

LiquiSonic®

Ultrasonic analyzer for liquids

LiquiSonic® Method

LiquiSonic® is a highly sophisticated inline/in-situ liquid analyzer well-suited to biotechnological, pharmaceutical, supersaturation, concentration and crystallization processes. Using sonic velocity and temperature measurement technology combined with a unique sensor design, the system allows control and monitoring of concentrations and general process trends at different points.

LiquiSonic® supplies the operator with real-time knowledge needed to optimise the process. A **LiquiSonic®** system consists of one or more intelligent sensors and a controller connected to each other by a digital line.

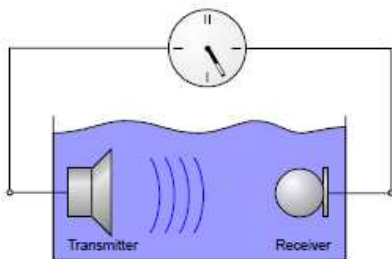
In addition, modern manufacturing technologies guarantee precise measuring results and convenient device operation. This includes the simultaneous presentation of mass-concentration or crystal content, product temperature, and product or recipe identifiers.

The data memory and event tracking capabilities (according to FDA 21 CFR Part 11) in conjunction with user-defined passwords ensure a maximum degree of process and application safety and security.

A multi-channel, real-time chart and the non-volatile configuration and process data RAM card allow easy system adaptation for lab, pilot, or production scale applications.

LiquiSonic® sensors are available in different designs and process fittings to suit tubes or vessels. For installations in hazardous areas, an explosion proof sensor is available. All sensors can be equipped with an electro-polished finish, and all sensors may have an ultra-sanitary design without gaskets to handle the toughest process environments and typical CIP / SIP procedures.

All systems include inline validation capability, which guarantees precise, traceable, and reproducible results under every circumstance.



Variants

LiquiSonic 30 is the standard device with all functions. Up to 4 sensors can be connected with one controller. Several functions, like trend presentation or the logbook make this device to an efficient analyzer.

LiquiSonic 20 is the low budget version with only basic functions.

LiquiSonic 40 calculates the concentrations of 3-components liquids. The controller uses the sound velocity and an external signal like the density or the conductivity.

LiquiSonic 50 has features for optimal application in crystallization and polymerization processes.

General

measuring principle	determination of sound velocity in the liquid as dimension for components' concentration
measuring range	100 to 10,000 ms ⁻¹
typical accuracy	0.05 ms ⁻¹
accuracy of temperature measurement	0.1 °C
communication	data exchange with the PLC via current signal 4.. 20 mA, RS-232, fieldbus
sensor connection	connection of ultrasonic sensors with controller via BUS-cable

Range of application

- concentration measurement
- density measurement
- monitoring of polymerization, determination of the conversion ratio
- fast phase interface detection
- monitoring and control of crystallization, determination of supersaturation degree and metastable range
- control of neutralization processes of acids and lye's under higher process temperatures and pressure
- process monitoring
- control of scrubbers, distillations and absorbers



SensoTech - Benefits & Advantages:

- **Providing direct information about the state of the process at any time**
- **Automatic control of scrubbing liquid concentration in the range of maximum absorption or maximum efficiency of the gas scrubber**
- **Avoiding overdosing and underdosing**
- **Reducing time-consuming lab measurements**
- **Detecting process faults immediately**
- **Saving costs and materials**

Gas Scrubbers

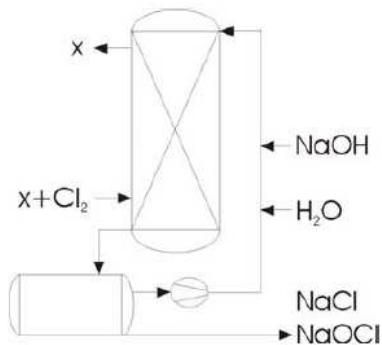
1. Benefits & Advantages

LiquiSonic® is an inline analytical system for determining the concentration of the scrubbing solution directly within the process and without delay. **LiquiSonic®** is based on high-precision measurement of the sound velocity and the process temperature, thus facilitating the calculation and monitoring of concentrations. Benefits and advantages for the user include:

- provides direct information about the state of the process at any time;
- automatically controls the scrubbing liquid concentration in the range of maximum absorption or maximum efficiency of the gas scrubber;
- avoids overdosing and underdosing;
- reduces time-consuming lab measurements;
- detects process faults immediately;
- saves costs and materials;

Example:

When dichloroethane is produced, a chlorine residual remains in the exhaust gas which is scrubbed by using excessive 5% NaOH. Overdosing of 1% NaOH causes high costs as any excessive volume of NaOH needs to be discharged with NaCl.



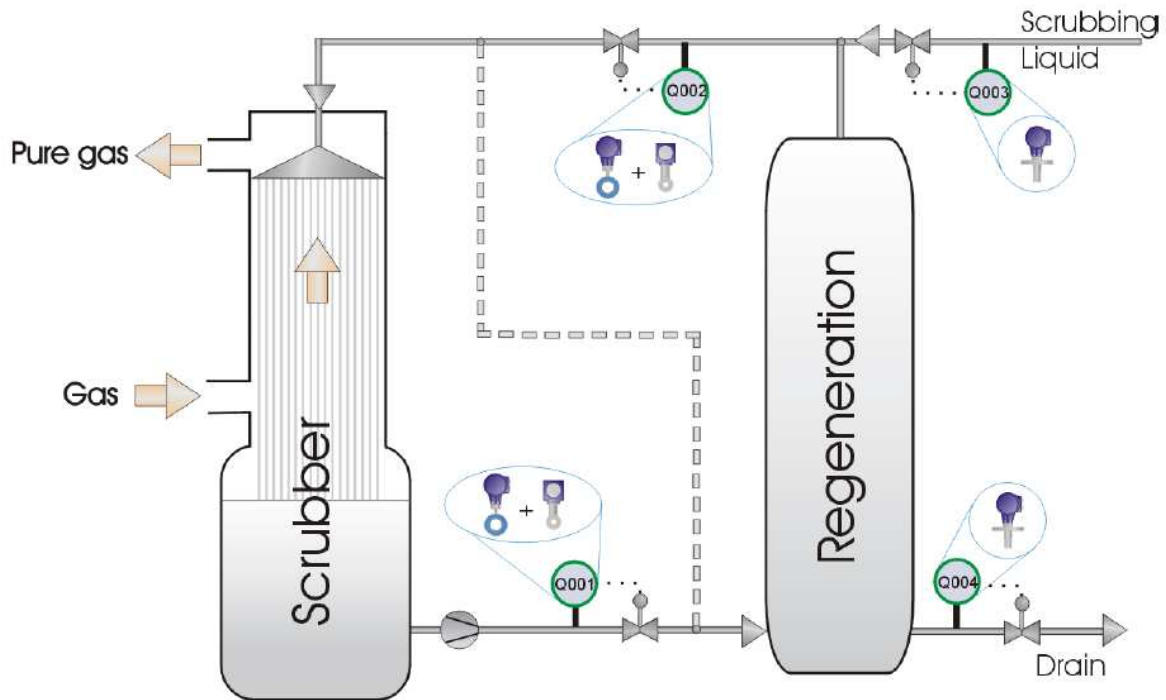
Circulation flow	50m ³ /h
Addition of 20% NaOH	300kg/h
Costs of NaOH (100%)	250€/MT
Maintenance costs of LiquiSonic	0€/a
Excessive 1% NaOH in the circuit corresponds to	60kg/h = 105MT/a
Costs caused by overdosing	26250€/a
Costs of installing a measuring point	27000€/unit

In this example the installation costs of a **LiquiSonic® 40** measuring system are amortised within **1 year**. Savings in the subsequent years amount to **26250 €/a**.

2. Processes

In a gas scrubber a liquid flow (scrubbing solution) is brought into contact with the gas stream to be scrubbed. The liquid absorbs certain gaseous, liquid or solid components. Depending on the quantity of the absorbed components the recirculated scrubbing liquid needs to be refreshed or regenerated.

The figure below depicts the principle of operation as well as the application of the **LiquiSonic®** system by means of a jet scrubber.



Measuring point	Location	Measuring task	Fluid (example)
Q001, Q002	Supplying and conveying pipes	Control and monitoring of scrubbing liquid and salt concentrations	NaOH/NaCl, K ₂ CO ₃ /KHCO ₃
Q003	Supplying and conveying pipes	Control and monitoring of the fresh scrubbing liquid concentration	NaOH, K ₂ CO ₃ , H ₂ SO ₄
Q004	Conveying pipes	Detection of intrusions when discharging the salts	NaCl, Na ₂ SO ₄ , Na ₂ CO ₃

The **LiquiSonic®** system facilitates separate inline determination of the scrubbing solution concentration and the salt concentration without any time delay. Hence, it is possible to top the scrubbing liquid in a defined manner and keep the liquid concentration always in the range of maximum solubility and absorption. Underdosing, i.e. insufficient scrubbing, is avoided as well as overdosing, i.e. excessive input of materials and related costs.

To date, inline **LiquiSonic®** systems are using successfully for the scrubber applications listed below:

- Chlorine gas scrubbers
- Phosgene scrubbers
- SO_x, NO_x and CO_x scrubbers
- Syngas scrubbers
- Benfield process
- Gas drying

In-line determination of sulphuric acid and oleum concentrations

Because of its hygroscopic properties, sulphuric acid is widely used as a desiccant (e.g. for drying chlorine in chlor-alkali analysis) in the concentration range between 90 and 100%wt. As a result of the water absorption, the concentration decreases, so that it is necessary to continually add fresh acid in order to maintain the concentration at the required level.

The permanent in-situ measurement of the acid concentration is required for controlling such processes. LiquiSonic sonic velocity measuring instruments are excellently suited for this purpose.

In the range between 80 and 100%, the sonic velocity changes by about 300m/s. LiquiSonic systems are capable of measuring the sonic velocity with an accuracy of 0.1m/s. This makes it possible to achieve accuracies of 0.05%wt in concentration measurement applications.

For cost reasons, conductivity sensors are widely used for measuring the concentration of sulphuric acid. However, a comparison of the characteristic curves obtained in conductivity and sonic velocity measurements clearly shows the drawbacks of this approach. The conductivity curve shows a strongly non-linear shape. As a result, unambiguous measurements are no longer possible below a value of 94 %, because the conductivity of an 88 % acid is the same as that of a 95 % acid.

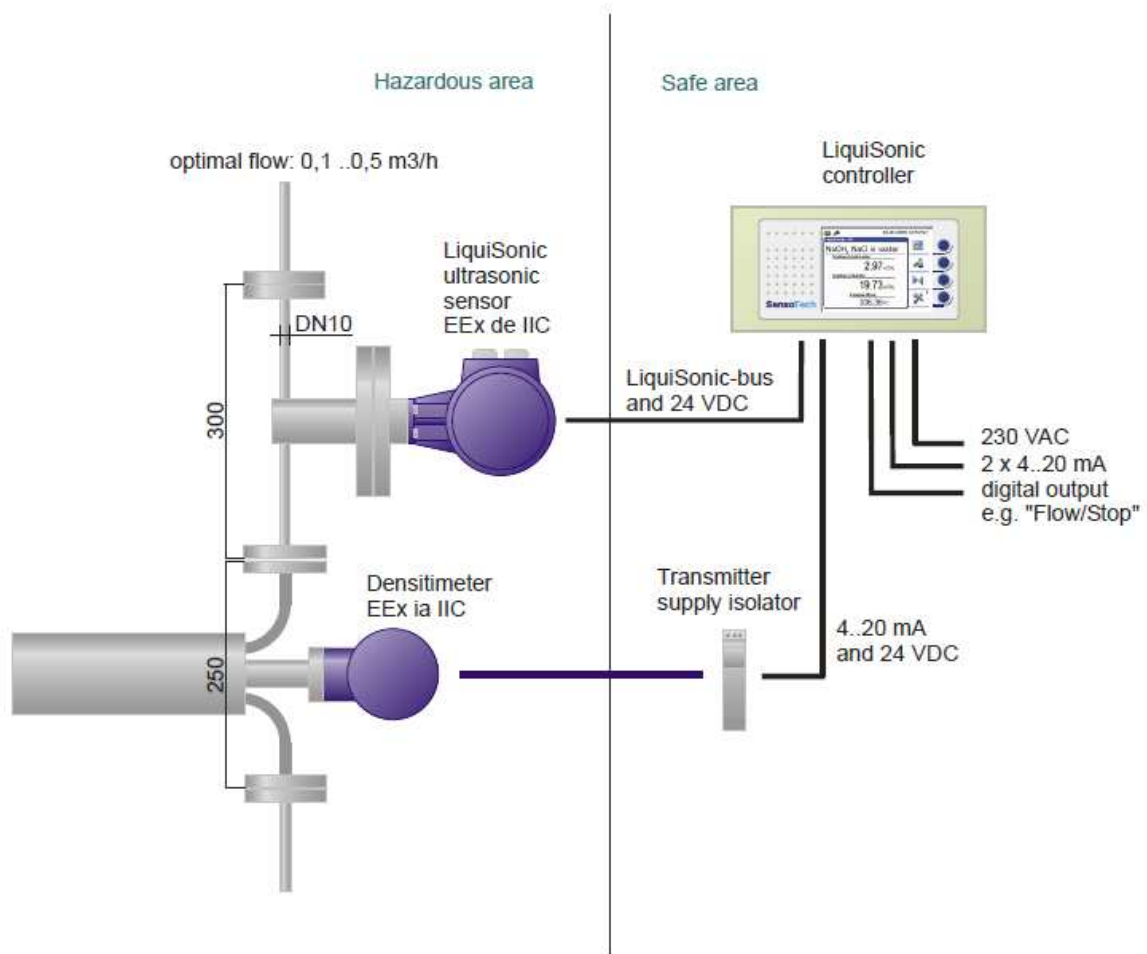
Oleum is obtained by solving SO_3 in 100% sulphuric acid. The resulting acid is designated as fuming sulphuric acid. Depending on the quantity of SO_3 dissolved, concentrations from 100 to about 130 %wt are obtained.

It is possible to set any desired values of highly concentrated sulphuric acid by blending (mixing) sulphuric acid and oleum. For setting these values, permanent in-situ monitoring of the concentration is necessary. Unfortunately, the sonic velocity characteristic has a reversal point at 100%, so that, by using just one sensor, it is not possible to carry out an unambiguous measurement within the sulphuric acid/oleum system.

Therefore, the LiquiSonic40 system uses, in addition, a density sensor, which is of decisive importance for the accuracy of the concentration measurement in the range above 100 %:

The accuracy of the used oscillating type sensor is 0.1 kg/m^3 . In the range considered, the density changes by about 100 kg/m^3 in the range between 100 and 130 %wt, which means that an accuracy of 0.05 %wt is also achieved in this case. For achieving this accuracy, however, the temperature needs to be determined with an accuracy of $0.1 \text{ }^\circ\text{C}$.

The following diagram shows the arrangement of the individual components of the LiquiSonic40 system. The oscillating type densitometer and sonic sensor are implemented as separate sensors. In contrast to the compact configurations found in some applications, only this configuration allows a **highly accurate temperature measurement to be carried out via the sonic measurement performed by devices which are directly immersed in the medium**, thus making it possible to achieve a measurement accuracy of 0.05 %wt.



Another advantage of our system is the **caloric flow detector (flow/no flow) which is integrated** in the sonic sensor. In view of the fact that the measuring system will normally be used in the by-pass, this flow detector monitors the by-pass for the presence of a medium flow through the by-pass and, by doing so, it is monitoring the overall configuration for proper functioning.

Phase detection in processes

There is a large number of industrial processes in which, as a result of the techniques used, various phases are produced in the liquid media involved. This applies to both continuous and batch type processes.

Due to the continually increasing degree of automation, measuring techniques have to be developed which allow users to reliably detect these phases as well as phase transitions during the running process. In the development of appropriate solutions, the following aspects need to be taken into account:

- Substitution of manual detection techniques (e.g. visual ones) so as to eliminate subjective influencing factors.
- Substitution of erroneous, indirect detection techniques (e.g. timing) so as to enhance the safety of plants
- Improvement of product yield through the reduction of losses
- Safety aspects

The sensors can be used in a wide range of applications. The figure shows two typical installation situations.

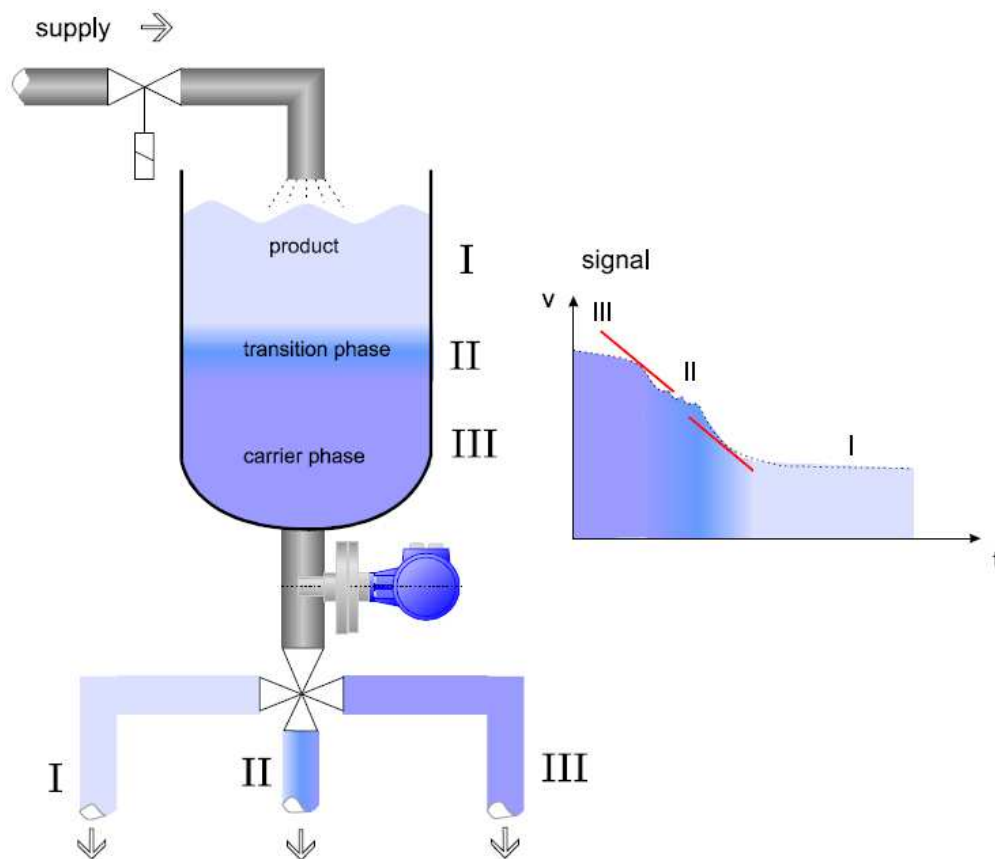


Fig. 3 Phase separation in a batch process

In a batch process (Fig. 3), the temporal change of the sound velocity signal is used for controlling a multiway valve. After the filling of the settling tank the phases are separated the configuration provides for a precise separation between the product, intermediate and carrier phases.

The phases are typically detected within a second.

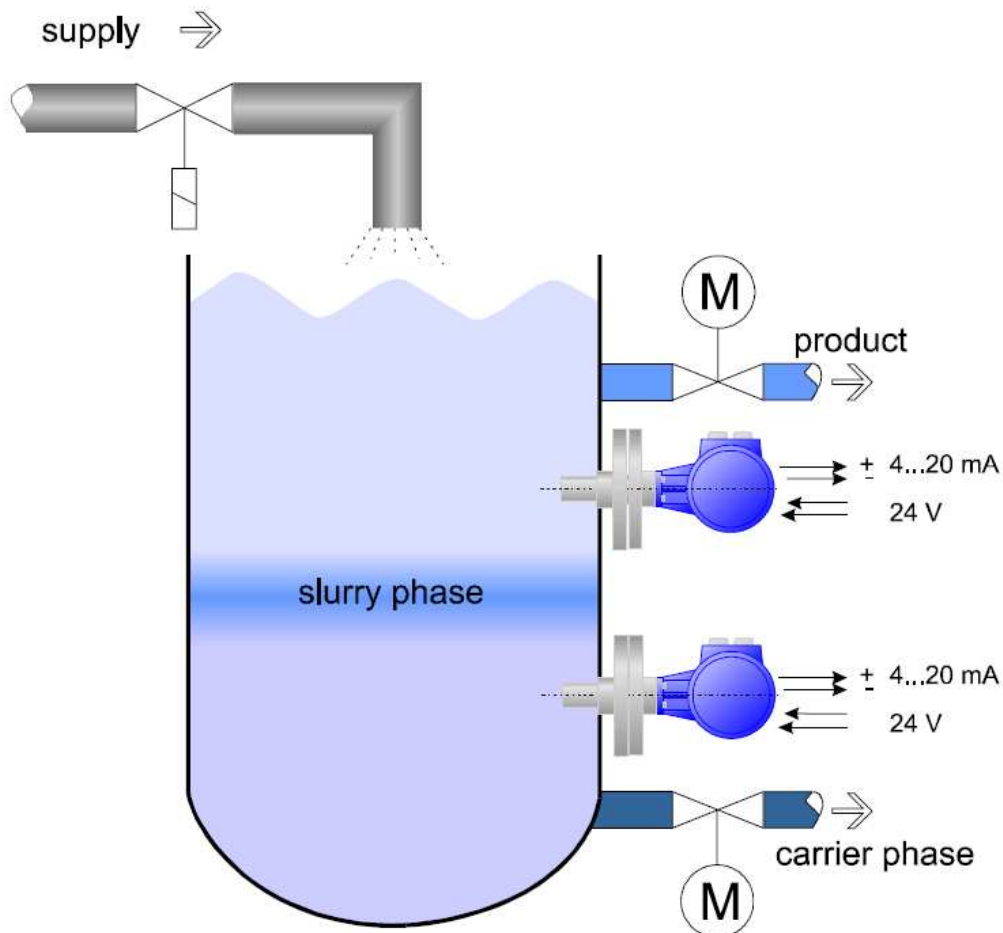


Fig. 4 Phase separation in a quasi-continuous process

In a quasi-continuous process (Fig. 4), two sensors installed in a settling tank are used to control the separation of a process flow into a product and a carrier phase. In this case, only the sound velocity value which is characteristic of the respective phase is monitored and kept constant.

In this way it is possible, for example, to control the separation of a waste-water flow into waste water requiring treatment and waste water for which no treatment is required.

Using ultrasonic measurement for phase separation is one possibility for automating phase separation services. It also offers a useful alternative to existing measurement solutions. In view of the fact that the sensors are maintenance-free and due to the very quick response speed, the ultrasonic measuring method has proven its worth in a large number of other applications:

- Silicone resin phases
- Production of Biodiesel
- Fatty alcohol phases in waste waters
- Epoxy resin phases
- Polyether phases
- Polyetherphasen

Steel- and rolling mill

Rolling

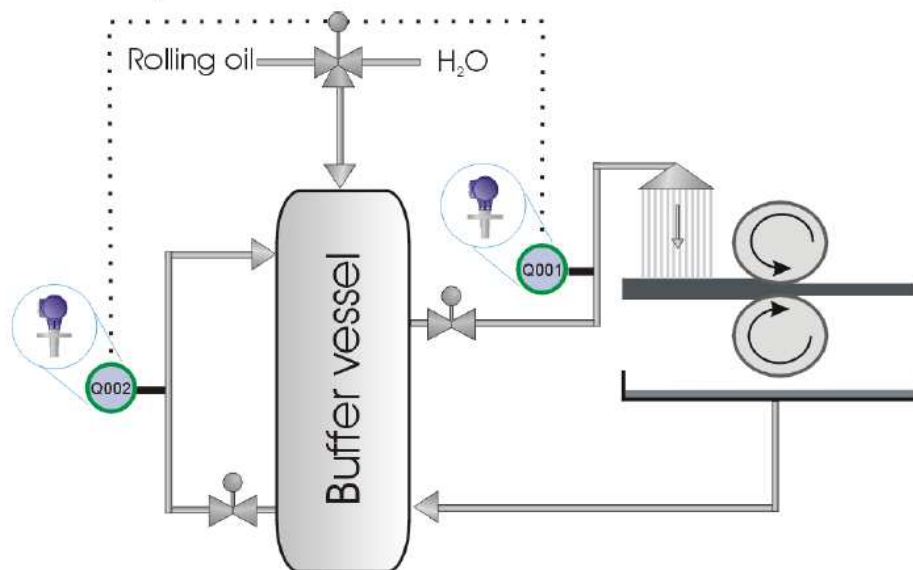
Hot and cold rolling processes require the use of technical liquids, such as rolling oils, rolling oil-in-water emulsions, temper rolling liquids or process detergents.

Typically, these process liquids are circulated and regenerated in closed circuits. SensoTech offer tried and proven solutions to ensure continuous quality surveillance of these liquids for a great many of typical makes, such as from Quaker, Henkel or Houghton.

SensoTech's solutions for rolling mills are successfully employed for all types of rolling stands and treatment plants for stainless steels, but also for carbon steels and non-ferrous metals.

In a specific application the concentration of the rolling oil-in-water emulsion is directly measured in the buffer tank of the rolling stand and controlled such that it is kept on a constant level. This prevents variations in the quality of the rolled stock caused by under lubrication or over lubrication. Such effects mainly occur as a result of unavoidable water loss and foreign oil migration.

Another measuring point is arranged where the emulsion is supplied to the rolling stock. Thus, **LiquiSonic**[®] provides an in-line solution to ensure continuous quality of the rolled stock without delay, while making optimum use of the rolling oil.



Measuring point	Location	Measuring task
Q001	Inlet at rolling stand	Monitoring of emulsion concentration at the inlet
Q002	Circulation loop of buffer tank	Monitoring of emulsion concentration in the buffer tank of the rolling stand
Q003	Recovery	Monitoring of emulsion regeneration

Calculation ROIC for rolling oil:

Price of rolling oil e.g. for China Steel corp.: 2,56 EUR/l

Top up rolling oil emulsion ~100 Liter /hr

Target: minimize the excess of oil in emulsion.

Saving Oil by In-line Control up to 5% /day (614,40 EUR/day)

Cost of installation ca. 13.000 EUR (viz. 26.000 EUR for complete installation)

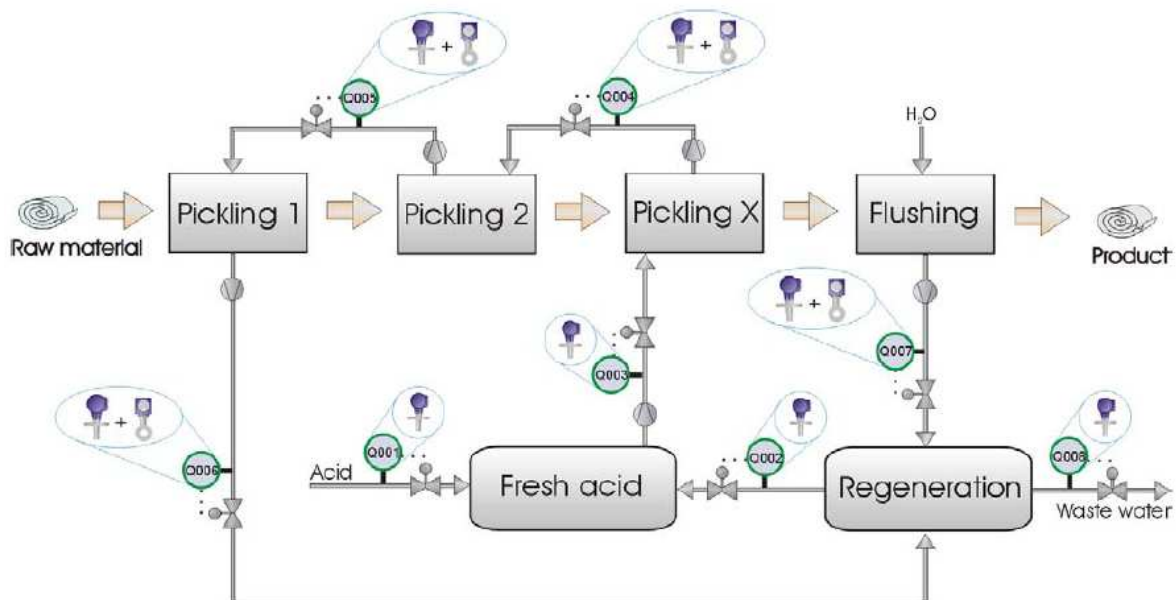
ROIC = 90 days

Pickling

Pickling baths are used downstream of the hot rolling process, but also in many other fields of the metalworking industry to remove, modify, passivate or clean surfaces in a defined manner.

To this end, pickling solutions are employed, mainly consisting of a mixture of mineral acids. The concentration of these acids decreases during the pickling process, whereas the portion of disturbing components, such as contaminants and migrations, increases.

The **LiquiSonic**[®] measuring technology provides a solution for online measurements of pickling bath concentrations facilitating redosing of the required quantity of fresh acid. This ensures a continuous, optimum pickling bath quality. Delays in time as a result of sampling and lab analyses are avoided.



The table depicts the typical **LiquiSonic**[®] measuring points for determining concentrations in pickling baths.

Measuring point	Location	Measuring task
Q001, Q002, Q003	Fresh acid inlet	Concentration measurement and fresh acid redosing control
Q004, Q005, Q006	Transporting pipe	Pickling bath monitoring
Q007	Transporting pipe	Flushing bath monitoring
Q008	Flow out pipe	Detection of acid irruption in process water

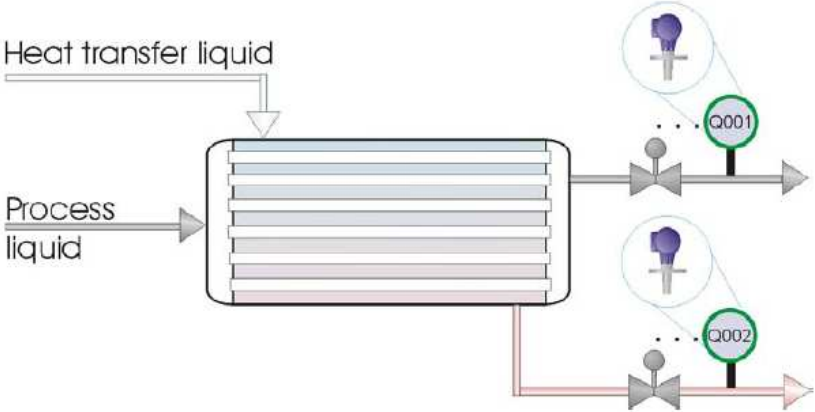
To date, inline **LiquiSonic**[®] measuring systems are using successfully for the pickling bath applications listed below:

- Sulphuric acid (H₂SO₄)
- Hydrochloric acid (HCl)
- Nitric acid (HNO₃)
- Hydrofluoric acid (HF)

Heat Exchanger Monitoring

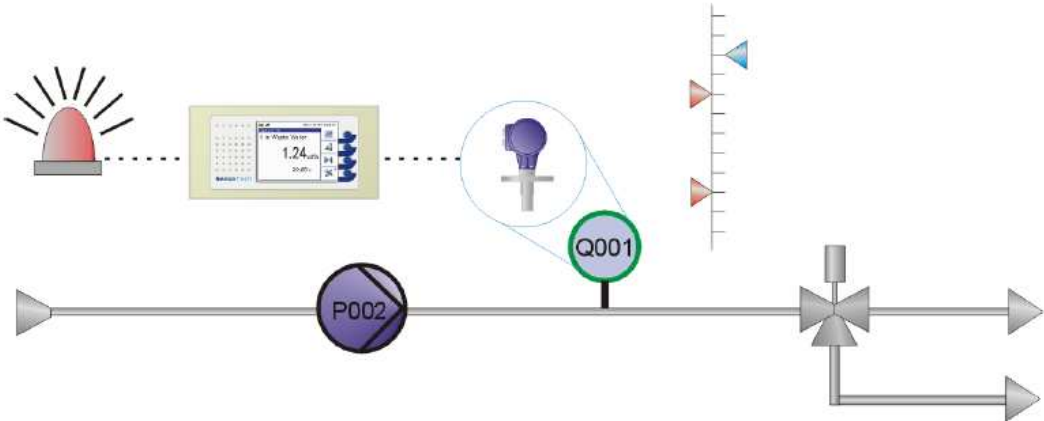
Monitoring of primary and secondary circuits in heat exchangers is necessary to detect leakages and incursions immediately and, thus, prevent contamination of the product or the cooling liquid.

In the production process of steel heat exchangers are employed, for instance, to bring the rolling oil to an optimum temperature. **LiquiSonic**[®] can be used for any tempering fluid in a range from -90°C to $+200^{\circ}\text{C}$.



Effluent Monitoring

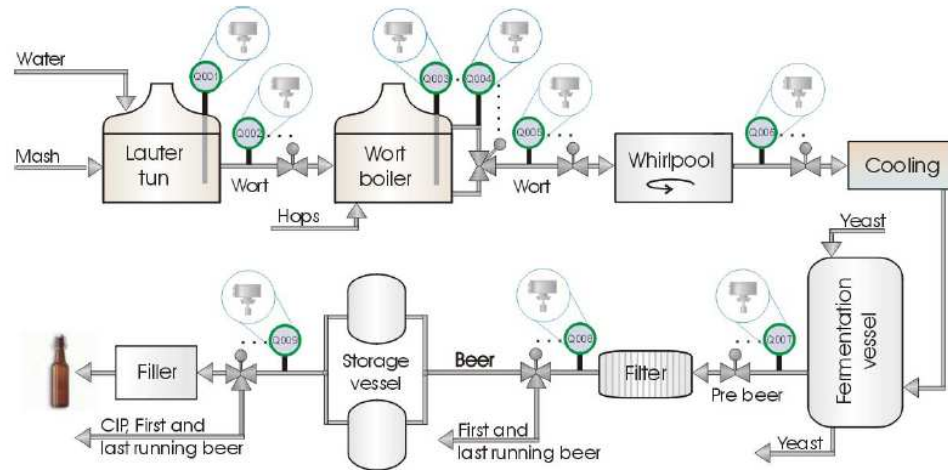
Effluents are produced at many different stages of production. Their content of dissolved matter is subject to applicable regulations. If pre-defined limit values are exceeded due to leakage or misdisposal, problems may be encountered in the treatment process. Therefore, **LiquiSonic**[®] systems are employed for monitoring effluents, reporting incidents immediately.



Brewery

Application in breweries

- Mash filter
- Lauter tun
- Wort boiler
- Filtration
- Filler
- Blending
- CIP
- Yeast recovery



Lauter tun

Application

- Control of the complete lautering process
- Precise detection of the sparge water point at the end of the lautering process
- Continous determination of the actual extract concentration in the buffer tank or wort boiler using an external flowmeter

Advantages

- Optimal initial extract concentration in wort boiler or buffer tank
- Reduction of fresh water demand
- Optimal utilization of lauter tune and an efficient wort production

Precise monitoring of the actual wort concentration in buffer tank or boiler

- Optional feature using a external flow meter
- Actual wort concentration will be multiplied with actual flow to have the real wort concentration in the buffer tank or wort boiler
- Allows precise set-up of the boiling start up concentration
- Allows reduction of energy consumption during the wort boiling

Wort boiler

Application

- Monitoring of the concentration during the complete boiling process
- Precise control of the final wort concentration
- Immersion type sensor for application in the boiler

Advantages

- Avoid of extensive maintenance effort because of traditional bypass solutions
- Reduction of additional rinsing and cleaning cycles
- Reduction of energy consumption and workforce
- Reproducible boiling process.

Filter

Application

- Precise, gravity controlled separation of first and last runnings
- Feed control of recovery beer or first and last runnings in the filter inlet

Advantages

- Extensive cost reduction

Filler and blending

Application

- Continuous monitoring of original gravity during the filling process
- Documentation and data logging of original gravity (ISO9000 und HACCP).
- Event tracking according FDA recommendations
- Separation of different brands.
- Control of original gravity at High-Gravity Brewing.

Advantages

- Display of wort concentration temperature of beer.
- Continuous logging of original gravity during the filling.
- Immediate detection at faulty process conditions (mismatch of bottles and labels)
- Control of the filler, if the actual wort concentration leaves a specified range, the filler stops and the beer can run into a buffer tank.
- Separation of first and last running beer: Automatic separation of first and last running by setting of reverse points.
- Analog input for any CO₂-analyser including automatic compensation function

Pharma

LiquiSonic sets the standard

SensoTech analyzers:

- Supply reliable data about solvent concentrations
- Are used as detectors or to monitor eluate concentrations in HPLC processes
- Control phase interface detection in vessels and pipes

Another field of application is the control of crystallization processes for the reproducible generation of crystal sizes and the purification of substances.

Unbeatable Utilization Rates

LiquiSonic sensors provide a stable and reliable measurement. Autoclavable or CIP/SIP, SensoTech sensors are specially engineered for use in laboratory- pilot- or production –scale reactors and conform to all FDA, 3A standards or EHEDG recommendations.

Direct, real-time measurement eliminates the need for sampling, eliminating volume loss and dilution errors in addition to reducing the risk of contamination. LiquiSonic allows you to automate manual operations and achieve a new level of productivity and process control.

The fundamentals for this quality are the high-grade materials used such as stainless steel with electro polish finish. The sensor design consists of one homogenous body and requires no gaskets.

Well Connected

SensoTech sensors may be equipped with all common process adapters and fittings like Tri-clamp, Ingold or Varivent, and can be used under the most complex application conditions.

By changing immersion lengths or the process adapters, SensoTech sensors fit into tanks and vessels of almost every design as well as directly in any tube size.

Because the measuring technology is not dependent on color, turbidity or other optical attributes, the user will have no limitations, even under the most difficult application conditions.

Monitoring 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 process measuring methods have been developed, including methods based on density measurement, refraction index measurement, conductance measurement, the measurement of colour, turbidity and viscosity, all of these methods being characterized by specific physical and technological limitations.

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

Ultrasonic sensors with greatly improved technical parameters have made the determination of concentrations and conversion in polymerization reactions a highly promising field of application of ultrasonic measuring technology.

Typical polymer systems

The fundamental correlations described above have been verified in the past in a number of polymerization reactions:

- solvent polymerization of butadiene
- emulsion polymerization of vinyl acetate
- emulsion polymerization of butadiene and styrene
- emulsion polymerization of vinyl chloride
- emulsion polymerization of acrylates
- suspension polymerization of styrene
- suspension polymerization of vinyl chloride
- polycondensation of urea and formaldehyde
- polycondensation of phenol and formaldehyde

Typically, the sonic velocity of all components involved is determined as a function of temperature. From the time course of the sonic velocity, you can then derive the course of reaction and calculate the materials conversion.

Optimal crystallization processes with inline control

LiquiSonic® is an inline analyzer using in liquids. The precise determination of both sonic velocity and process temperature enables the monitoring of concentrations, compositions, changes in characteristics or material properties directly in liquid processes.

The use of modern digital signal processing technologies guarantees an extremely precise and fail-safe measuring of absolute sonic velocity. The calibration models are based on know-how growing in numerous applications. The high sophisticated design of the sensors ensures a high reliability, outstanding durability and maintenance free operation.

LiquiSonic® systems consist of one or more sonic velocity sensors and the evaluation unit (controller).

The outstanding advantages of the method are:

- Absolute sonic velocity as unique and traceable physical property
- Installation directly in pipes, tanks and vessels
- Independent on color, conductivity and transparency of the process liquid
- Rough sensor design; all wetted parts are stainless steel without sealing, optical windows or adhesive bonding
- Maintenance free
- No influences by mechanical vibrations and pressure peaks as well as non-corrosive by special alloys or plastic coats

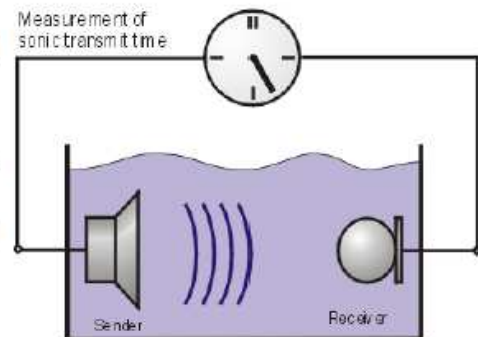


Fig.1: Measuring principle of the ultrasonic velocity realized via the measuring of the sonic transmit time

The following **advantages** result for the user in crystallizations:

- 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 seedy 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 seedy point

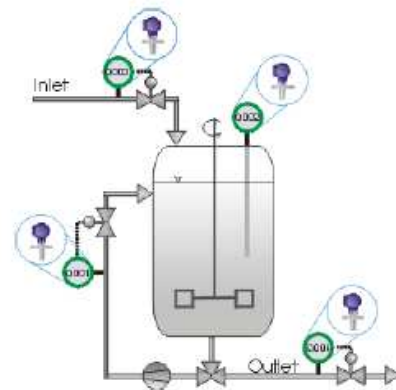


Fig.2: Possible installation points of sonic velocity sensors within crystallization process.

Successful Cooperation

TEAMWORK IS OUR TARGET!

