SensoTech

Polymerization monitoring

Inline analytical technology for

- monomer concentration
- polymer concentration
- degree of polymerization
- polymerization break off

Increase quality, save resources: LiquiSonic®. With high-quality, innovative sensor technology. Robust, precise, user-friendly.

LiquiSonic®

LiquiSonic® is an inline analytical system for determining the concentration in liquids directly in the production process. The analyzer is also used for phase separation and reaction monitoring. Sensor installation within the product stream means an extremely fast measurement that responds immediately to process changes.

User benefits include:

- optimal plant control through online and real-time information about process states
- maximized process efficiency
- increased product quality
- reduced lab costs
- immediate detection of process changes
- energy and material savings
- instant warning of irruptions in the process water or process liquid
- repeatable measuring results

LiquiSonic's® ‚state-of-the-art' digital signal processing technology guarantees highly accurate, fail-safe measuring of absolute sonic velocities and liquid concentrations.

Integrated temperature detection, sophisticated sensor design, and know-how from SensoTech's extensive measurement history in numerous applications promises users a highly reliable, long life system.

Advantages of the measuring method are:

- absolute sonic velocity as a well-defined and retraceable physical quantity
- independent from conductivity, color or optical transparency of the process liquid
- installation directly into pipes, tanks or vessels
- robust, all-metal, gasket-free sensor design with no moving parts
- corrosion-resistant by using special material
- maintenance-free
- use in temperatures up to 200 °C (390 °F)
- accurate, drift-free measurements
- stable measurements even amid gas bubbles
- controller connection capacity reaching up to four sensors
- data transmission via fieldbus (Profibus DP, Modbus), analog outputs, serial interface or Ethernet

Inline process analysis

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Fundamentals of polymerization

Introduction

In connection with the need to closely monitor and control processes, the capability to determine conversion in chemical reactions in general and in particular in polymerization reactions is of outstanding significance.

Just like concentration measurements, conversion measurements are becoming more and more important in all branches of industry on account of their impact on the efficiency of processes, their potential for material and energy savings, quality improvements as well as for environmental reasons.

For measuring concentrations and conversion, a number of measuring methods have been developed, including methods based on density measurement, refraction index measurement, conductivity measurement, the measurement of color, turbidity and viscosity. All of these methods are characterized by specific physical and technological limitations.

It has been known for quite some time that concentrations can be determined by measuring the sonic velocity, and this method has become the standard measuring technique in recent years.

Physical fundamentals

The propagation velocity v of ultrasonic waves in liquids is dependent on their density and adiabatic compressibility in accordance with the following relationship:

$$v^2 = \frac{1}{\rho \cdot \beta_{ad}}$$

v = sonic velocity
ρ = density
βad = adiabatic compressibility

The fact that the compressibility is the determining variable for the sonic velocity causes that, as the sonic velocity increases, density and compressibility may show a differing behavior. This, in turn, causes that even if there are only minor differences in density or none at all, large differences in the sonic velocity may occur. It very rarely happens that the reverse case takes place.

The sonic velocity is determined by the structure of the material concerned, i.e. by groups of atoms and molecules, isomerism or chain lengths. This correlation, thus, allows to characterize materials with the help of ultrasonic.

The table below shows the sonic velocity v of a few selected monomers and polymers at 20 °C.

As concerns monomer and polymer systems, it generally applies that the differences existing in the sonic velocity between monomers and polymers are primarily determined by the chain length and the extent of their branching and cross-linking. The table already shows significantly that the differences existing between monomers and polymers are extremely large and, thus in some extent, also between start and end of polymerization reaction.

Monomer	v [m/s]	Polymer	v [m/s]
styrene	1,354	polystyrene solid	2,330
vinyl chloride	897	polyvinyl chloride solid	2,260
vinyl acetate	1,150	polyvinyl acetate, dispersion 50 wt%	1,940
butyl acrylate	1,233	polybutyl acrylate, dispersion 50 wt%	1,375
butadiene	961	polybutadiene, solution 20 wt%	1,373

Processes

Depending on the reaction mechanism the polymerizations are divided into:

- solvent polymerization
- emulsion polymerization
- suspension polymerization
- polycondensation

Depending on the number of copolymers and the product changing additives, the change of sonic velocity shows a characteristic course. Typically, the sonic velocity of all components involved is determined depending on the temperature to be able to compensate it later on. Then it is possible to derive the course of reaction from the time course of the sonic velocity and to calculate the materials conversion.

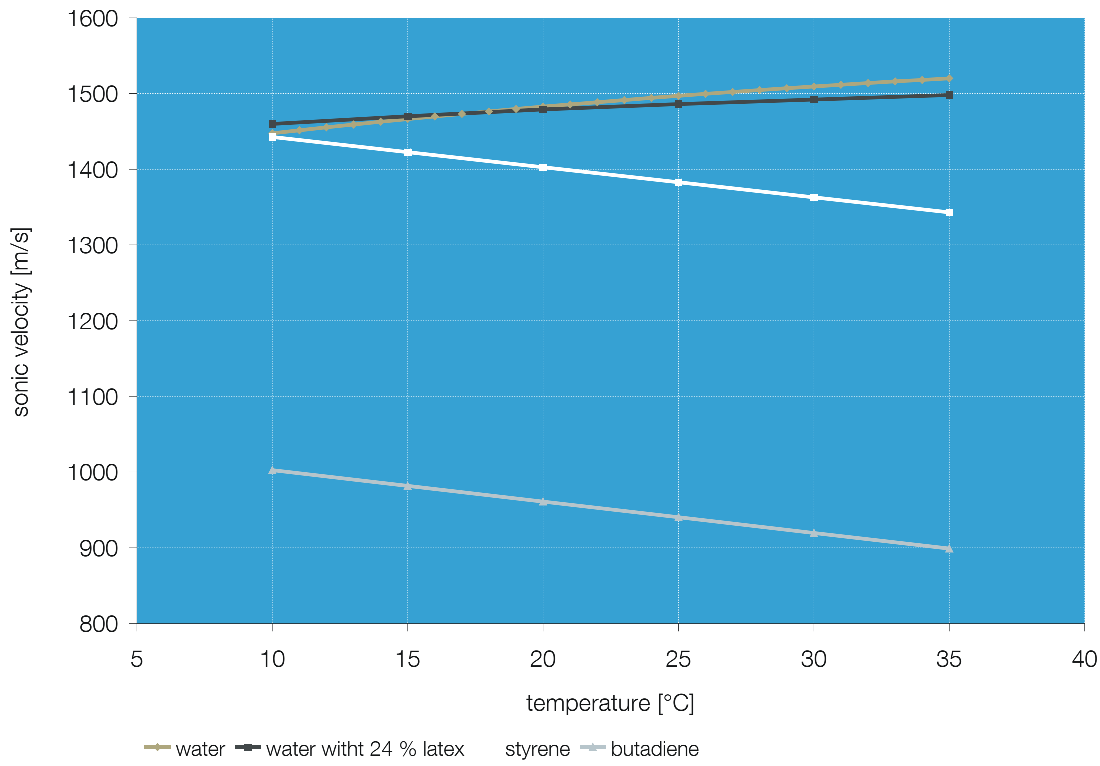
The following description explains this as an example for the emulsion polymerization of styrene butadiene latex. The determination of parameters like concentration, degree of polymerization etc. occurs analog in other polymerization types.

Emulsion polymerization of styrene-butadiene-latex

The individual components and lattices were examined for a butadiene-styrene emulsion polymerization reaction system.

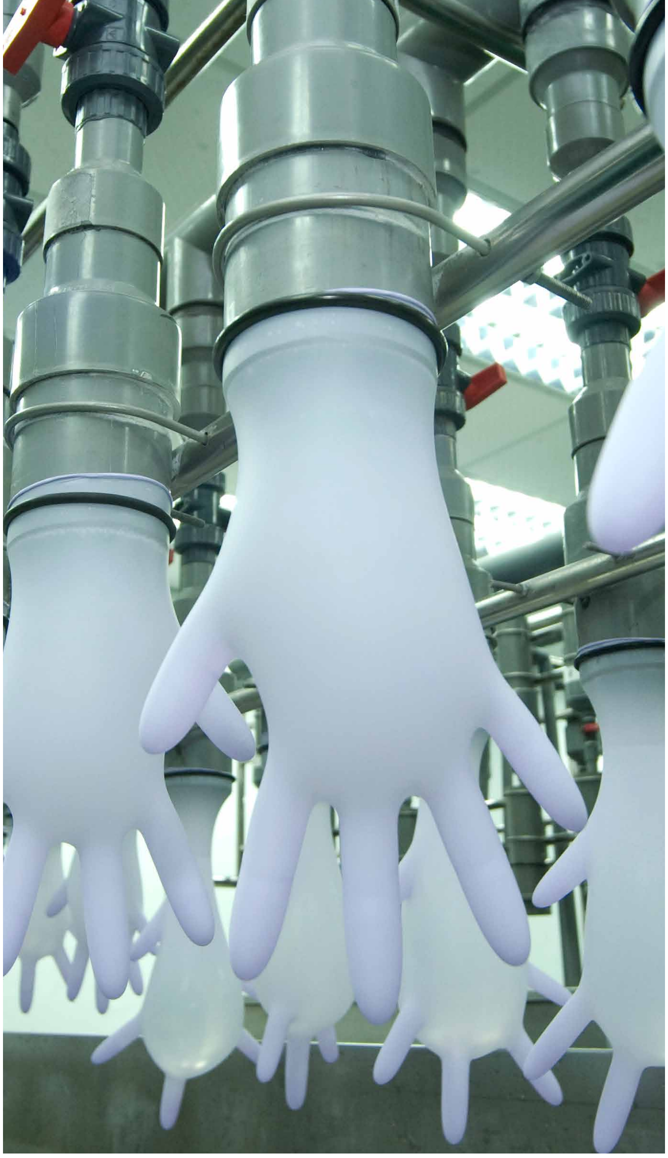
In the diagram below it can be seen, that the sonic velocity of the monomers clearly differs from that of the polymers.

Sonic velocity of components of butadiene-styrene polymerization

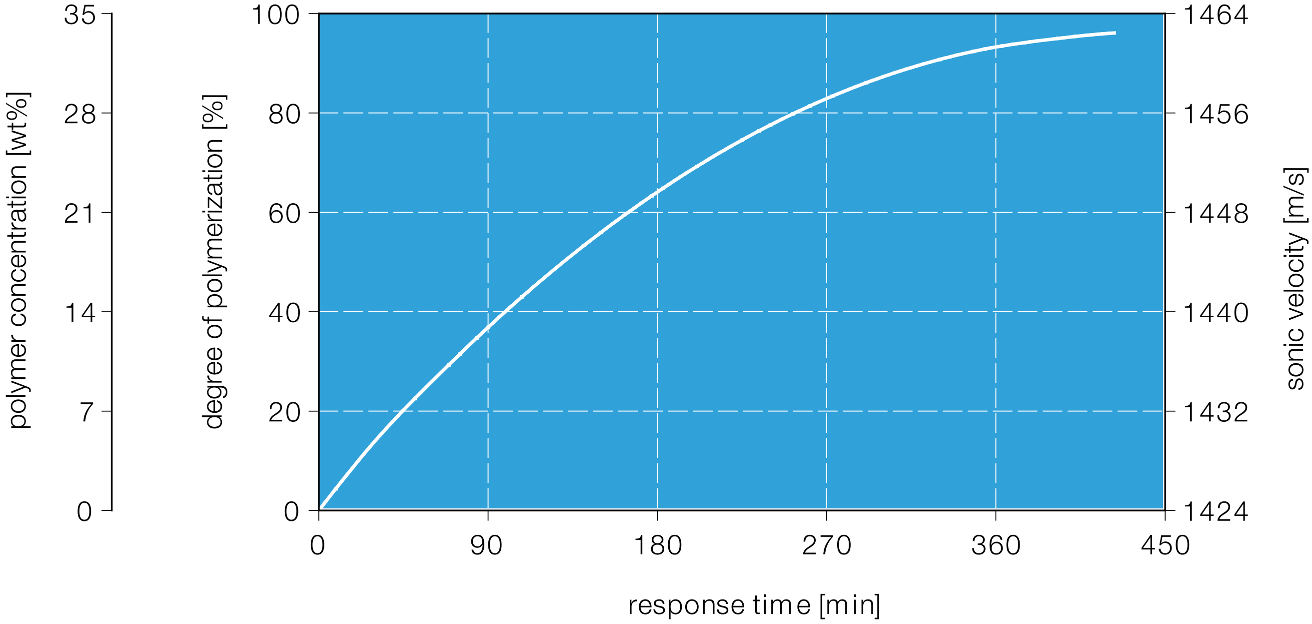


The sonic velocity and the concentration are linked to each other. Furthermore, the degree of polymerization reflecting the share of polymer in monomer correlates with the concentration. With ultrasonic measurement systems, it is possible to determine the concentration and the degree of polymerization. The following diagram describes this relationship during a polymerization of butadiene-styrene.

In case of emulsion polymerization of butadiene and styrene, the degree of polymerization can be determined with an accuracy of 0.1 %.



Variation of sonic velocity, concentration and degree of polymerization



Applications

Our knowledge in the field of polymerization acquired by numerous applications at the customer and by in-house technology is the result of varied experiences over 20 years. This knowledge is integrated into new projects by always handling customer data as confidential.

In polymerizations, not only polymers are the focus of monitoring, but also a number of monomers, additives and solvents as well as recovery processes.

SensoTech offers the following secondary literature to different production processes:

- optimization of polyamide production
- optimization of polyurethane production
- styrene-butadiene-latex (SBR) production safe and efficient

The following applications have been examined so far:

- caprolactam-polymerisation
- styrene-butadiene-latex
- phenol-formaldehyde resin
- polymethyl-meta-acrylate PMMA
- polyvinyl acetate PVA
- polyvinyl chloride PVC
- polyamide PA
- polyvinylidenchloride PVdC
- epoxy resin
- polystyrene PS
- polycarbonate PC
- polyester PE
- polyethylene
- formaldehyde urea resin
- elastane
- aldol in acetaldehyde
- polyurethane PU
- polysiloxane
- isoprene rubber IR
- methyl silicone resin
- slicone acrylate
- potassium methyl siliconate
- silicone resin
- polysulfide polymer
- paraphenylen-terephtalamide PPTA
- hindered Amine Light Stabilizers HALS
- methacrylamide MAA
- customer-specific compositions

The LiquiSonic® measuring device facilitates monitoring and control of various reactions, in particular in the batch process. Depending on the process and liquid, catalytic and enzymatic reactions, such as polymerization, crystallization as well as mixing processes can be optimized to ensure the quality of the final product.

In monomer and polymer systems in general, the differences of sonic velocity between monomer and polymer are determined primarily by chain length and the degree of branching and crosslinking.

Product	Sonic velocity
styrene	1354 m/s
polystyrene	2330 m/s
vinyl chloride	897 m/s
polyvinyl chloride	2260 m/s

The table shows clearly, that these differences in sonic velocity between monomer and polymer and also between start and end of polymerization reactions are very large.

The sonic velocity and concentration are directly linked to each other. Furthermore, the degree of polymerization determining the polymer content in the monomer correlates with the concentration. Using the LiquiSonic® measuring technology, it is possible to determine the concentration and the degree of polymerization.

Application example Caprolactam production

Polyamides are among the most common thermoplastic synthetic materials. Caprolactam (CPL) is the main raw material for these polymers, especially for PA6 (nylon 6). The monomer caprolactam is produced in a complex process, consisting of 4 main parts:

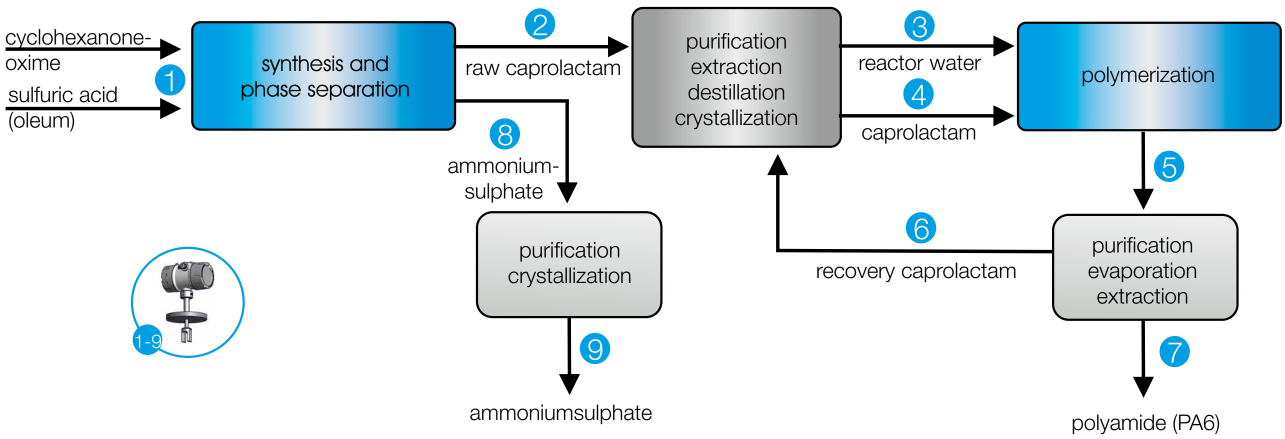
- synthesis of raw caprolactam
- separation and crystallization of ammoniumsulphate
- purification and preparation of raw caprolactam
- polymerisation of polycaprolactam (PA6)

The raw caprolactam production is based on the key intermediate cyclohexanone oxime, which is usually made by cyclohexanone, hydroxylamine and H_2SO_4. By adding of ammonia and oleum, the raw caprolactam is synthesized and separated of the ammonium sulphate phase. The following steps for the monomer caprolactam are the purification and concentrating (extraction, destillation and crystallization) and the polymerization step. Finally, residual monomers are separated from the polymer (recovery CPL).

The robust inline analyzer LiquiSonic® offers several advantages within quality monitoring and optimal process control at several measuring tasks:

- incoming goods control
- Beckmann rearrangement: H_2SO_4 / oleum
- phase separation of CPL and ammonium sulphate
- crystallization monitoring of ammonium sulphate
- extraction: concentration CPL in solvent
- destillation: concentration CPL in water
- reaktor water: water in caprolactam

LiquiSonic® is used for high-precision concentration value determination (incoming goods, caprolactam), phase detection / separation and process monitoring (crystallization). The internal limit value monitoring signals exceeding or falling below thresholds and transfers the real-time information via analog or digital outputs, serial interfaces, or fielbus (Profibus DP, Modbus) to the process control system.



Measuring point	Installation	Measuring task
1, 2, 3, 4, 5, 6, 7	feed and transport pipeline	concentration and quality control (incoming goods and caprolactam)
8, 9	transport pipeline	phase detection and separation
9	crystallization unit	monitoring and control of ammoniumsulphate crystallization

The LiquiSonic® analyzer provides a precise caprolactam concentration measurement with real-time monitoring. The devise is successfully implemented for phase separation between caprolactam and ammonium sulphate.

The robust sensor construction and the optional special materials, like HC 2000 oder PFA, promote long process life. In addition, SensoTech offers sensors approved by ATEX, IECEx and FM.

Trough precise LiquiSonic® monitoring, the concentration of residual CPL monomers can be reduced to a minimum, which increases the plant productivity.

The LiquiSonic® immersion sensor is easily installed into the feed and transport pipelines. Installing the LiquiSonic® sensor eliminates dead space and avoids high installation costs for bypasses.

By using the LiquiSonic® controller 30, up to four sensors can be connected, allowing the simultaneous monitoring of several measuring points.

Typical measuring ranges:

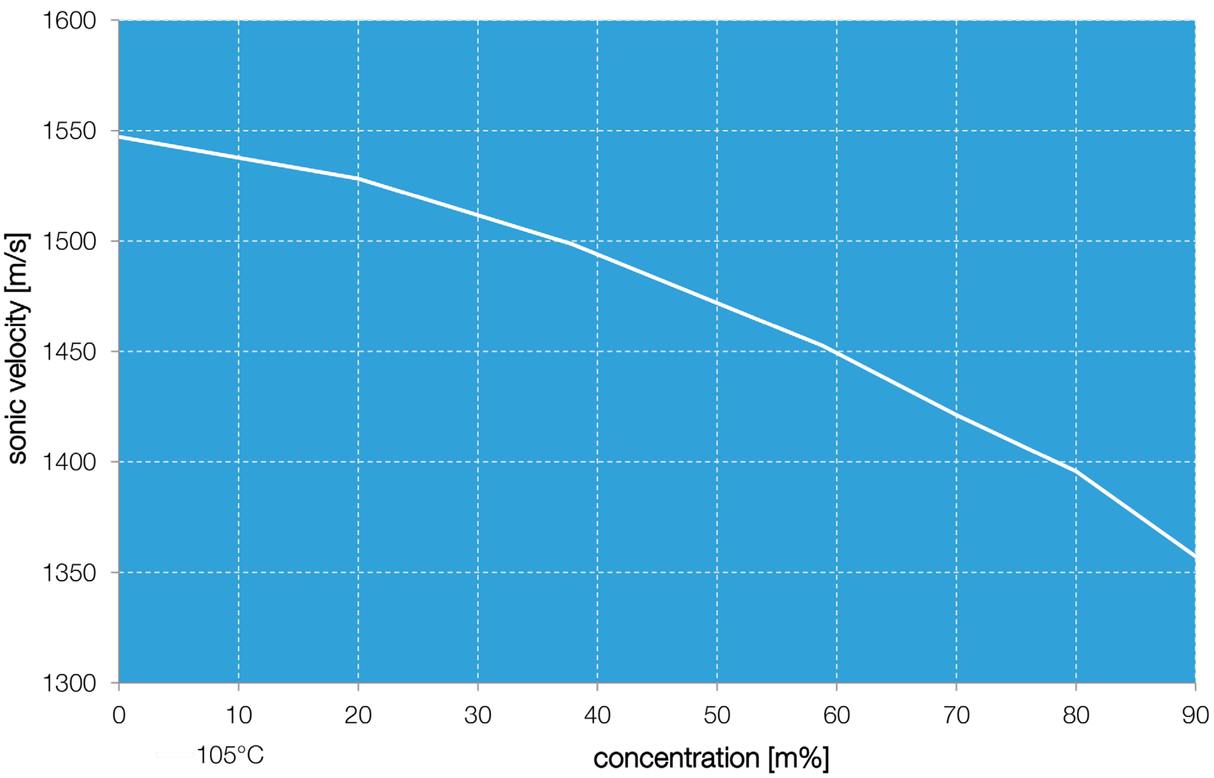
- concentration range caprolactam: 70 - 100 wt%
- temperature range: 80 - 130 °C (175 - 265 °F)

- concentration range caprolactam: 0 - 10 wt%
- temperature range: 20 - 70 °C (70 - 160 °F)

Incoming goods control:

- concentration range oleum : 0 - 30 wt%
- temperature range: 10 - 60 °C (50 - 140 °F)

LiquiSonic® sonic velocity measurement in caprolactam



Quality and Support

Enthusiasm for technical progress is the driving force behind our company as we seek to shape the market of tomorrow. As our customer you are at the center of all our efforts and we are committed to serving you with maximum efficiency.

We work closely with you to develop innovative solutions for your measurement challenges and individual system requirements. The growing complexity of application-specific requirements means it is essential to have an understanding of the relationships and interactions involved.

Creative research is another pillar of our company. The specialists in our research and development team provide valuable new ways to optimize product attributes, such as testing new types of sensor designs and materials or the sophisticated functionality of electronics, hardware and software components.

Our SensoTech quality management also only accepts the best production performance. We have been certified according to ISO 9001 since 1995. All device components pass various tests in different stages of production. The systems have all gone through an internal burn-in procedure. Our maxim: maximum functionality, resilience and safety.

This is only possible due to our employee`s efforts and quality awareness. Their expert knowledge and motivation form the basis of our success. Together we strive to reach a level of excellence that is second to none, with passion and conviction in our work.

Customer care is very important to us and is based on partnerships and trust built up over time.

As our systems are maintenance free, we can concentrate on providing a good service to you and support you with professional advice, in-house installation and customer training.

Within the concept stage we analyze the conditions of your situation on site and carry out test measurements where required. Our measuring systems are able to achieve high levels of precision and reliability even under the most difficult conditions. We remain at your service even after installation and can quickly respond to any queries thanks to remote access options adapted to your needs.

In the course of our international collaboration we have built up a globally networked team for our customers in order to provide advice and support in different countries.

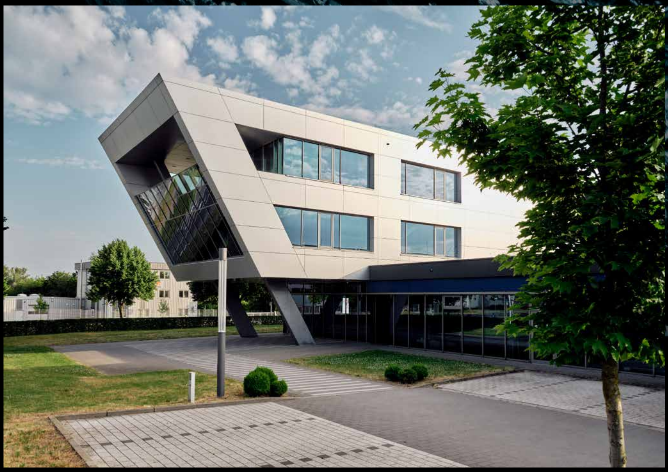
We value effective knowledge and qualification management. Our numerous international representatives in the important geographical markets of the world are able to refer to the expert knowledge within the company and constantly update their own knowledge by taking part in application and practice-oriented advanced training programs.

Customer proximity around the globe: an important element of our success worldwide, along with our broad industry experience.

In liquids, we set the measure.
With innovative sensor technology.
Tough, accurate, user-friendly.

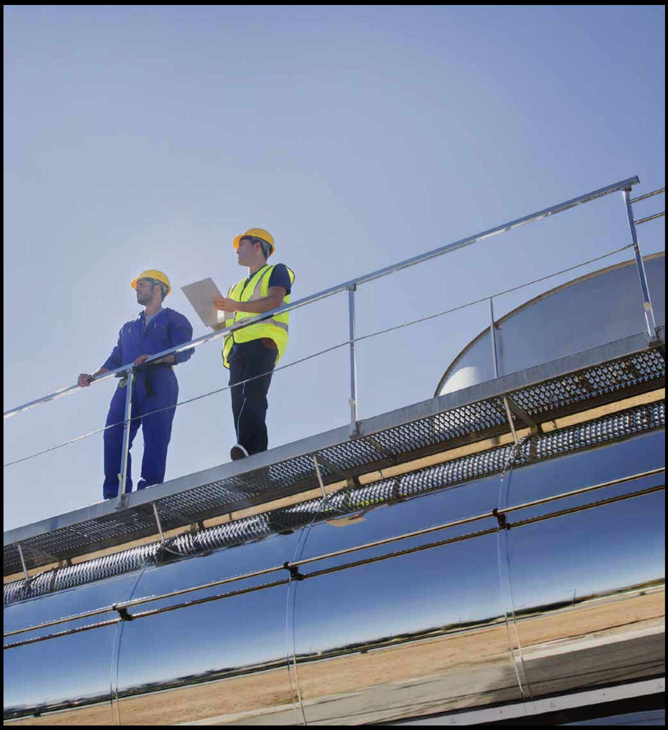
SensoTech

SensoTech is a provider of systems for the analysis and optimization of process liquids. Since our establishment in 1990, we have developed into a leading supplier of process analyzers for the inline measurement of liquid concentration and density. Our analytical systems set benchmarks that are used globally.



Manufactured in Germany, the main principle of our innovative systems is to measure ultrasonic velocity in continuous processes. We have perfected this method into an extremely precise and remarkably user-friendly sensor technology. Beyond the measurement of concentration and density, typical applications include phase interface detection or the monitoring of complex reactions such as polymerization and crystallization.

Our LiquiSonic® measuring and analysis systems ensure optimal product quality and maximum plant safety. Thanks to their enhancing of efficient use of resources they also help to reduce costs and are deployed in a wide variety of industries such as chemical and pharmaceutical, steel, food technology, machinery and plant engineering, car manufacturing and more.



It is our goal to ensure that you maximize the potential of your manufacturing facilities at all times. SensoTech systems provide highly accurate and repeatable measuring results even under difficult process conditions. Inline analysis eliminates safety-critical manual sampling, offering real-time input to your automated system. Multi-parameter adjustment with high-performance configuration tools helps you react quickly and easily to process fluctuations.

We provide excellent and proven technology to help improve your production processes, and we take a sophisticated and often novel approach to finding solutions. In your industry, for your applications – no matter how specific the requirements are. When it comes to process analysis, we set the standards.

SensoTech

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