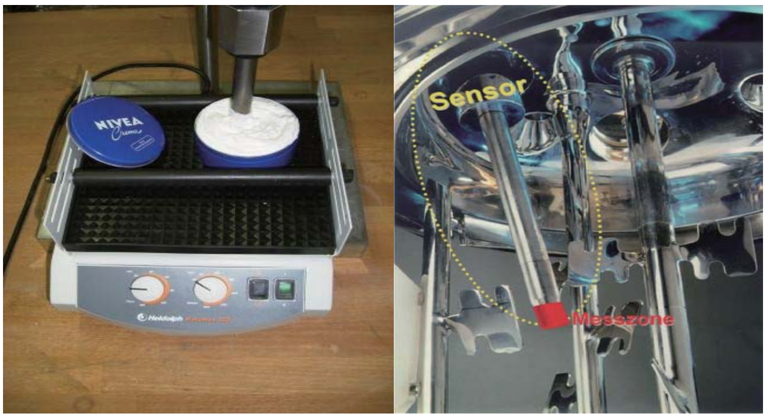
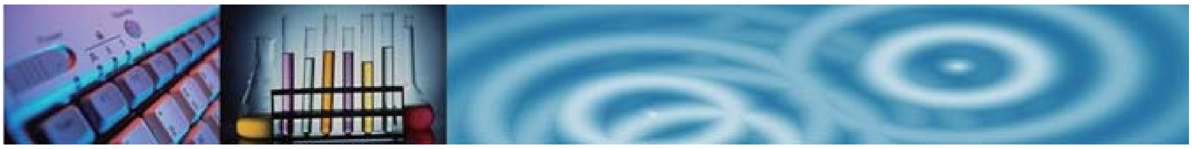
ECA

Emulsion Characterisation Analyser
Stability characterization in emulsions



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 입도분석기로써 모든 분말 파우더 생산공정에 적용이 가능.

2. Emulsion Characterisation Analyser(ECA)

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

Live Particle Sizer

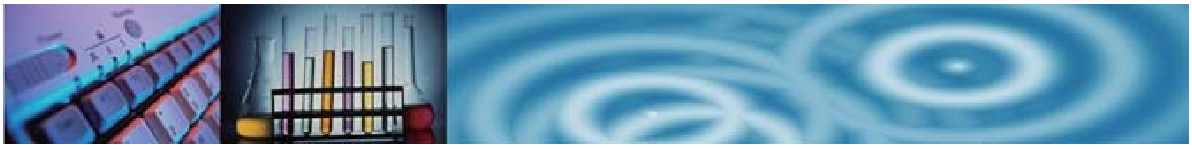
PAT Sensor

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 체취에서부터 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



ECA–Emulsion Characterisation Analyser

ECA - Emulsion Characterisation Analyser

Droplet sizes in emulsions

Migration- and stability analysis
of high concentrated disperse
phased emulsion and suspension
with insitu sensors in lab and pilot plant

ECA Application

ECA- Sensor allows measuring of size changing in original formulas of suspension and emulsion under insitu conditions with a minimum distance of 1 µsec between two droplets.

Therefore the formulation can be detected in streaming dispersed phase in real time.

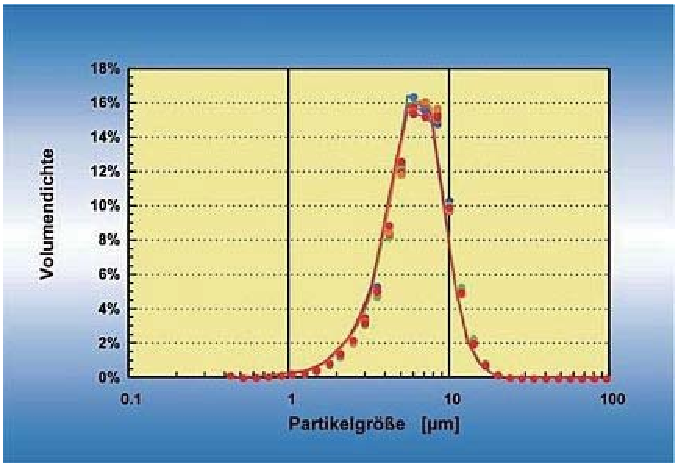


Fig. 01*: Results of replicated measurements on a 20 % o/w emulsion demonstrate that in situ particle size analysis highly reproducible.

Particle size was measured the with an ECA .
In parallel, we prepared freeze fractured replicas of the samples and examined the specimens by TEM.
The results are shown in fig. 02 to 04:

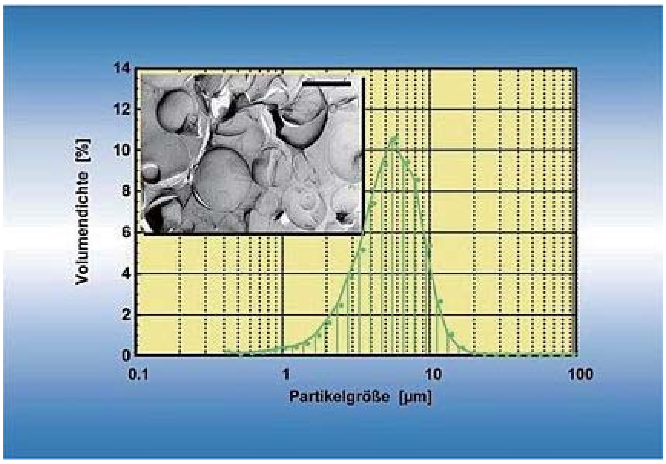


Fig. 02*: Volume distribution and corresponding TEM micrographs of o/w emulsions with **20, 40, 60, and 80 % oil content; bar: 5µm.**

For this experiment the ECA Sensor will directly insitu placed in undilluted original concentrated dispersed phase. For instance in a beaker or mini reactor for formulation of new products

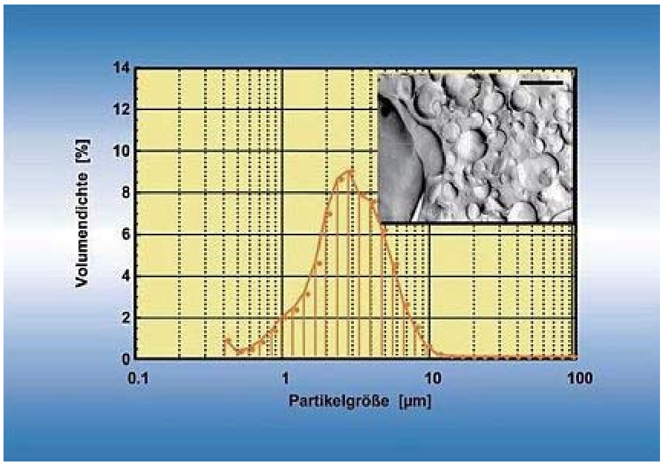


Fig. 03*:

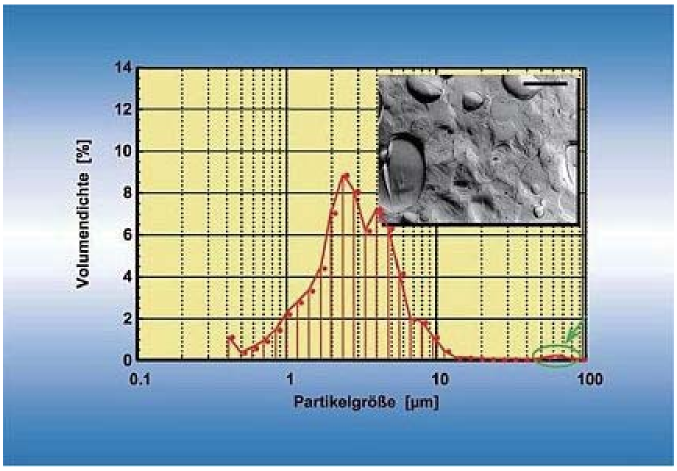


Fig. 04*:

Emulsions with 20 % oil phase revealed the most shallow size distribution. By increasing the oil content the spread broadens and the mode of the distribution moves towards smaller values. 80 % emulsions gave almost the same particle size distribution as the 60 % emulsions.
Only a small but reproducible peak in the size range from 50 to 80 µm indicated the inhomogenity of this preparation. However, this is not sufficient for a quantitative characterization of the system.

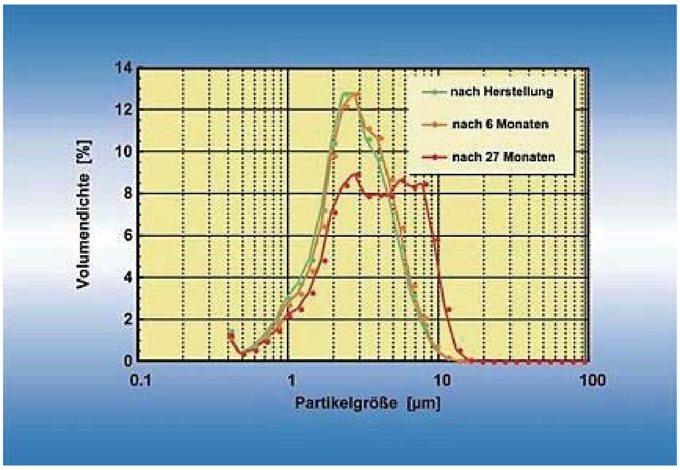


Fig. 05*: Particle size of the model emulsions after preparation, 6 months, and 27 months storage emulsions containing 20 % oil,

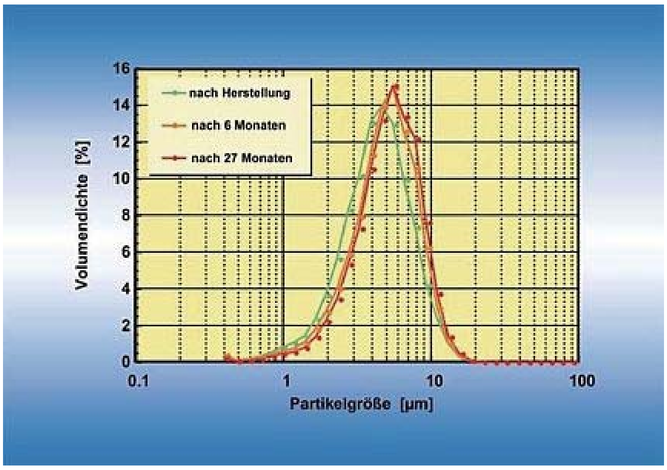


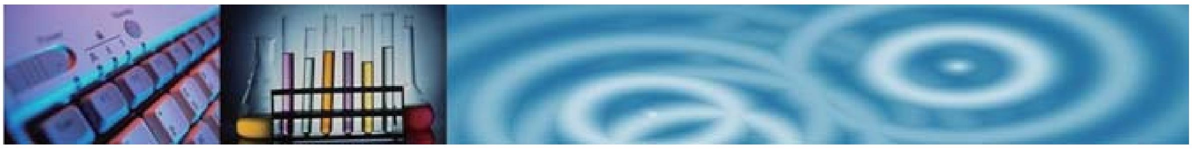
Fig. 06*: Particle size of the model emulsions after preparation, emulsions containing 60 % oil.

The measurement enables to monitor coalescence in 60 % emulsions as well as Ostwaldt ripening which occurs in 20 % emulsions. From these results, we conclude that 3D ORM technology is able to in situ characterize emulsions with up to 60 % oil content.
The method is sufficiently sensitive to monitor severe changes due to coalescence as well as to trace small changes produced by Ostwaldt ripening.
Emulsions with 80 % oil could not quantitatively be characterized. However, we must bear in mind that these emulsions are susceptible to dilution.
Thus, reliable results can only be expected when the samples can be analyzed without extensive dilution. Thus, this challenging problem cannot be solved by currently existing methods.
The results of the ORM-Measurement, however, can be taken as a fingerprint which is characteristic for this sample and allows to identify changes which occur during manufacturing or storing.

Detecting with **ECA - Sensor** :

- Agglomeration of products,
- Stability of dispesphased systems,
- Dissolution processes of substances.

No sample taking and preparation needed.



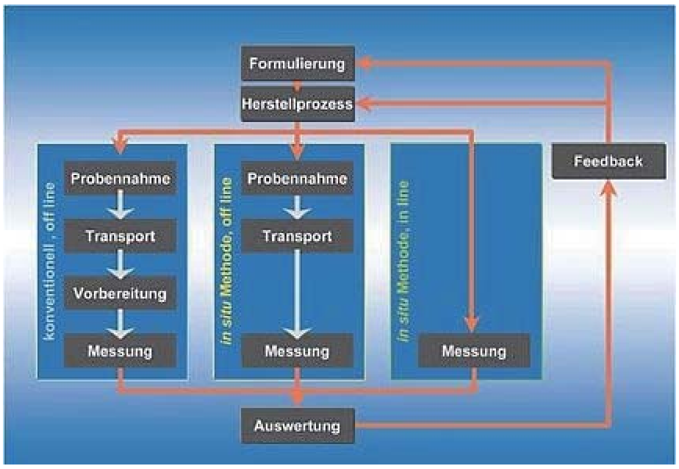


Fig. 07*: Comparison of the required steps
for particle size analysis.

)*Fig. 01 to 07 and 13+14, kindly provided by Prof. Rolf Daniels, Lehrstuhl für Pharmazeutische Technologie, Eberhard Karls Universität Tübingen.

ECA Mode of Operation

ECA-Sensor-technology based on Time of Flight Technology (TOF) and Optical back Reflection Measurement (ORM) and is a further development of the well known 3D ORM technology.

Particles and droplets and their structures were detected and measured by a laser beam with higher energy as 3D ORM and with <10mW. At crossing the particles and droplets the laser beam detected their geometric expansion. This time periods in µs will displayed in a statistic of all counted events.

The patented measuring method of the optical visibility of the particles works in a band of wave length of 680 nm. So 300 nm will be the deepest detection limit for diffuse dispersphased systems. Clear dispersphased systems can also be detected with a laser power more than 10 mW.

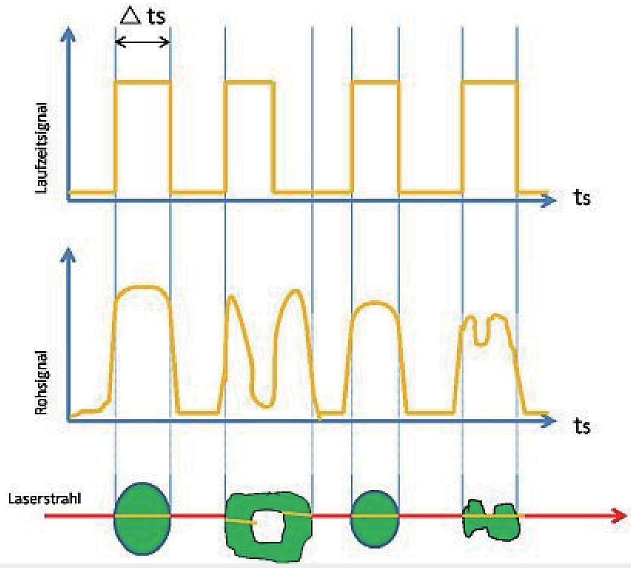


Fig. 08: Display of a signal orgination

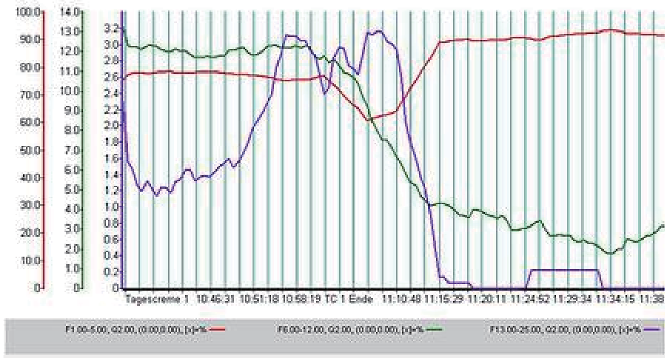


Fig. 09: instabile formulation of a sun lotion

ECA Results

Results of inline stability analysis

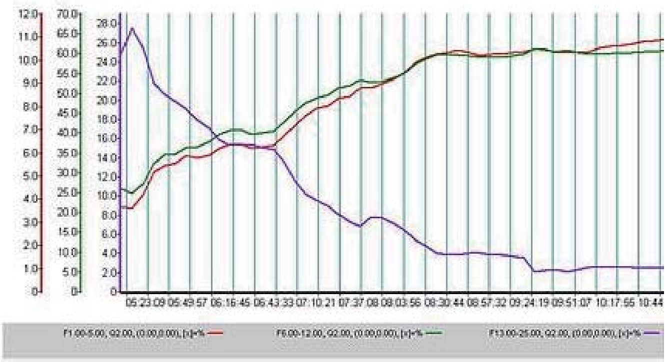
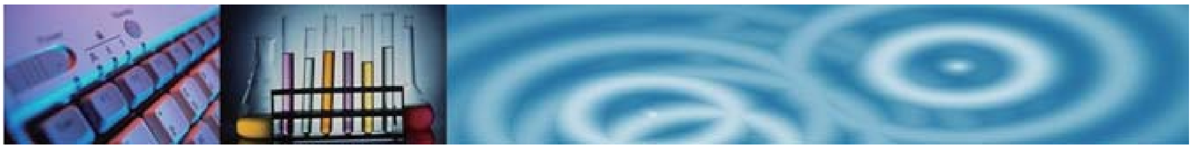


Fig. 10a: instabile formulation of a cleaning lotion

Sequip Sensor & Equipment GmbH



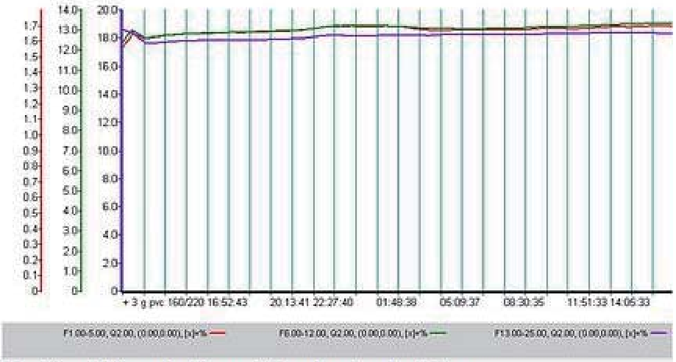


Fig. 10b: stabile perfect formulation of a cleaning lotion

Fig. 11: Inline installation in a homogenizer

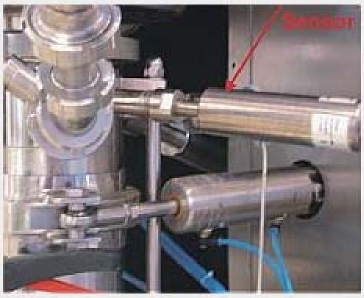


Fig. 12: Inline installation between mixing machine and homogenizer

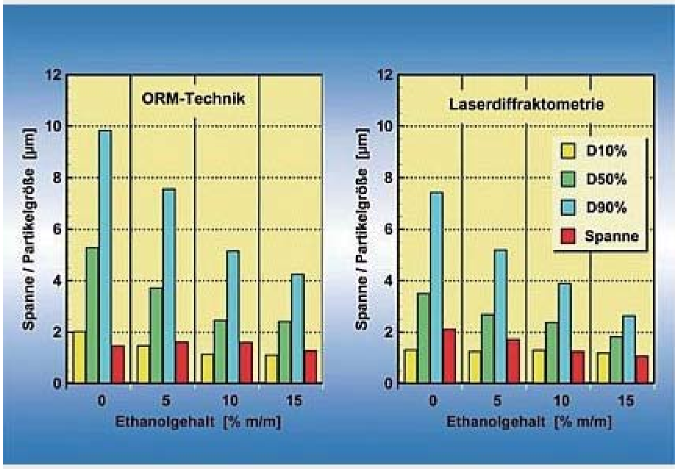


Fig. 13*: Comparison of volume distribution and span of model emulsions containing different amounts of ethanol obtained from ORM measurement or laser diffraction.

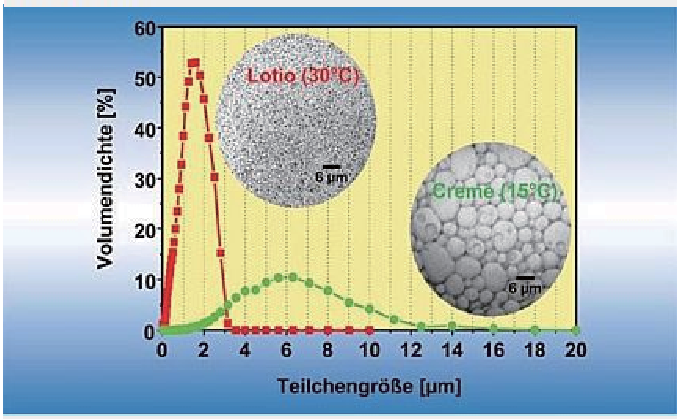


Fig. 14*: Volume distribution of a w/o type lotion and a o/w cream which have identical composition and use both ethylcellulose as polymeric emulsifier but they were prepared at different temperatures (15 or 30 °C).

ECA Technical Data

Dynamic insitu ECA - Sensor

Hardware

Elektronic	cabinet IP 44; optionally IP69 Data connection RJ 45 Bus voltage 230V , when required 110 V
Optical fiber	2 m cable between sensor and electronic

Automation

Software	SeQuip ORM–Software ist Bestandteil des Lieferumfanges Microsoft Windows 2000 and XP compatible without PC

Installation and conditions

Installation and Training	1 day at the customer
Documentation	Manual in English available
Requirements	Fitting of the in situ ECA Sensor: Inline Sensor configuration for lab reactors 100 – 240 Volt AC; 5 Amai PS, earth cable needed Electrical connection Dust free environments with controlled temperature for the PC

ECA - Sensors

Type	Measuring range in µm	Max. concentration Cv in %
40 ECA 60 ECA 125 ECA	< 0,5 – < 40 < 0,8 – < 60 <1 - <125	80 75 70
Sensor dimensions:	diameter 10 mm; length: 200 mm diameter 14 mm; length 300 mm diameter 18 mm; length 255/ 478 mm	
Conditions:	Pressure: optionally vacuum up to 6 bar	
	Temperature: optionally 5 up to 85°C **Out of action insitu sterilizable up to 165°C!**	

Hardware Specifications

Material of the Sensors:	1,4571 (SS 316) for all parts which are in contact with the medium –other materials as required Electrochemical polished sapphire window at the sensor head optical grade: MIL– PRF-1383B 10-5 Sealing: Hifluor – O-rings (other materials as required)
Max. operating pressure:	Vacuum up to 3 bar, Optional 16 bar
Working temperature	Plus 5°C - 85°C, optional minus 20 - 165°C
Installation	300 m – max. distance between measuring device and PC Max. 4 sensors, which can be connected with the PC
Validation	*Option: Validation and 21 CFR Part 11*
Software	Sequip ORM
Weight	15 kg (Sensor + Electronic)

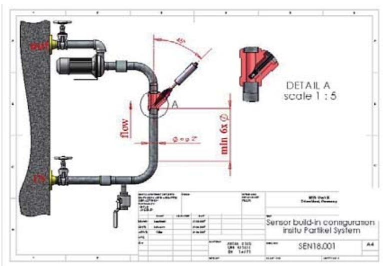


Fig. 15: By pass Installation of the sensors in a recirculation pipe