



Pharmaceutical Industry

Inline Analyzer for: · Phase Detection

- Concentration Solvents
 - · Suspensions
 - · 3-Component mixtures
- Crystallization Monitoring
- Incoming Goods Control

saving resources: LiquiSonic®.

innovative sensor technology.

user-friendly.

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-lived system.

Advantages of the measuring method are:

- absolute sonic velocity as a well-defined and retraceable physical quantity
- independence 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

1 Applications



1.1 Application Fields

The pharmaceutical industry has high quality demands regarding the production process. Inline process monitoring is therefore a key factor.

In numerous pharmaceutical applications SensoTech provides reliable solutions for the monitoring of process liquids.

Additionally, the measuring systems are optimally suited to monitor phase separation and crystallization processes - continuously and in real time.

This enables efficient process control and increased process safety. Even in aggressive process liquids, LiquiSonic® systems are robust and maintenance-free.

Process step	Application
Incoming goods department	Product identification for solvent, acids, alkalis, etc. · water, isopropyl alcohol, hexane, potash Check of the incoming goods quality and concentration · water content in sulfuric acid Avoidance of incorrect fillings and blending with highly reactive substances · hydrogen peroxide, isopropyl alcohol, monochloroacetic acid
Concentration Measurement	Quality control in a matter of seconds · diverse buffer solutions, batches Water content in solvents · methanol, ethanol, butanol Distillation of substances and products · lactulose in water Control of the raw material concentration · caustic soda Blending of liquids · isopropyl alcohol and water Dissolving station for solids · citric acid
Phase Detection	Detection of phase position in tanks · solvent / product phase Separation of oily and aqueous phases · ethanol-based herbal extracts
Solvent Recovery	Concentration control of the reactant and the distillate · methanol / tetrahydrofuran in water · acetonitrile / Methyl-tert-butylether in water
Crystallization	Process monitoring and control · API (active pharmaceutical ingredients) in organic solvents Target crystal seeding · with seed crystals Characterization of new substances · R&D with LiquiSonic® Lab Determination of the metastable area, saturation and nucleation process · for optimization of production processes (kinetics) Crystal content · salicylic acid in ethanol
Suspension Measurement	Precipitation of substances · ibuprofen / ethanol in water Measuring in mother liquors and suspensions · magnetic particle in salt water

1.2 Concentration Measurement

LiquiSonic® sensors provide clear and temperature compensated concentration values. Compared to other measuring methods, the principle of sonic velocity is independent from color, transparency and conductivity of the liquid.

The following tasks are successfully controlled:

- Monitoring and detection of incoming goods
- Quality monitoring of intermediate products
- Control and Monitoring (dilution, concentrate, leakage)
- Quality monitoring of the final product

Directly installed in the pipe, LiquiSonic® systems provide an excellent process monitoring - highly precise and maintenance-free. Rapid interventions in the process prevent from incorrect batches and safety risks.

1.2.1 Example: Solvent Recovery and Treatment

In the production of pharmaceuticals, solvents are used as carrier component, which can be diluted or contaminated by water over time.

Subsequently, this water contamination need to be separated from the solution just like other impurities.

Most organic solvents are recycled in distillation columns. The recovered solvent is used for downstream processes. In this way resources are saved, waste is avoided and new solvent purchases are minimized.

The LiquiSonic® analyzer determines the concentration of raw material as well as of recycled product – reliably, precisely and maintenance-free. As a result, the process is accurately controlled.

Benefits at a glance:

- Plant automation
- Higher distillation efficiency
- Optimal quality of the final product
- Increased plant capacity
- Savings of resources
- Sustainable usage of material



1.2.2 3-Component Measurement

The measuring system LiquiSonic® 40 enables the concentration measurement in 3-component mixtures. A 3-component mixture usually means a liquid of two substances in a carrier fluid or solvent.

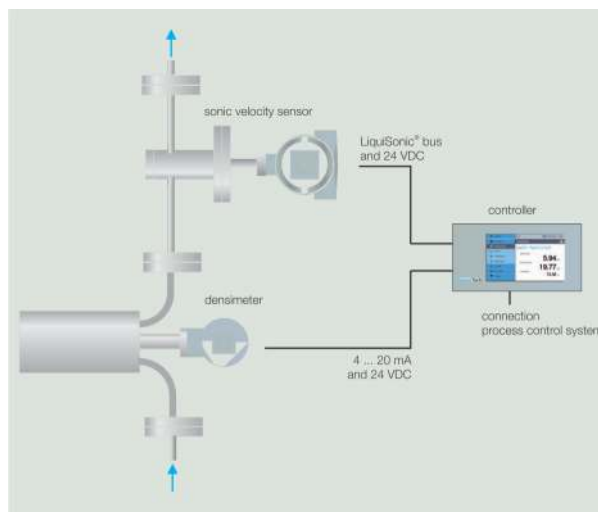
The measuring principle is based on the fact that the concentration changing of individual components of a liquid have different effects on certain physical values. For example, these physical values include sonic velocity, density, pH, refractive index, or other known variables.

If in a carrier liquid, the concentration of two components changes, two physical values are necessary to determine the concentration.

If the relationship between changing in concentration and changing of physical values is unambiguous, the concentration of each single component can be determined from a known change of the physical value according to an analytical mathematic relation description.

Usually the relationship can be graphically illustrated. The graph below shows an example of sonic velocity and density at different concentrations of each component and at a constant temperature:

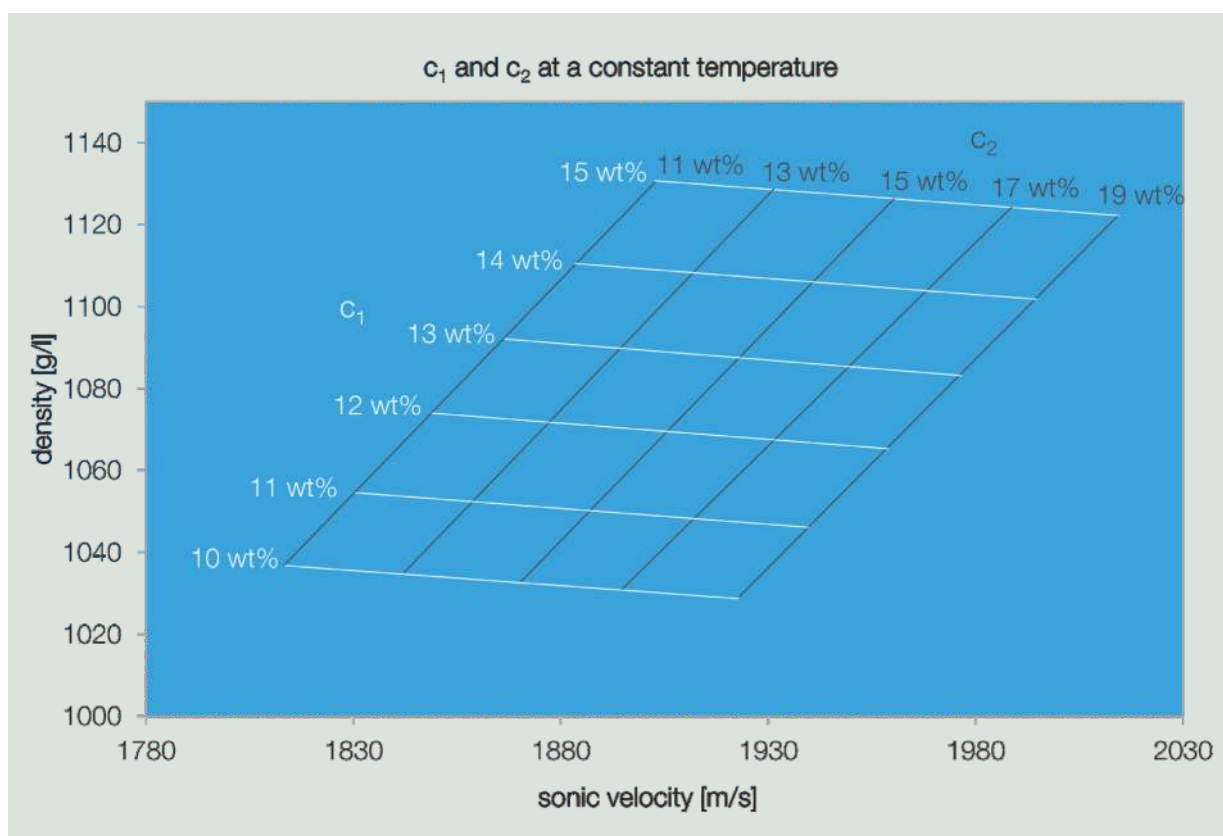
concentration 1: $c_1 = 10\% \dots 15\%$
concentration 2: $c_2 = 11\% \dots 19\%$



LiquiSonic® in combination with density

As a calculation model, these relationships are stored in the LiquiSonic® controller 40. So from the relevant physical value the controller calculates and displays the concentrations of the single components. Hence, due to the parallel analysis of two physical values, two concentrations simultaneously can be determined at a varying temperature.

The process temperature is also recorded and taken into the calculation model. Therefore, the concentration values of the components are always temperature compensated.



1.3 Incoming Goods Control

For an optimal production result, a high incoming good quality is indispensable. This begins with the monitoring of the delivered process liquids. An early detection of deviations or defective products leads to rapid reactions and thus to resource-saving production.

In addition, delivered substances can clearly be identified and separated by product specific values of the sonic velocity.

With the LiquiSonic® analyzer simple and fast monitoring of the delivered products are possible directly in the incoming goods department. As in the process, the liquid is sampled directly at the delivery point. In this way, confusions and product quality fluctuations within different batches are detected immediately.

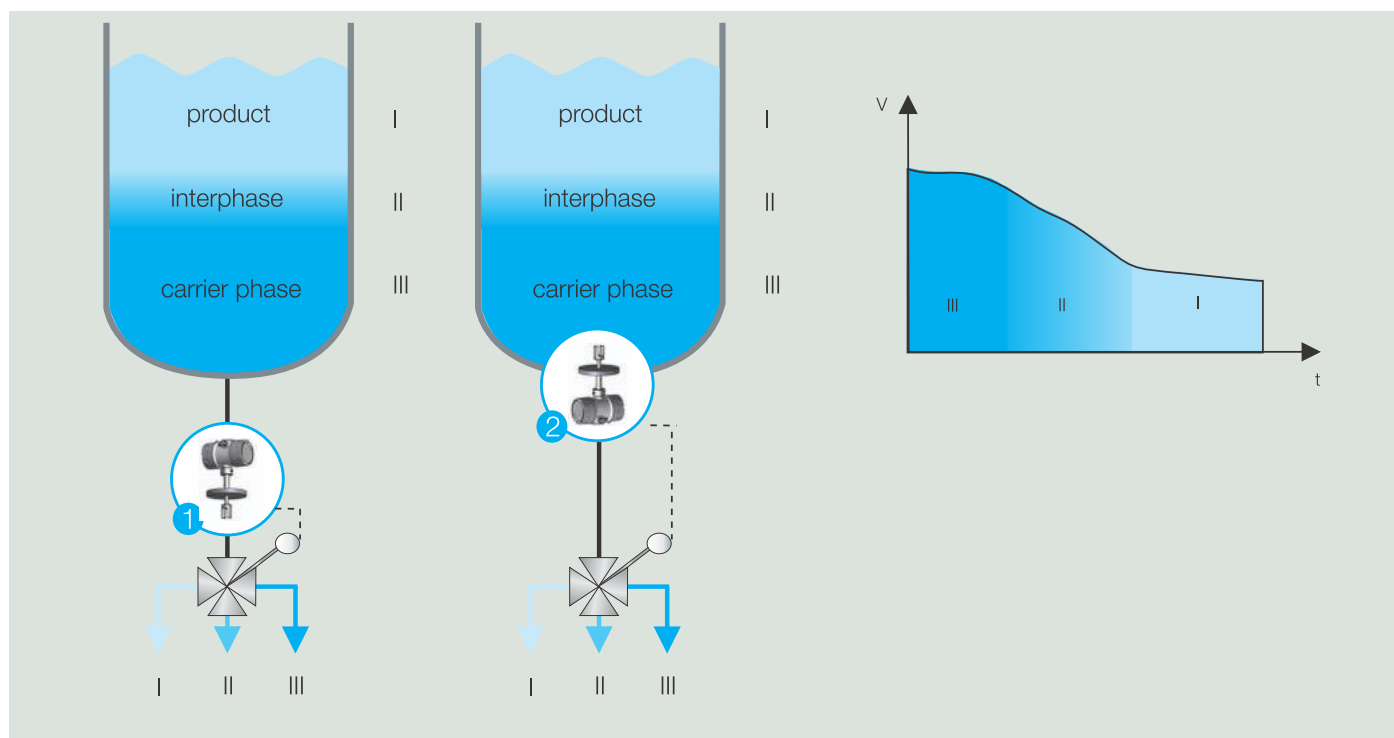
In particular, accidental fillings have to be avoided in case highly reactive substances are wrongly mixed. LiquiSonic® protects and is certified useable for potentially explosive atmospheres.

1.4 Phase Detection

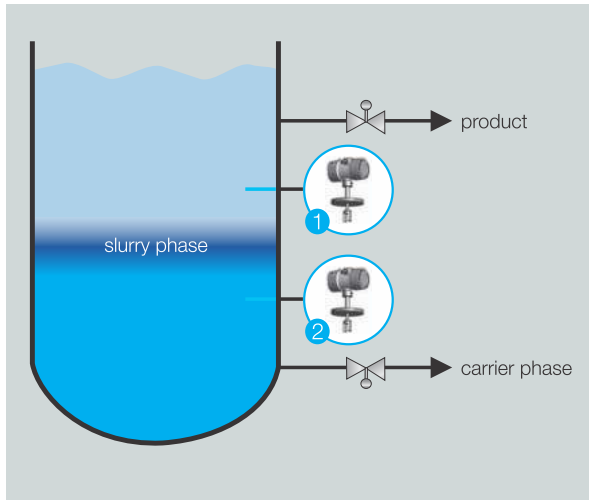
Different product phases must be efficiently separated safely from carrier phases during many process engineering intermediate steps. This is done both in continuous and batch processes. This is often achieved in batch processes by changing manually valves as well as by visual observation of phase transitions. Usually, sight glasses are used to observe the transitions visually. But this procedure is characterized by a low reproducibility. When using a LiquiSonic® immersion type sensor, the mentioned procedure is realized automatically.

The slope of the sonic velocity in the illustration below shows a typical and significant change of the signal between the individual phases. LiquiSonic® provides a clear signal to separate the product and carrier phase safely and reproducibly. In particular, the very fast response time of the sensor, within a few seconds, enables a high degree of selectivity. This results in excellent phase identification and subsequent product saving.

Similar to this is the application of LiquiSonic® in continuous phase separations. Here two sensors continuously allow the monitoring of the liquid feed and the phase flows to be separated in the settling tank. This enables an improved plant capacity in conjunction with increased product yields.



Phase separation in batch processes

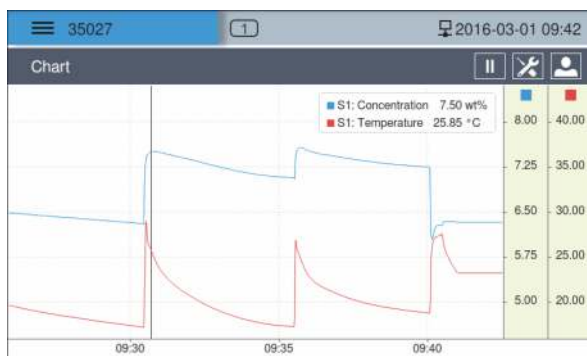


Phase separation in the continuous process

LiquiSonic® examples for application:

- silicone resin phases
- fatty alcohol phases in wastewater
- epoxy resin phases
- polyether phases
- gas-liquid phases

Compared to conductivity sensors, LiquiSonic® sensors can also be used in many different separation processes from aqueous into organic phases or vice versa. The continuous measurement allows the regulation of almost any separating ranges within the transition or slurry phases. The trend view of the controller shows directly the signal waveform. At the same time, it is possible to adjust thresholds for parameterization of digital controller outputs. For stand-alone solutions, these outputs can directly switch available diverter valves or valve clusters.



1.4.1 Drain Valve with LiquiSonic® Sensor

In pharmaceutical and fine chemical processes disc bottom outlet valves are commonly used to drain or feed non-viscous liquids from vessels or reactors. Integrating process analytical technology into the drain valve provides a real-time process monitoring along with an improvement of product quality and a reduction of batch cycle time, product sampling as well as energy and material costs.

In addition, this incorporation enables a suitable entry point to the process for an effective use of the LiquiSonic® analyzing technology. This eliminates the need to modify existing reactor vessels.

SensoTech developed, with a famous manufacturer of bottom outlet valves (BOV), the innovative bottom outlet valve with integrated LiquiSonic® sensor. As the drain valve including the sensor is mounted in the bottom part of the vessel, even low volume batches can be monitored in real-time. The sensor has an ATEX and IECEx approval. Additionally, to the concentration measurement the LiquiSonic® sensor includes two Pt1000 temperature sensors.

The data processing is managed by the proven LiquiSonic® controller hardware and software. For example, via fieldbus the measuring results can be provided to the process control system.



1.5 Crystallization

Crystallization processes in continuous as well as in batch processes can be monitored by measuring the sonic velocity with the LiquiSonic® system.

Failures or deviations from the process conditions, therefore can easily be corrected to achieve the required product quality.

In most cases, the characteristic process conditions, that result into an optimal reaction course by preliminary investigations.

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.

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

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

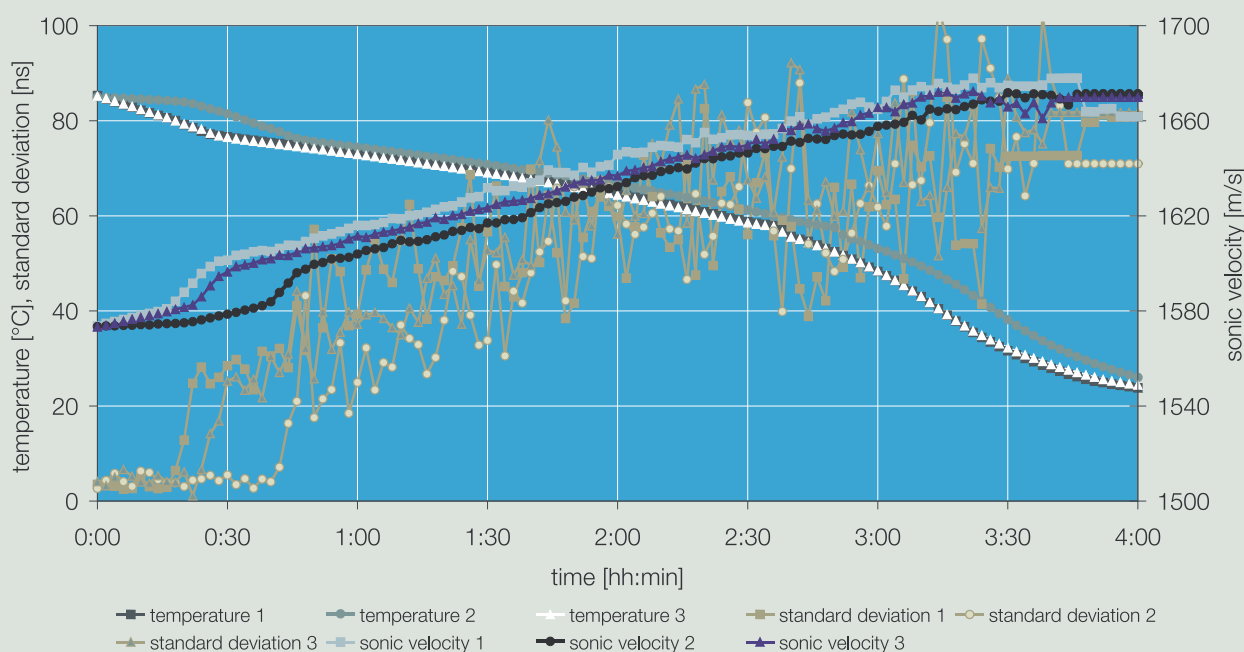
- fast achieving of required seeding point
- continuous determination of crystal content
- optimal approaching of the final process point

Saving of raw material by

- precise setting of the required product quality
- reproducible approaching of the seeding point

The determination of crystallizations parameters as well as nucleation and saturation points, the so-called metastable range, can easily be realized with LiquiSonic® Lab in the laboratory or the technical center. The system has helpful functions that greatly simplify the documentation and evaluation of the measuring data.

Statistical Evaluation of Several Sonic Measurements per Second



1.6 Crystallization Monitoring

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.

As a second physical value the attenuation is used. As the size of crystals increases the attenuation goes up. A calibration curve recorded in a laboratory makes it possible to draw conclusions on the crystal size.

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.

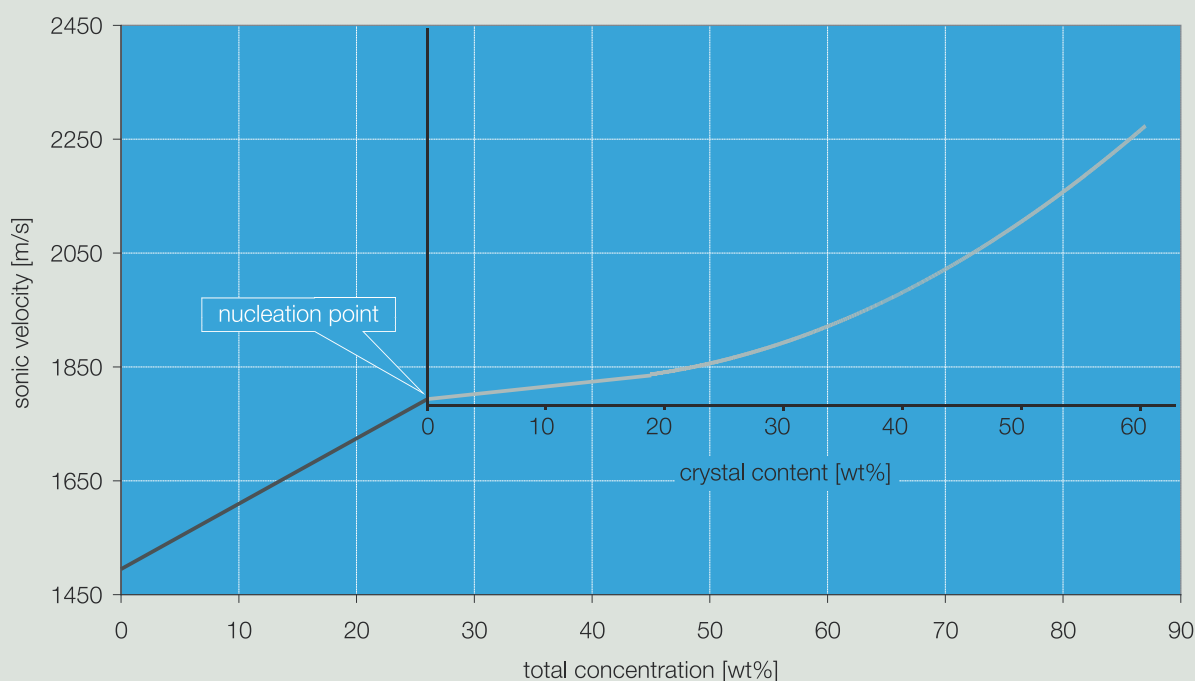
The liquid contacting sensor parts are standard made of stainless steel 316Ti. The robust and completely enclosed design requires no gaskets or "windows" for process and is thus completely maintenance free.

In very abrasive suspensions materials like Hastelloy or Titanium are used. They are the optimal choices because of their hardness.

The picture below shows the sonic velocity progression of a NaCl dissolution.

The linear increase comes about through the permanent addition of NaCl. At the nucleation point the solvent is saturated. As a result, from this point the sonic velocity rises progressively with the rising crystal content. Thus, with the LiquiSonic® analyzer crystallization processes can be optimally monitored and the nucleation point is precisely determined.

Sonic Velocity Depending on the Concentration of NaCl in Water at 25 °C



2 LiquiSonic® System



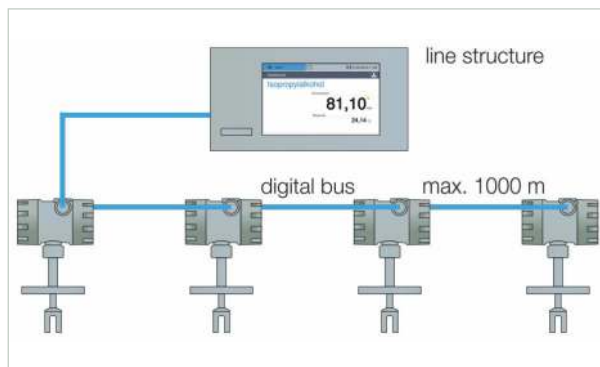
2.1 LiquiSonic® 20 und 30

The LiquiSonic® system consists of one or more sensors and one controller.

The ultrasonic sensor has the actual ultrasonic measuring path and the highly precise temperature detection.

The controller 30 is a highly efficient device which includes up to four sensors. They can be installed in different steps with a standard maximum distance of 1,000 m between controller and sensor. As option, higher distances are possible.

The controller 20 is a variant with a reduced scope of functions and only to one sensor connectable.



Controller with connection of maximum four sensors

Each sensor works autonomous and can be used in different applications. The liquid-wetted parts of the sensor are made of stainless steel DIN 1.4571 as standard. The rugged, completely enclosed design requires no gaskets or "window", making it totally maintenance-free.

Additional sensor features such as flow / stop or full / empty pipe monitoring greatly advance process control. The LiquiSonic® high-power technology stabilizes measuring results, even when facing gas-bubble accumulations or large-scale signal attenuation through the process flow.

The special sensor electronics are integrated in a closed die-cast housing with a protection degree of IP65. If necessary, the electronics housing can be mounted apart from the sensor.

For use in hazardous areas, the immersion sensor Ex 40-40 is approved by ATEX and IECEx certification (Ex d IIC T1 to T6 Ga/Gb, zone 0 / zone 1) and FM certification (Class I, Division 1, Groups A, B, C, DT1-T6).



Immersion sensor Ex 40-40

The controller 30 processes and displays the measuring results. The operation via the high resolution touch screen is easy and intuitive. Secure network integration including web server allow operating the controller alternatively via browser with a PC or tablet.

The process data is updated every second. The displayed value can be adjusted to internal reference values. If the measuring values exceed or fall below the threshold, the display shows an alarm message and a signal will be sent immediately.

The data can be transmitted in several defined analog or digital forms or through different fieldbus interfaces to communicate with process control systems or computers.

The controller features an integrated data logger which can store up to 2 GB of process information with up to 32 (optional 99) data sets for different process liquids. For processing on the PC, the data can be transferred via network or USB port. In addition, the controller enables creating easily process reports for documentation purposes.

The event log records states and configurations such as manual product switches, alarm messages or system states.

2.2 LiquiSonic® 40

The LiquiSonic® 40 analyzer enables the determination of concentration in 3-component liquids. For example, in neutralization processes it is possible to determine separately the concentration of the scrubbing solution and the salt.

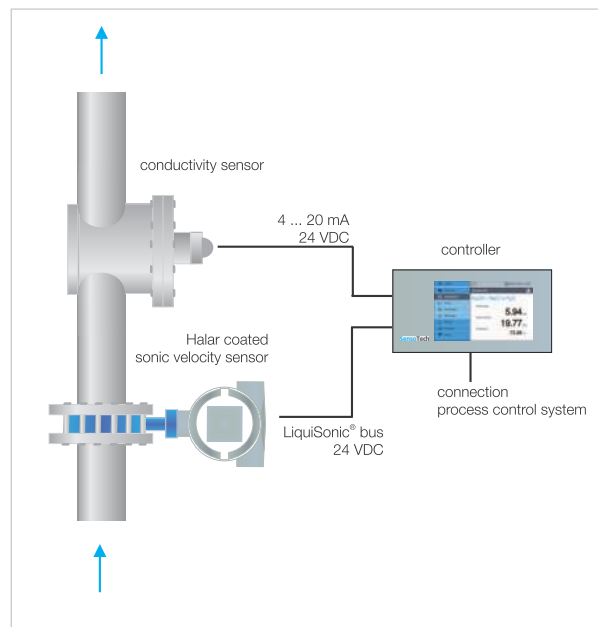
The measuring principle is based on the fact that concentration changes of individual components of a liquid affect physical quantities like sonic velocity, conductivity or density. This characteristic is stored as calculation mode in the evaluation unit (controller) to convert the physical variables in concentration values.

With the parallel detection of two physical variables (sonic velocity and conductivity), it is possible to determine two concentrations at the same time.

The measuring values are available for the user or process control system over analog outputs as well as fieldbus.

For the application in aggressive liquids, the standard LiquiSonic® 40 is equipped with a Halar (also known as E-CTFE) coated flange sensor and a PFA or PEEK coated conductivity sensor, which are chemically resistant to a number of substances.

The flange sensor has a highly efficient ultrasonic ceramic to ensure the measurement even at high portion of gas in the liquid. For the application in hazardous areas, the flange sensor has an ATEX and IECEx approval (II 1/2 G / Ex d IIB T1 to T6 Ga/Gb).



LiquiSonic® 40 measuring point



LiquiSonic® controller and Halar coated flange sensor

2.4 LiquiSonic® Lab

The LiquiSonic® Lab is especially used in laboratories, miniplants or as mobile device in process plants.

The system is provided with a splash water-proof housing, and hence is also suited for rough operating conditions.

The electronics of the controller and sensor are integrated in the housing, to which a laboratory sensor is connected. This lab sensor can be made of various materials depending on the application.

The controller processes the sensor data and is the interface to the operator by displaying the concentration values. Being equipped with a high-tech microprocessor, the controller even copes with complex concentration calculations.

The internal data memory has a capacity of up to 32,000 records. By reading out via the integrated TCP/IP or RS-232 interface, it is possible to create easily own process reports.

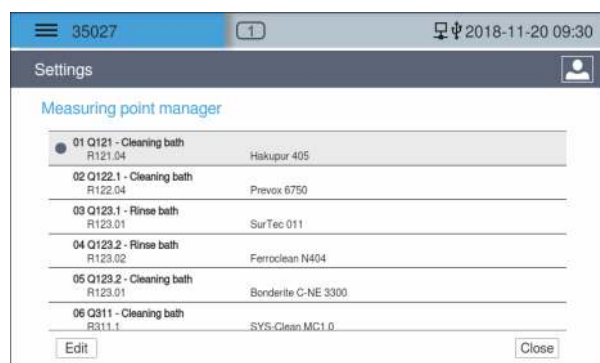
The displayed value will be updated every second.

Measuring Point Manager

To further reduce efforts of monitoring the process and simplify the handling of the LiquiSonic® Lab Unit, the Measuring Point Manager is provided. This allows an easy switching of the measuring point and thus the product, depending on where the employee is currently located.

The actual product to be measured is assigned to the measuring point in advance. Thereby time is saved and operating errors are avoided.

Measuring Point Manager of the LiquiSonic® Lab Controller



Limit Value Display

In addition to the numeric concentration, density and temperature displaying, the visualizing of limit value ranges is available. That simplifies the monitoring of an approximation to critical values.



Limit Value Display of the LiquiSonic® Lab Controller

Users can identify the segment in the measuring range where the current value is settled on and get an overview quickly. When approximating the borders of the measuring range, the arrow turns into yellow signaling a warning. A range excess is shown by a red arrow.

2.3 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.

2.3.1 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.



Controller housing 19" 4 HU

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

2.3.2 UMTS Router

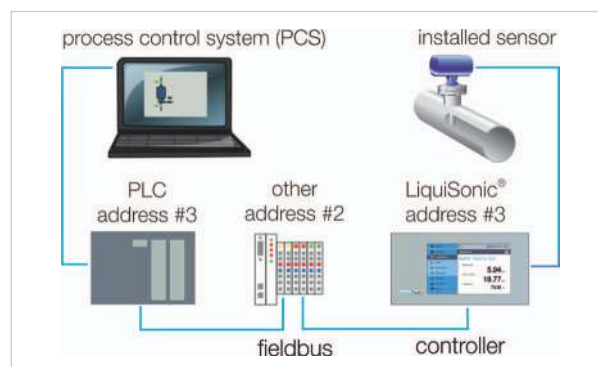
With a UMTS router it is possible to operate remotely the LiquiSonic® controller. For this purpose, the controller is connected to the UMTS router and appropriate IP address must be entered in the browser on the PC.

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

2.3.3 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).



Connection interfaces

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

2.3.4 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).



Setting standards in liquid analysis



3 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.



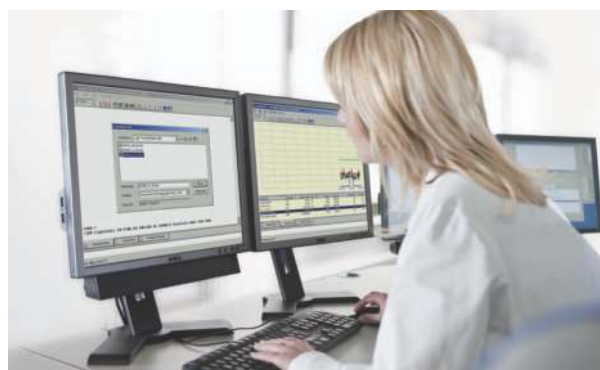
Creative research is another pillar of our company. The specialists in our research and development team provide valuable new ways to optimize product attributes, such as testing new types of sensor designs and materials or the sophisticated functionality of electronics, hardware and software components.

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 maximum: 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 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.



SensoTech

sensosquad

Setting standards in liquid analysis

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.



Setting standards in liquid analysis

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