addition to serveral laser diffraction systems

The principle

The sensors are used to analyze suspensions and their systems in wider size spectra to have easier laboratory application additional to the common laser diffraction, like Malvern Mastersizer, Cilas, Horiba etc.

The insitu 3D sensors use the patent-registered 3D ORM technology with its ToF method as a basis. A focused laser beam moves circular above a particle flow. The size is defined by the period of time that is necessary to scan the focused particles. The sum of the data is used to create a statistical firm distribution.

The special kind of the selective 3D ORM measuring room and the combination with a electronically evaluation of the back reflection pulse are the particular characteristic of the 3D ORM technology.

In the statistical evaluation are just particles that are directly in the focus. Particles and drops outside the focus are excluded from the statistical evaluation by the patented optic.

Technical Data

Measurement range:	0,5 –125 μm 1 – 500 μm
Concentration:	< 40 %
Temperature:	5° up to 165 °C
Pressure:	Up to 6 bar
Sensor:	18 mm, 25 mm

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Application range:

- √ Insitu particle system measuring
- \checkmark Optimization and control of crystallization processes
- \sqrt{C} ontrol of polymerization processes
- $\sqrt{}$ Dynamic dispersion of disperse phase systems in original concentration
- $\sqrt{10}$ Addition to existing lab devices for agglomerisation and precipitation formulation
- \checkmark Synchronized operation with ultra sonic disperser, e.g. Dr. Hielscher
- \checkmark Drop and particle system dispersion in suspensions and emulsions
- \checkmark Low cost version available



PAT Systems

Insitu Particle Analysing System—IPAS

The principle

Based on the ToF technology of the PAT sensors (cf. page 12) the focus is moved circular and also vertically into the particle flow.

By the signal tracing the dynamical and selective measurement range is aligned automatically for the crystal and particle sizes in the range of <0.5 up to 2000 μ m.

Technical Data

Measurement	0,5 – 2000 μm
range Concentration:	< 40%
Temperature:	-90° to 220 °C
Pressure:	Vakuum up to 16; 32; 64 or 300 bar
Sensor:	14 mm; 18 mm; 25 mm; 30 mm; 45 mm



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Insitu Morphology Analysing System—IMAS

The principle

Additional to the IPAS system also signal factors are captured and signals dependent on the form are evaluated by high performance electronic system. Information on the finish and the size of particles and crystals are prepared for a real particle image.

The evaluation is made by the Multi Capture Signal Analysis. All form signals are saved and available for the evaluation.

The open signal system is based on a high performance laser system. Surface structures are capture as a signal form, presented and saved in which particle pitches of 1 μ sec are detected.





PAT Systems

APAS - Advanced Particle Analysing System

The principle

Sequip offers the APAS for basic research in universities and research institutes. The technology is based on the already commented methods and the Multi Capture Signal Shape Technology.

The decisive advantage is the possibility to grip raw signals by an oscilloscope exit. Therefore it is possible to research and explain conclusions of the particle signal development.

This system represent the high end solution for particle measuring technology!





PAT Systems

Advantages for the user

- \Rightarrow Insitu stability control in production without sample taken and pump
- \Rightarrow In production control in real time
- \Rightarrow Online quality control with reducing lab analysis
- \Rightarrow Optimisation of solid matter quality and product cleanness
- \Rightarrow Formulation of new products in shorter time
- \Rightarrow Quality saving 24h / 365 days a year





Benefit

- \Rightarrow Modular systems reg. customer specification
- \Rightarrow Dynamic measurement ranges
- \Rightarrow Very low and very high temperature range
- \Rightarrow ATEX available
- \Rightarrow Working in high pressure and concentration ranges
- \Rightarrow Comfortable analysing software
- \Rightarrow Statistically results—based on facts
- \Rightarrow Scale up and down



Installation

The insitu PAT sensors are installed in industrial apparatus, basins or pipes with a installation branch. The following picture shows the typical inline installation of the sensor. Due to the flow conditions you have to pay regard to special installation guidelines to have optimal measurement conditions.



Example: Installation of a sensor in a milling plant and a pneumatic conveyor line as well as in a reactor

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Measured products

Examples of products from 20 years of particle measurement

Aluminum oxide Solution of washing powder Eye drops Skin cream for babies Beta-carotene Brewer's yeasts Organic flakes Organic ointment Epsomite Bitumen emulsion Butter Calcium chloride precipitation crystals Antiperspirant cream Dextrose Dextrose monohydrate Diamond slurry Emulsion paint Print colors Print colors paste Fertilizers Iron ore slurry Emulsions of microcapsules Antifoam Pigments Putrid slimes Fermentations Commercial sauce Emulsions of fruits Fruit juice concentrate Animal feed Gas bubbles in margarine Gas bubbles in Mouse au Chocolat Gas bubbles in vegetable fat Grain Medical plaster Precipitation of plaster Glass powder Glucose Gold colliery waste Granules Hair tinting lotion Ground meat /mincemeat /sausage meat Hand lotion Skin lotion with pigments Yeasts High speed granulation Chipped wood Hosta form powder Immunization sugar <150 µm Inert gas distribution in oil Yoghurt Insulin Coffee Coffee extracts

Coffee concentrates Cocoa Cocoa butter Potassium chloride Lime milk Caramel Cat litter Ceramic slurries <5µm Ketoglucolic acid Ketchup sauces Silica gel Silica gel 2 – 250 µm Sludge (s) Carbon slurry after milling Crystals in emulsions <125 µm Coolant emulsions Copper ore <125 µm Copper ore slurry Lactose Lactose precipitation Lactose powder LPG liquid Air bubbles in diet oil Air bubbles in LPG Magnesium chloride precipitation crystals Malt products with form identification Margarine MDF shavings Flour Multicomponent emulsions Molasses Metal powder Metal powder suspensions $MgCl_2 \times 6 H_2O$ Micro capsules Milk 1.5 and 3.5 % fat Natural cosmetics Kidney stone Orange juice NIVEA cream Breadcrumbs Paper fiber Paper additives PCC Peeling lotion Penaten cream Pentaerythritol Seeds Phosgene crystals Phosphate gravel 100 - 800 µm Photo emulsions Casein paint Polyethylene (PE) Polymers and antifoams

Polymer granules Polymer crystals Polystyrene Polyvinyl chloride (PVC) Porcelain slurry Pulp Powder lacquer PVC emulsion **PVC** suspension Shaving foam Plaster rock Activated sludge Salad creams and dressings Salt Foam dissolution Chocolate Bruised grain granules Sulfur crystals Shampoo Silver pigment paste Silicone emulsions Soda Sorbic acid Steel / water in counter flow Coating color with additives Styrene Disperse phase Ink Titanium dioxide Titanium dioxide in emulsions Tomato juice Toner slurry Over plus sludge Uranium particles Vitamine flakes Wax emulsions Washing base Washing lotion Washing powder Water-based varnish Fluidization granules Toothpaste Cellulose Cellulose slurry Cement <45µm Cinnamon Sugar crystals



Our Service for you

Our service:

- + **Guidance round about solutions for your particle measurement** We offer consulting by phone or individually face to face about suitable systems for particle measurement before purchase decision.
- + **Measurement with your samples** You will send your samples to our laboratory and we measure them with the suitable system and send the results to you.
- + **Consulting with our personal and our system at your company** If you have a particle measurement problem? Our specialist will guide you and measure with our system at yours.

Reference customer

Shell; Fraunhofer Institut Physikalische Prozesstechnik; Siemens; BASF; Universität Cottbus; Weleda;Switzerland TU Wismar, Solvay Sanofi UNIST, Ulsan, Korea Unilever China, Germany

A special thank on this place to our partner and customer, which give us suggestions and feed back. Every discussion and every request brought us considerably closer to improve our technique.

We also thank to universities, which use our technology for Master- and doctoral dissertation. These insights gained are integrated in our research and technology work.

Because : more than hundred thousand of particle measurement systems are available and also many different solutions: We have the right one for you!

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