SensoTech

Crystallization monitoring

Inline analytical technology for:

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

Increasing c

With high

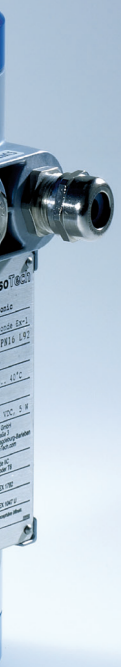
Robust, ac

LiquiSonic®

quality, **saving resources: LiquiSonic®.**

-value, **innovative sensor technology.**

curate, **user-friendly.**



LiquiSonic® is an inline analytical system, that detects the concentration in a liquid directly in the process and without any time lag. The device is based on the accurate measurement of sonic velocity and temperature and enables so the monitoring of processes and complex reactions.

Benefits for the user include:

- optimal plant control through online information about the state of the process
- maximum efficiency of processes
- increasing of the product quality
- reduction of costs for laboratory measurements
- saving of energy and material costs
- optimal process utilization
- reproducible process management with proprietary “fingerprint“ technology

Using the latest digital signal processing technology ensures a highly accurate and fail-safe measurement of the absolute sonic velocity and the concentration. In addition, integrated temperature sensors, a sophisticated sensor design and the know-how resulting from numerous series of measurements and many applications guarantee a high reliability of the system with a long lifetime.

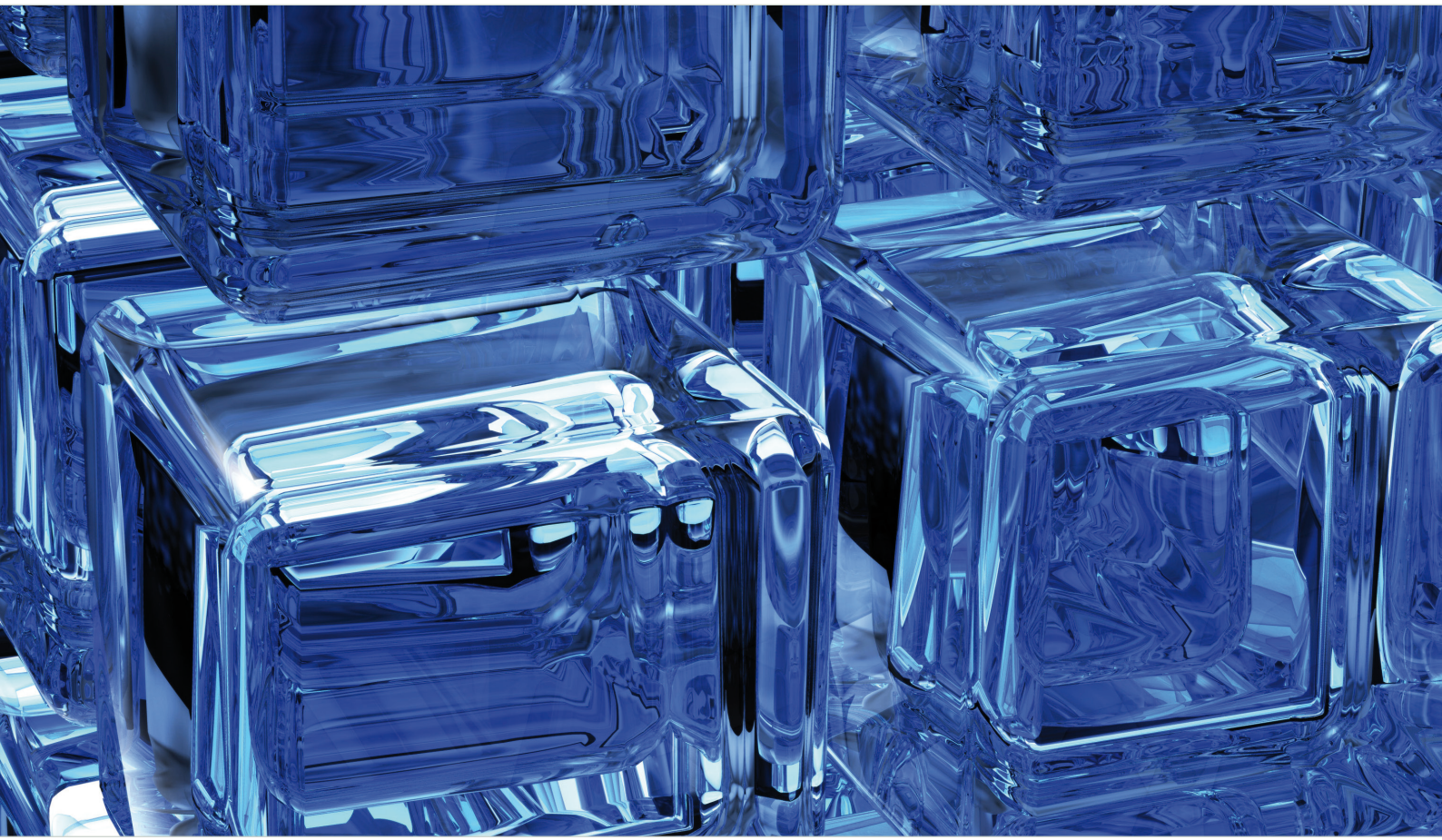
Advantages of the measuring method are:

- absolute sonic velocity as a well-defined and retraceable physical value
- independent of color, conductivity and transparency of the process liquid
- installation directly into pipelines as well as tanks and vessels
- robust and completely metallic sensor design without gaskets or moving parts
- maintenance-free
- corrosion resistance by using special material
- use at temperatures up to 200 °C
- high, drift-free measuring accuracy even with high concentrations of gas bubbles
- connection of up to four sensors per controller
- forwarding of measuring results through field-bus (Profibus DP, Modbus), analogue outputs, serially or Ethernet

Inline process analysis

Contents

1 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
c_{tot} = total concentration
c_{sat} = 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 tem-perature, 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

2 Processes

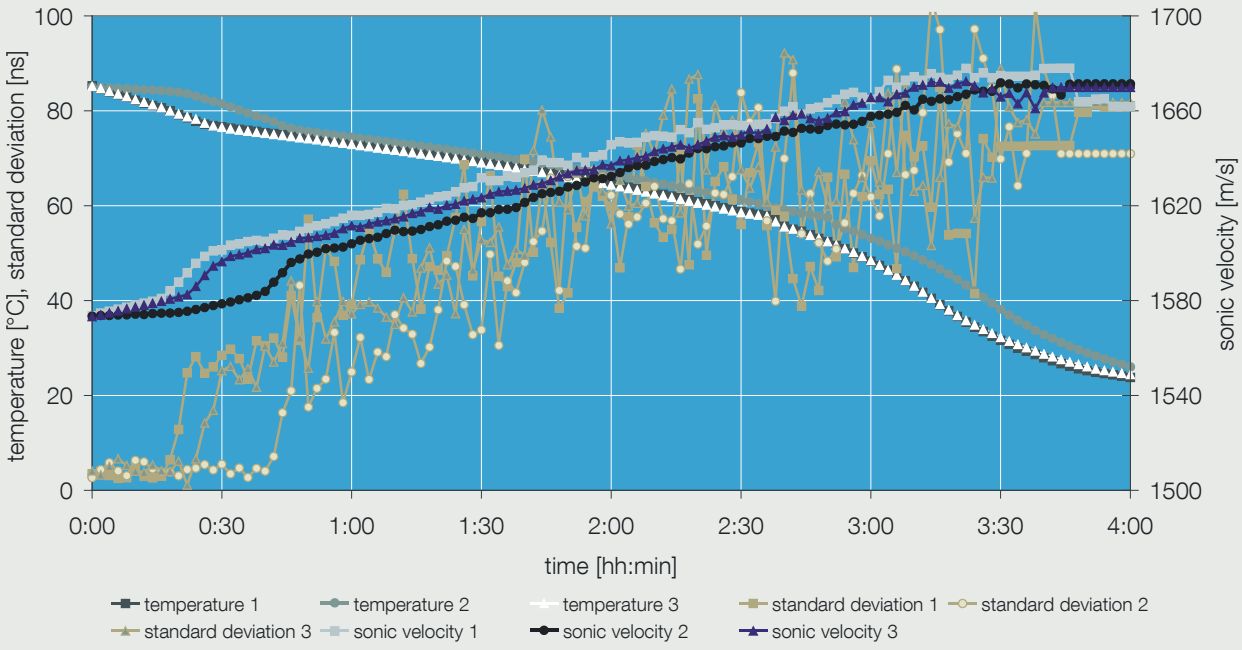
Crystallization processes in continuous as well as in batch processes can be monitored by measuring the sonic velocity with the LiquiSonic® system. In case of failures or deviations from the process flow, it can easily be reacted to achieve the required product quality.

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



3 Applications

3.1 Crystallization parameters

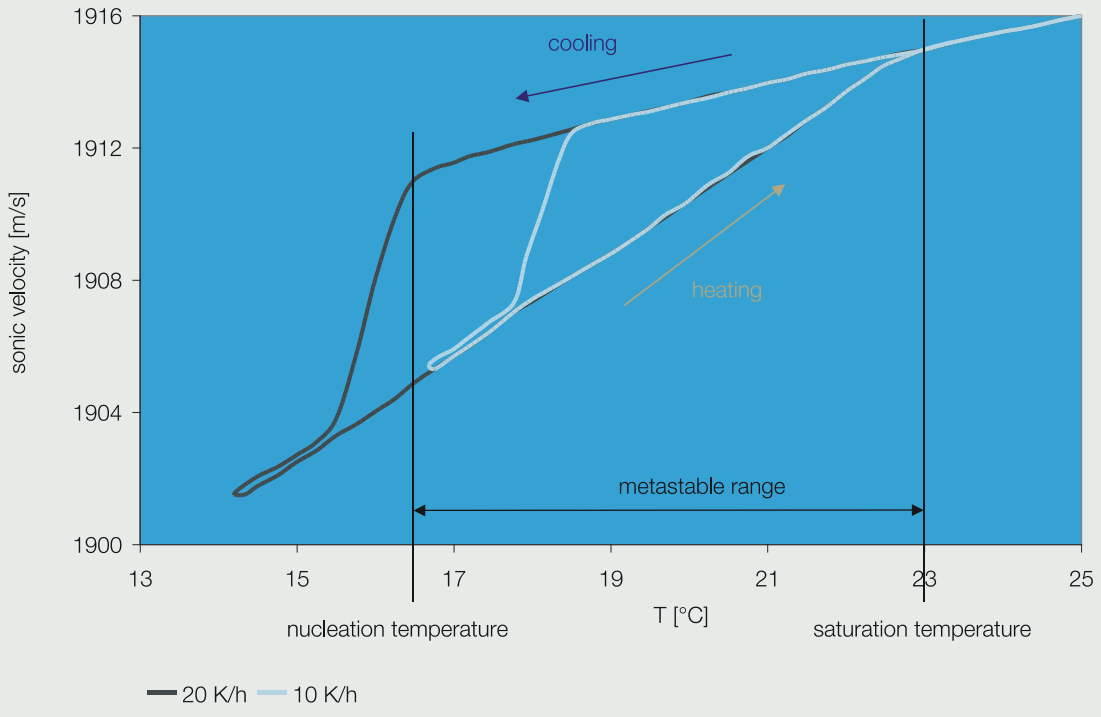
Sonic velocity and temperature are measured during cooling and heating the solution to establish the relevant parameters within the process. The sonic velocity presented as a function of the temperature, important crystallisation parameters, such as saturation temperature, nucleation temperature, and 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%



3.2 Degree of saturation

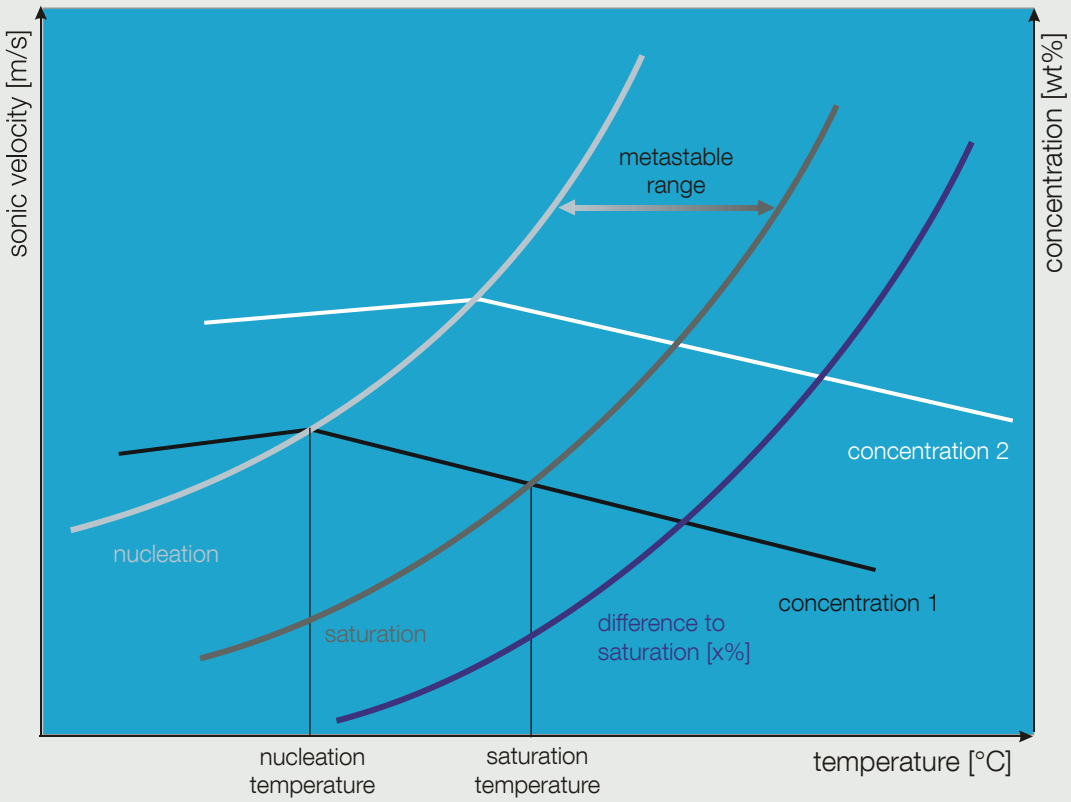
Online measurement of the degree of saturation is based on saturation concentrations varying at different temperatures.The following diagram shows exemplary the saturation behaviour of a large crystallization process stored in the LiquiSonic® 50 system.

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



3.3 Supersaturation

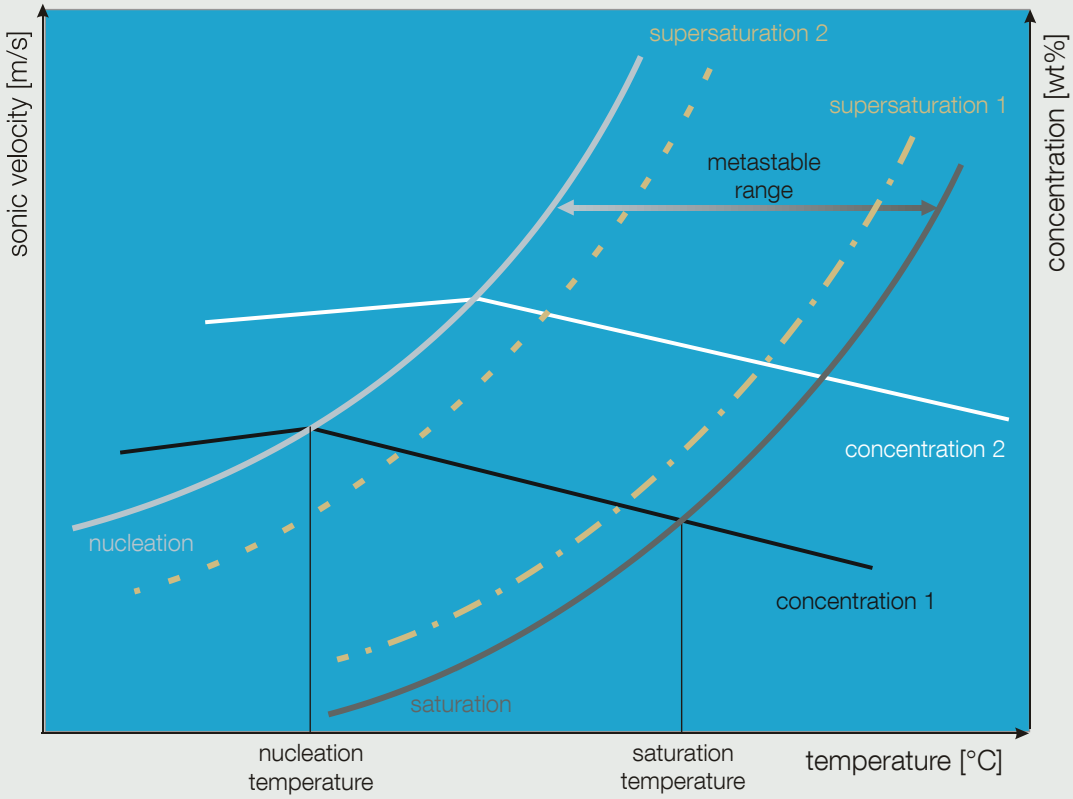
The degree of supersaturation can also be determined by sonic velocity, as a function of the temperature. As shown in the figure below the degree of supersaturation reflects a specific point within the metastable range. The closer this point is to the nucleation curve, the higher is the degree of supersaturation.

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



3.4 Supersaturation decreasing and crystal growth kinetics

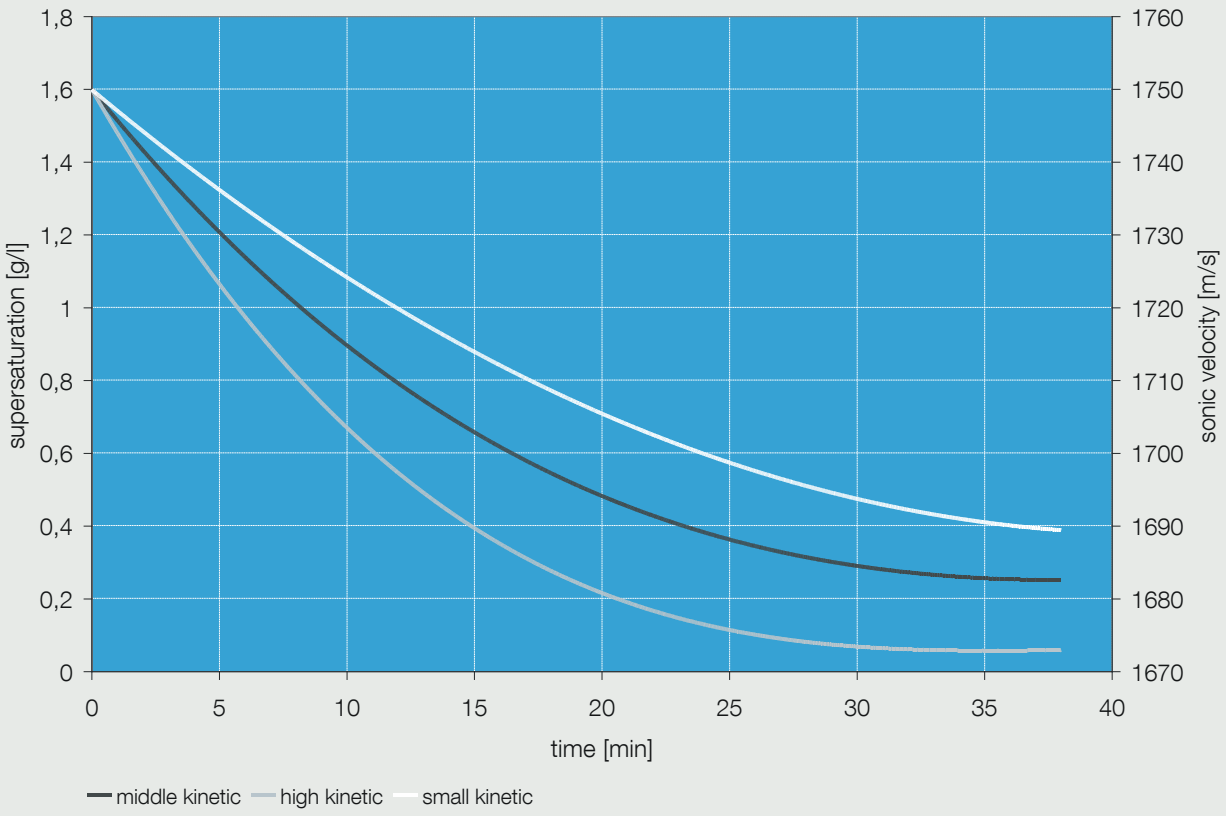
The degree to which supersaturation is decreasing during crystallization can be represented as a function of time (supersaturation decline curve). The following diagram shows different crystal growth kinetics which were detected by decreasing of sonic velocity as well as by supersaturation

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 Tavare 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

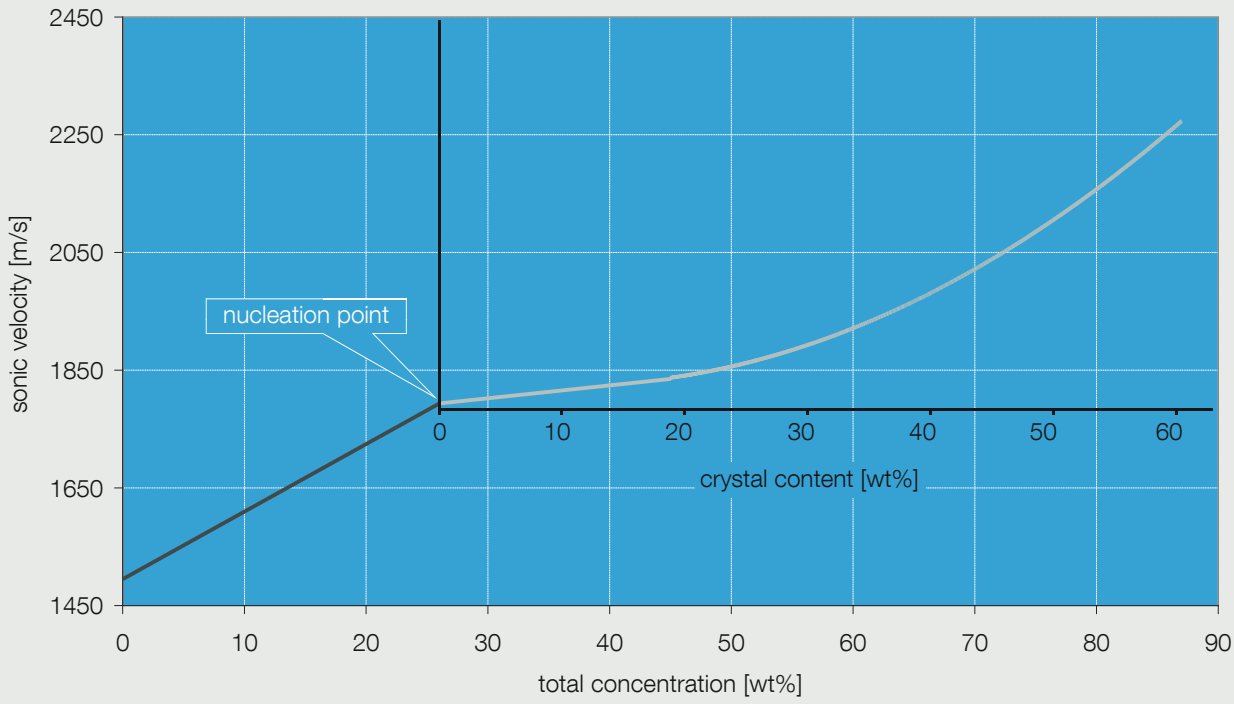


3.5 Crystal content

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



4 LiquiSonic® 50

LiquiSonic® 50 can be configured for reaction monitoring (fingerprint) or for crystallization monitoring (crystallization).

The controller processes the sensor data and is the interface to the operator by displaying the concentrationvalues. The controller is equipped with a high sophisticated microprocessor, even handles complex concentration calculations.

The large display with high resolution color TFT ensures an optimal view under any light conditions. Operator can define his own identifiers, process tags or physical units.

The clear, user-friendly and multilingual menu structure allows operating the system without extensive reading of the complex user's manual.

The internal data memory has a capacity of up to 15,000 data sets. By reading out via an integrated TCP/IP or RS232 interface it is possible to create own process reports in an easy way. The process data or related values will be refreshed every second. They can be transmitted via several adjustable analog or relay outputs as well as via different fieldbus interfaces.

Advantages for users are:

- storage of complex and dynamic characteristics including process belts and limits
- output of concentration, temperature and further important measured values
- simple parameterization e.g. of periphery
- interfaces like analog and digital outputs, fieldbus, or Ethernet
- degree of protection for device front IP65
- automatic self-monitoring
- comfortable handling due to tree structure within system menu
- recording of events e.g. exceeding the limit value
- memory of up to 256 products
- memory of process values for many days or years
- user management with authorization steps

4.1 Crystallization

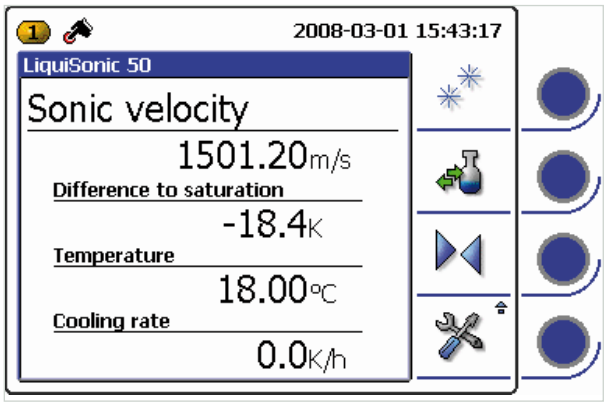
LiquiSonic® 50 detects and evaluates all relevant process parameters and is thus an efficient tool for characterization, visualization and control of crystallization processes of all types.
For example, the saturation and the nucleation line as well as the metastable range in the system can be implemented.

The state of the real-time crystallization process is submitted to the user. This can be the degree of the supersaturation, for example, which is relevant for the optimal seeding point.

The figures below explain the visualization function of the controller as an example to follow a crystallization process.

The main view of the controller displays up to four parameters. This includes:

- concentration of dataset,
- difference to the reference line, e.g. saturation line,
- current medium temperature,
- cooling rate.



Exemplary controller display

The controller contains also a chart view in which the timing of the concentration with the temperature is displayed for controlling a crystallization process.

The distance between the saturation line and the nucleation line is evaluated and visualized during the crystallization monitoring. The result is the distance to the reference line which is used as control variable for the process.

In case of the below shown chart view, the crystal grows currently at 4.1 °C supersaturation.

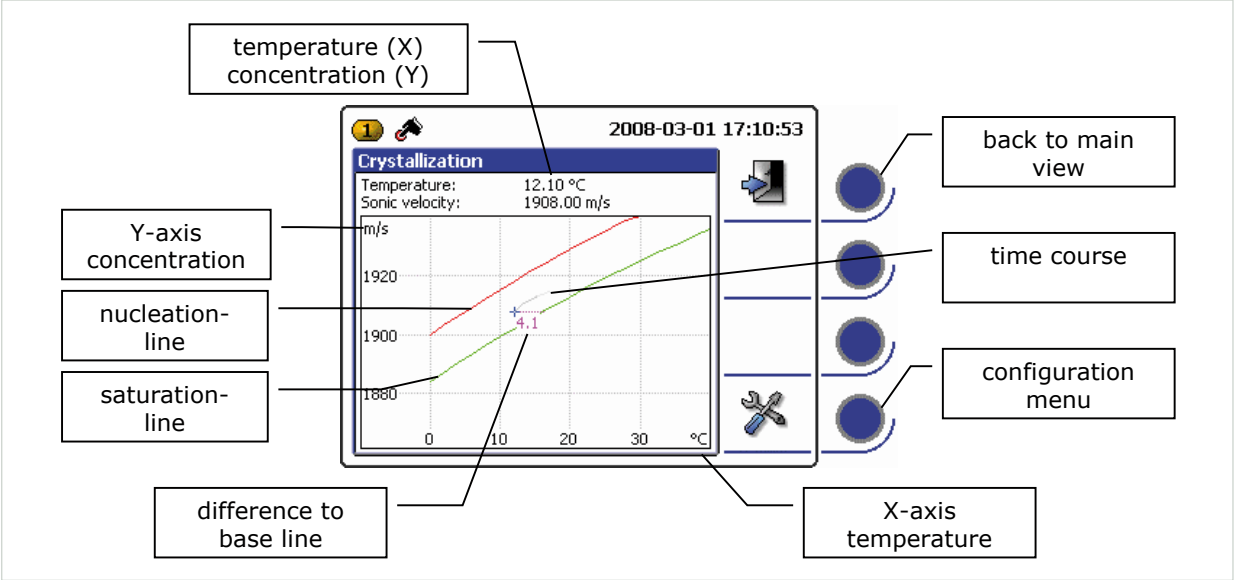


Chart view with current position of the process state

4.2 Fingerprint

In most cases, different process parameters, which lead to an optimal course of reaction and endproduct, were determined by preliminary investigations. This so-called “Golden Batch” can be implemented in the controller flexibly.

Diverse analog, digital outputs as well as BUS connections to the control system provide the user with little deviations from the ideal process. So, the user has the opportunity to intervene, for example to pass the reaction into an ideal process by adding catalysts.

5 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 centre 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.

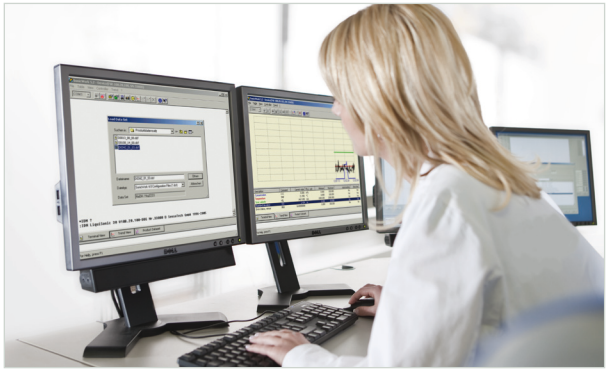
Creative research is another pillar of our company. The specialists in our research and development team provide valuable new ways to optimise product attributes, such as testing new types of sensor designs and materials or the sophisticated functionality of electronics, hardware and software components.

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 a 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 analyse 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 programmes.

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


Setting the sta
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SensoTech
LiquiSonic 30
Systemtest H2O
0.00 m/s
0.00 °C
SensoTech
Always exchange card while device is powered off!
Compact-Flash
Field bus
Ethernet
LNK
ACT

ndards **for process analysis.**

n, that **creates new solutions.**

osolute **spirit of development.**

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 the concentration and density of liquids. Our analytical systems set benchmarks that are used globally.

Manufactured in Germany, the main principle of our innovative systems is to measure ultrasonic velocity and density in continuous processes. We have perfected this method into an extremely precise and remarkably user-friendly sensor technology. As well as 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® measurement and analysis systems ensure optimal product quality and maximum plant safety. Thanks to their 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 reproducible measuring results even under difficult process conditions. Inline analysis eliminates safety-critical manual sampling and is immediately available for your automation system. All parameters can also be adjusted with high-performance configuration tools, so that you can 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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In liquids, we set the measure.

LSM050_01_14_R 02/2014