



# Crystallization monitoring

Inline analytical technology for

- saturation degree
- supersaturation
- crystal content
- metastable range
- particle size

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 interruptions 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 crystallization

Sonic velocity measurement is used to determine crystallization parameters and to control crystallization processes. This measuring method enables the detection of the nucleation and saturation point and thus the metastable range.

During the crystallization, it is possible to measure the difference to the saturation (degree of saturation), the degree of supersaturation or the crystal content, using as a control variable for influencing the crystallization. When a solid substance is dissolved in a liquid, the liquid is absorptive up to a certain concentration. If further substance is added to the liquid, it will no longer be dissolved, the solution is saturated and the substance remains in its solid form.

This “maximum” concentration of a solution is called solubility or saturation concentration. The saturation concentration depends on the temperature. The temperature, at which the solution becomes saturated, is called saturation temperature. If the temperature is increased, more substance can be dissolved (except for negative solubility), and the saturation concentration becomes larger.

If the concentration is lower than the saturation concentration, the solution concerned is called unsaturated solution.

It applies at constant temperature:

$$S = \frac{c_{tot} - c_{sat}}{c_{tot}}$$

S = saturation

ctot = total concentration

csat = saturation concentration

If the temperature of an unsaturated solution is decreased, it can be cooled down for many solutions to a temperature which is lower than the saturation temperature without causing the solid substance to become crystallized. Then the solution is supersaturated. If it is cooled down further, spontaneous nucleus or crystal formation occur at a certain temperature called nucleation temperature. If the suspension is then reheated, the crystals become dissolved again. When reaching the saturation temperature, all crystals are dissolved. The saturation temperature is usually higher than the nucleation temperature.

The supersaturated range between the saturation temperature and the nucleation temperature is called metastable range.

By using LiquiSonic® systems in crystallization processes the following advantages result for the user:

- improved plant utilization by
  - continuous display of undersaturation and supersaturation
  - process control via the crystallization parameters
  - avoiding spontaneous nucleation
- energy saving by
  - fast achieving of required seeding point
  - continuous determination of crystal content
  - optimal approaching of the final process point
- saving of raw material by
  - precise setting of the required product quality
  - reproducible approaching of the seeding point





# Processes

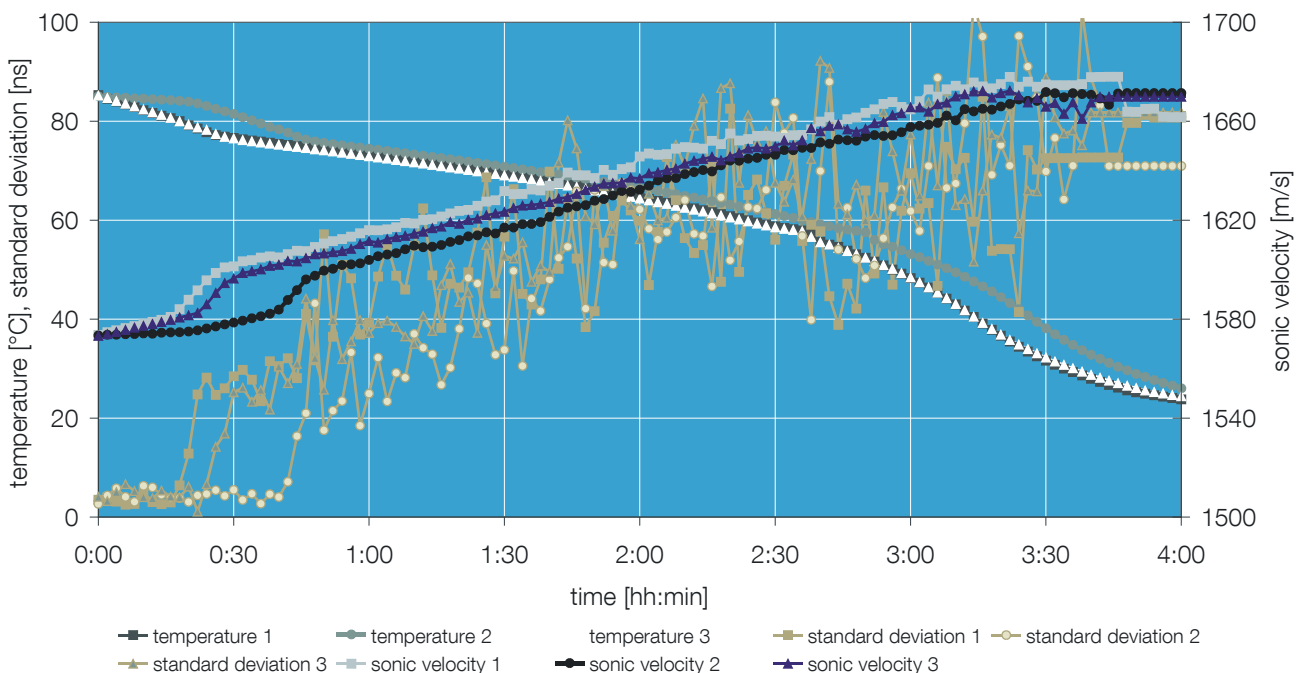
Crystallization processes in continuous as well as in batch processes can be monitored by measuring the sonic velocity with the LiquiSonic® system. In case of failures or deviations from the process flow, it can easily be reacted to achieve the required product quality.

The following diagram includes the evaluation of three different process flows concerning temperature, sonic velocity and standard deviation

In most cases, the characteristic process flow, which results into an optimal reaction course and thus to the required features of the end-product, is determined by a preliminary investigation. This ideal course can be implemented as so-called "fingerprint" of the process within LiquiSonic® 50.

By using typical analogue or digital interfaces, minor deviations from the ideal course are provided to the user or the process control, for example, to steer the crystallization via temperature control into the ideal course.

**Statistical evaluation of several sonic measurements per second**





# Applications

## Crystallization parameters

Sonic velocity and temperature are measured during cooling and heating the solution to establish the relevant parameters within the process. The sonic velocity presented as a function of the temperature, important crystallisation parameters, such as saturation temperature, nucleation temperature, and the position in the metastable range can be directly determined. The following diagram describes crystallization characteristics like ammonium sulphate with 42.6 w% during heating and cooling at different temperatures.

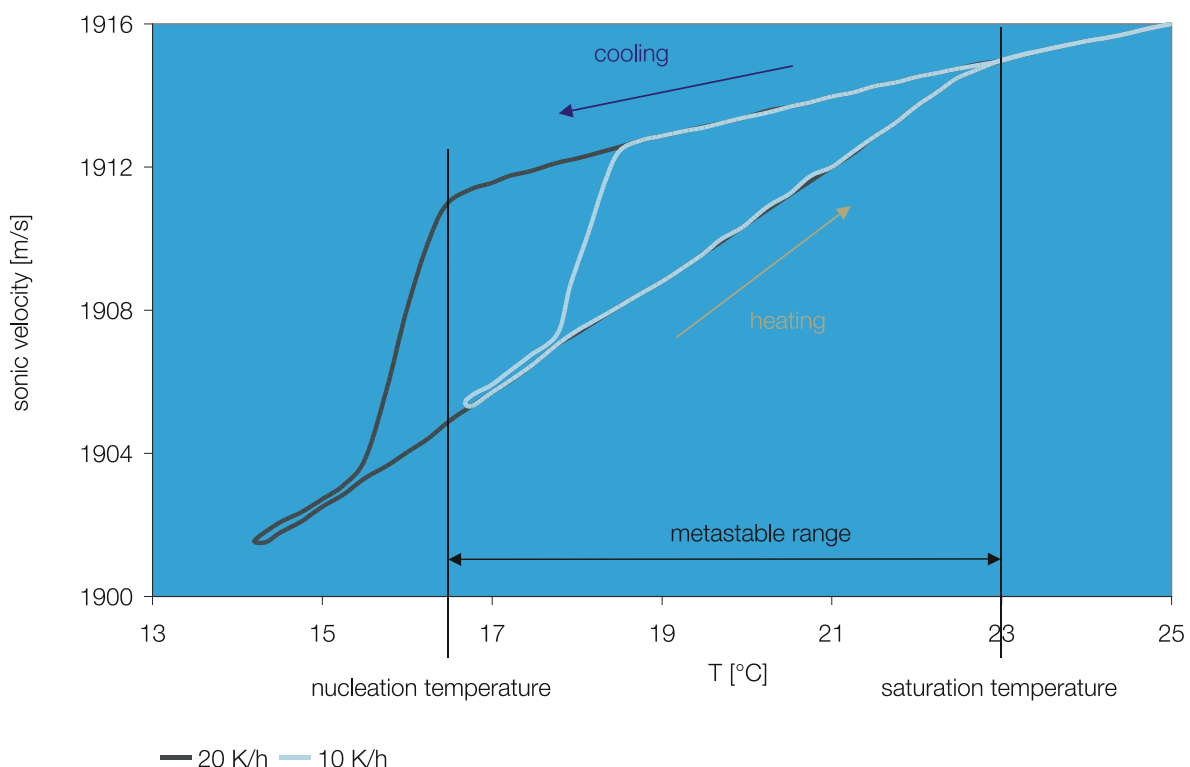
The diagram provides an explanation of this effect: if the solution is slowly cooled down, the sonic velocity changes at a specific temperature coefficient. From a certain temperature on a marked change of the sonic velocity can be

observed due to the crystal formation and the decrease of the supersaturation. The relating temperature is the nucleation temperature. If the solution is reheated, its sonic velocity curve differs from that obtained during cooling. Both curves meet again at saturation temperature.

Consequently, it is possible to determine the metastable range as well as the solubility curve via the sonic velocity. The metastable range depends on the chemical composition of the solution and on the cooling rate.

Using the sonic velocity as a function of temperature, you can determine the metastable range for any desired solution.

**Crystallization process in ammonium sulfate at a concentration of 42.6 wt%**



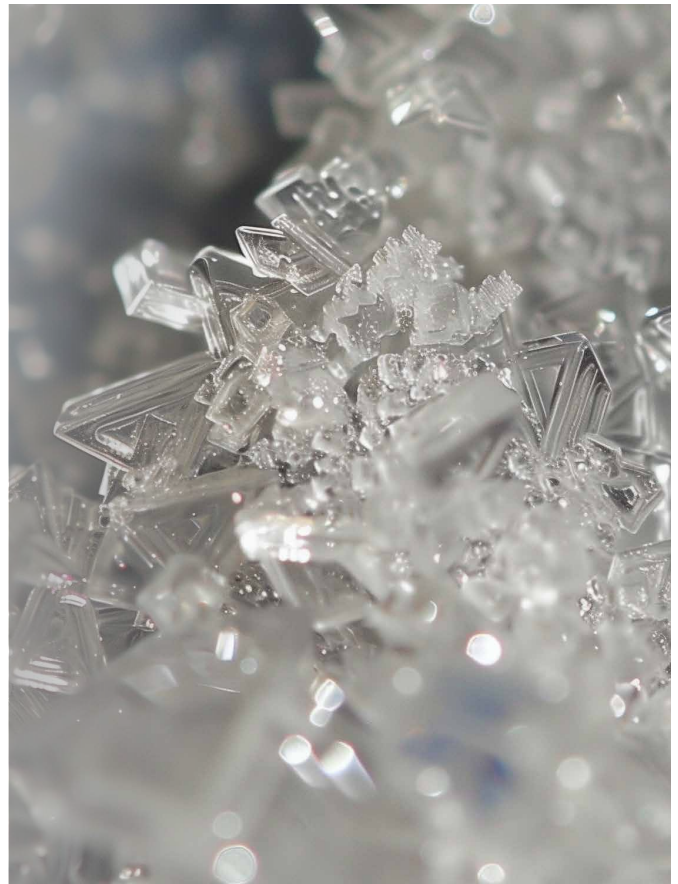
# Degree of saturation

Online measurement of the degree of saturation is based on saturation concentrations varying at different temperatures. The following diagram shows exemplarily the saturation behaviour of a large crystallization process.

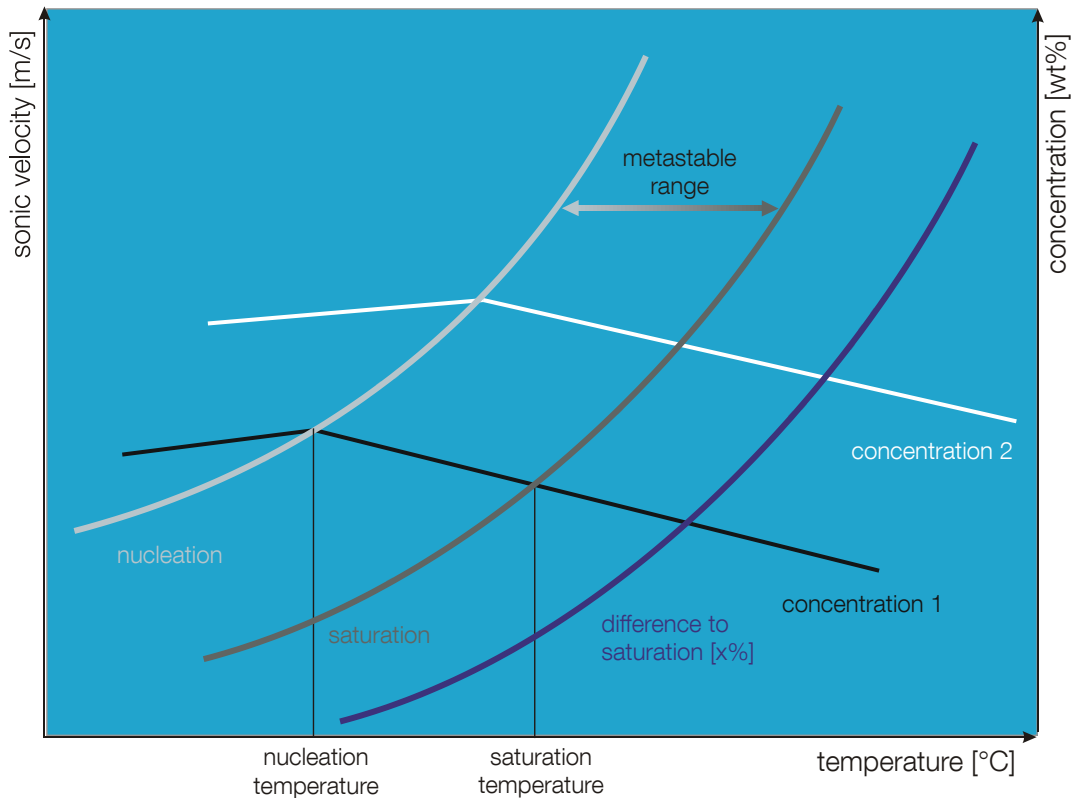
The current concentration is determined by sonic velocity and temperature measurement, and is provided as the saturation difference (degree of saturation) for downstream process control. Via the temperature this control variable serves to advance the process quickly to the stage of saturation to save time and energy. Therefore the process is individually controlled within concentration variations in the initial solution.

Then, a spontaneous nucleation occurs on the nucleation curve.

The range between saturation and nucleation is called the metastable (supersaturated) range. The supersaturation is the indicator for the perfect seeding point within the controlled nucleation.



## Saturation behaviour depending on the concentration, temperature and sonic velocity



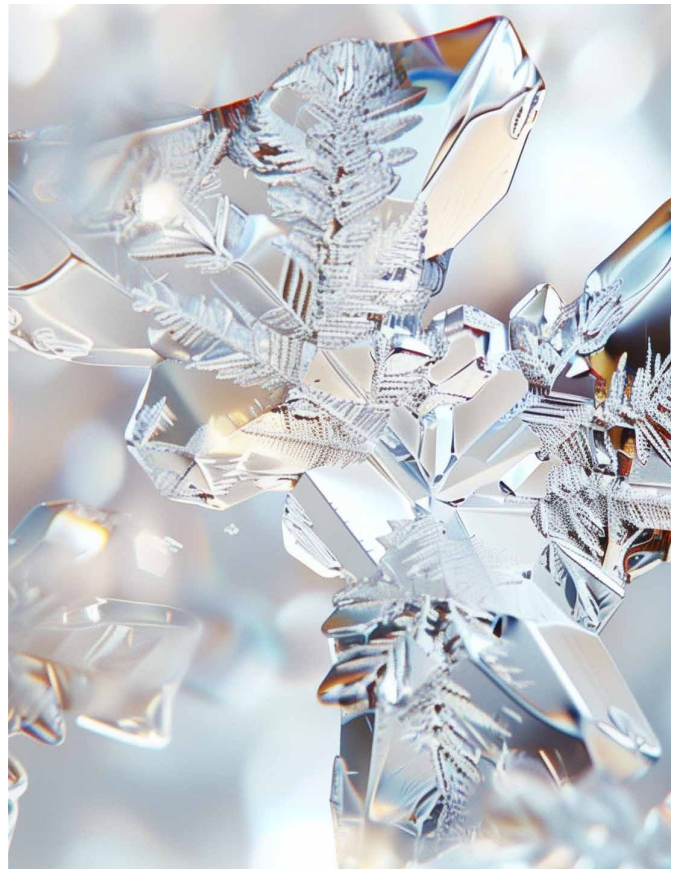
# Supersaturation

The degree of supersaturation can also be determined by sonic velocity, as a function of the temperature. As shown in the figure below the degree of supersaturation reflects a specific point within the metastable range. The closer this point is to the nucleation curve, the higher is the degree of supersaturation.

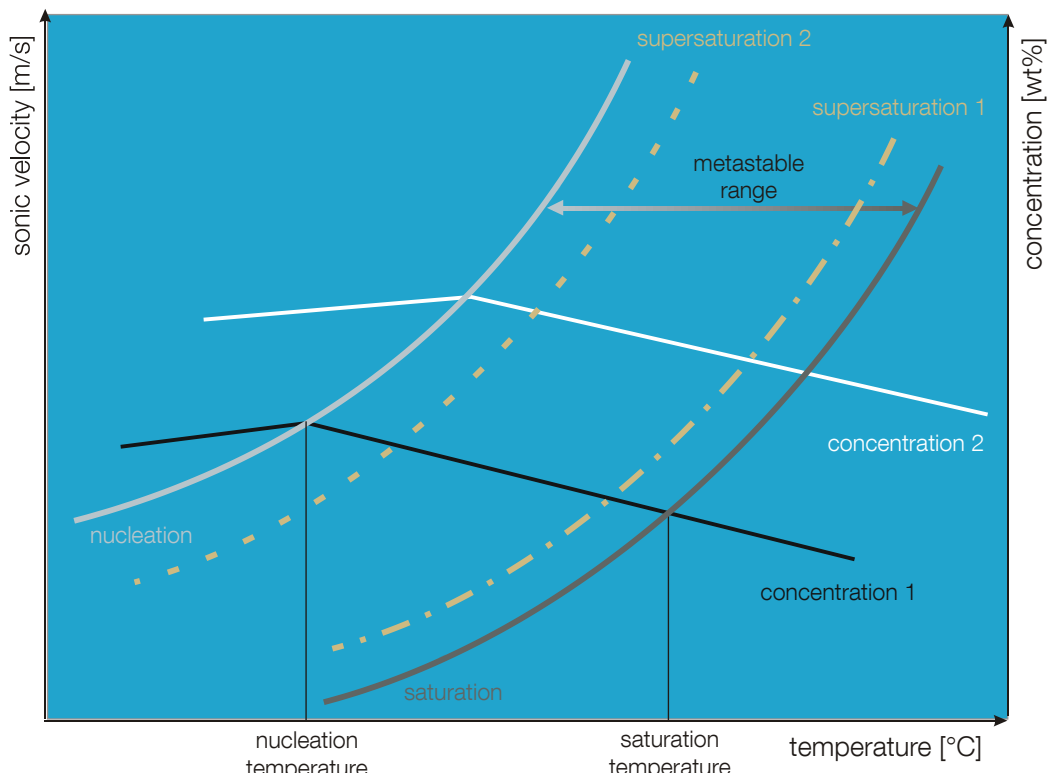
When the solution approaches the upper limit of the metastable range (supersaturation 2), the risk is high that it can result into spontaneous nucleation of a too fine end product. However, if the crystallization is too close to the saturation curve (supersaturation 1), then there will be less and large crystals only.

The supersaturation of the solution varies during crystallization due to crystal growth. Crystal growth reduces the degree of supersaturation. Supersaturation increases again when the temperature of the mother solution decreases or the solvent evaporates.

By measuring the sonic velocity and the temperature of the mother solution during crystallization, an optimization of the crystallization process control within the metastable range can be realized. This enables an direct influence on growth and so on the morphology of crystals.



## Supersaturation depending on the concentration, temperature and sonic velocity





# Supersaturation decreasing and crystal growth kinetics

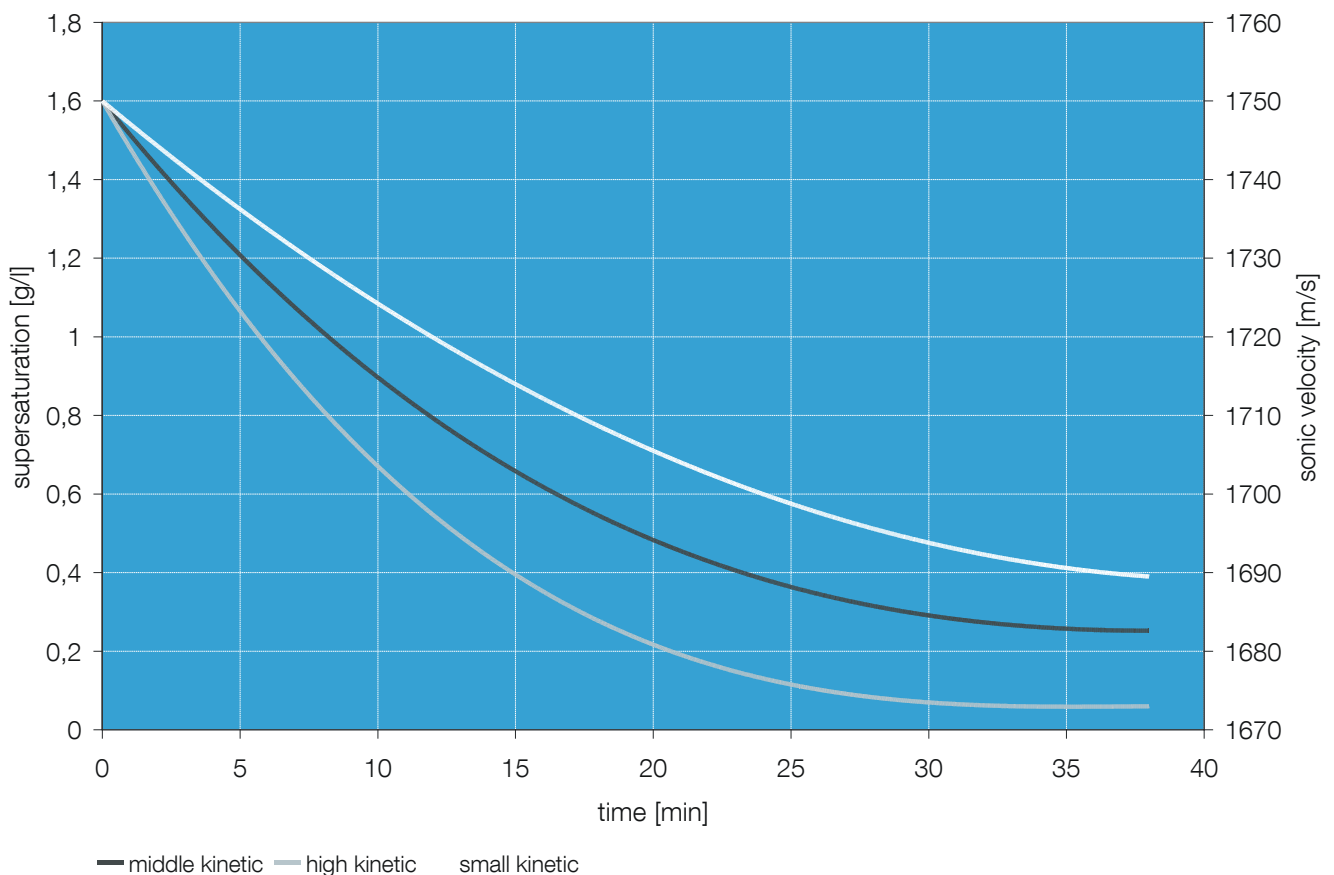
The degree to which supersaturation is decreasing during crystallization can be represented as a function of time (supersaturation decline curve). The following diagram shows different crystal growth kinetics which were detected by decreasing of sonic velocity as well as by supersaturation

As it can be seen, the time curve of the sonic velocity is identical as that of known supersaturation decline during crystallization. The figure shows the supersaturation decline curve calculated from sonic velocity which is compared with the chemical analysis suggested by Tavaré and Chivate.

The crystal growth kinetics can be determined from the supersaturation decline curve. This variable indicates how fast the crystals grow in the mother solution and is therefore an important variable for designing and dimensioning crystallizers.

It is possible to measure directly the supersaturation decline curve based on the correlation between supersaturation and the sonic velocity.

Supersaturation decreasing as a function of time

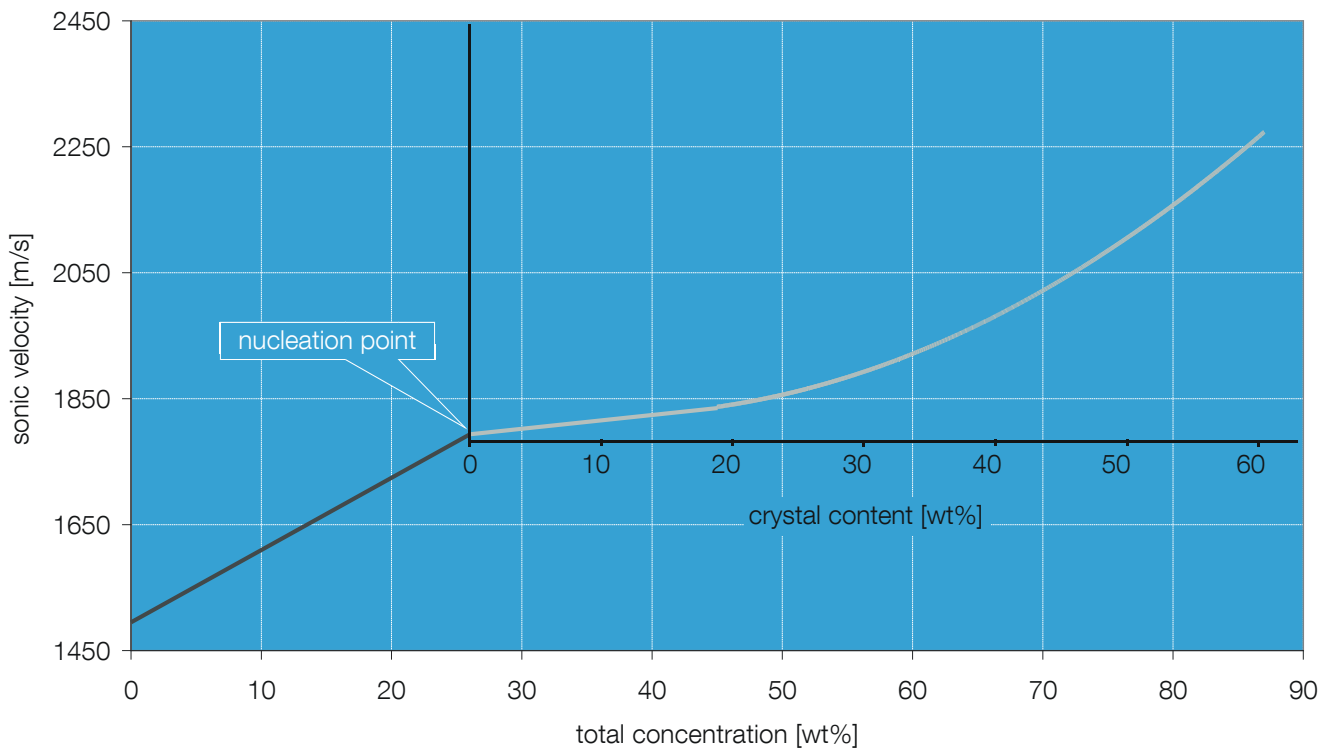


# Crystal content

Each suspension is characterised by a specific sonic velocity behaviour depending on temperature and concentration. The corresponding characteristic curves are also stored in the LiquiSonic® system. This enables the direct inline measurement of the solid concentration respectively crystal content or dry matter content.

It is possible to monitor and control the separation in continuous crystallization processes by determining the crystal content. In batch processes the end point of crystallization and the crystal growth can be determined and monitored.

**Sonic velocity depending on the concentration of NaCl in water at 25 °C**





In liquids, we set the measure.

# Accessories

There are several possibilities to install the LiquiSonic® analyzer appropriately and to facilitate the integration into the process control system. The following products have proved to be useful.

## Controller and field housing

The controller is designed for rack-mounted systems. It is alternatively available with a 19" housing 4 HU.

In order to be able to mount the controller into the field, two variants of field housings made of plastic or stainless steel can be delivered, which meet on-site conditions in an optimal way.

- material: anodized aluminum
- dimensions: 482.9 (19") x 177 (4 HU) mm
- application: rack-mounted system

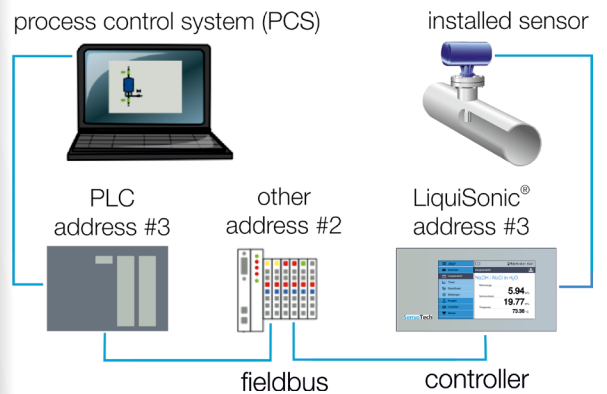


Controller housing 19" 4 HU

## Fieldbus

The fieldbus option provides the possibility to integrate the controller in a PCS or to automate the process flow via PLC. Beside the transfer of measuring values like concentration and temperature it is also possible to exchange parameters and control data (for example product switch).

The controller supports different fieldbus systems and follows the standards recommended by the respective standards organizations. Common variants are Modbus and Profibus DP.



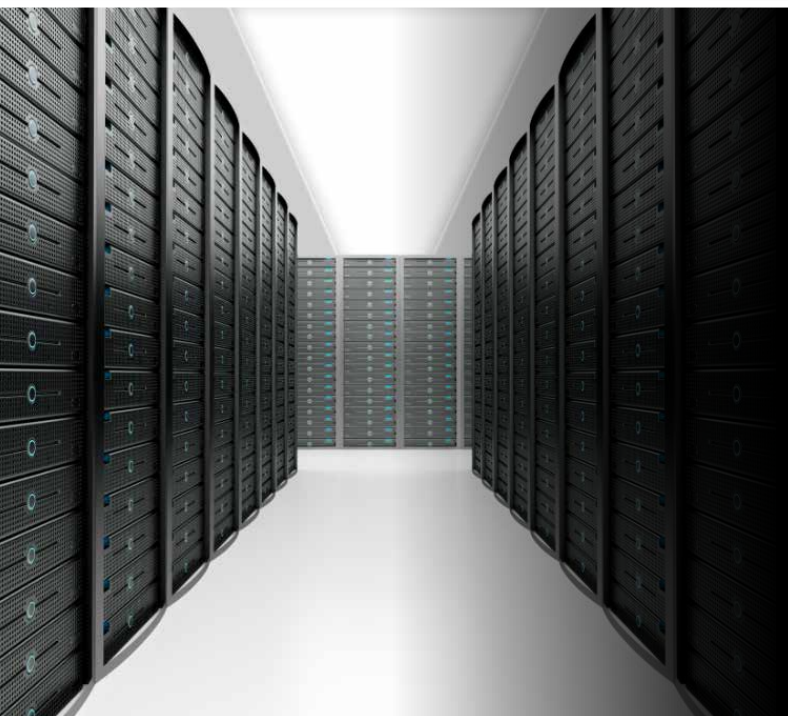
Connection interfaces

## 4G-industrial router

With a 4G-industrial router it is possible to operate the LiquiSonic® controller remotely. For this purpose, the controller is connected to the 4G-industrial router and appropriate IP address must be entered in the browser on the PC.

The remote connection includes the following features:

- uploading new product data sets on the controller
- reading out the controller data storage, e.g. to record product data for unknown liquids
- monitoring and configuration of the controller and sensors
- worldwide and fast customer support by Sensotech service



## Network integration

The LiquiSonic® controller has an Ethernet interface, that makes the integration into the corporate network possible. After entering the user name and password, the access to the stored logs is possible.

Integrating the controller into the network enables remote control, view of status information, transfer of product data sets or calibration of products.

The Network integration includes

- web server (HTTP),
- command line (TELNET),
- file transfer (FTP),
- time synchronisation (NTP),
- e-mail notification (SMTP).



# 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

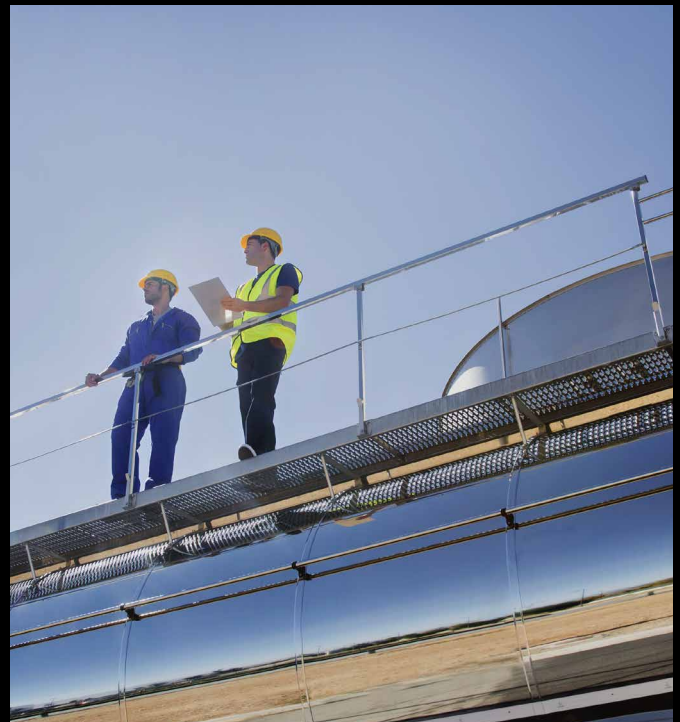
**SensoTech** is a provider of systems for the analysis and optimization of process liquids. Since our establishment in 1990, we have developed into a leading supplier of process analyzers for the inline measurement of liquid concentration and density. Our analytical systems set benchmarks that are used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





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