Density or Sonic or Both?

Comparison of density and ultrasonic measuring instruments

LiquiSonic® Application Report

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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.

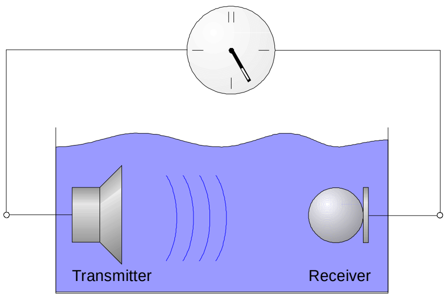
In addition, modern manufacturing technologies guarantee precise measuring results and convenient device operation. This includes the simultaneous presentation of mass-concentration or crystal content, product temperature, and product or recipe identifiers.

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.



Introduction

Process analysis is playing an ever greater part in modern chemistry. The measurement of the concentration of liquids, in particular, has increased in importance over the past few years.

This paper will deal with two important concentration measurement principles – determination of the concentration via measurement of density and via measurement of sound velocity. In the first part of this paper, the two principles will be compared, and in the second part, the impact of practical conditions on accuracy will be shown. The third part outlines the relationship with the concentration of liquids.

Measurement principles

Density measurement principle

The density measurement is based on a spring-mass system: A tube is made to oscillate mechanically (Fig. 1A). The frequency of oscillation is dependent on the spring constant and on the mass of the tube including its content.

$$f = \frac{1}{2\pi}\sqrt{\frac{k}{m_g}}$$

f = *oscillation frequency*
k = *spring constant of tube*
m_g = *mass of tube* m_r *and of liquid* m_{fl}

If the mass and the volume of the tube are assumed to remain constant, the density of the liquid contained in the tube can be determined by measuring the frequency.

$$\rho = \frac{m_{fl}}{V}$$

ρ = *density of liquid*
m_{fl} = *mass of liquid*
V = *volume of tube and of liquid*

To achieve a high resolution in the density measurement, the tube thickness should be very small and the mass of the tube very low. This will be achieved by the use of „light-weight“ materials such as titanium.

The spring stiffness of a tube is dependent on temperature. The temperature is usually measured in the instrument so that this dependence is compensated for.

Principle of sound velocity measurement

The sound velocity measurement is based on a time measurement. An ultrasound signal is sent through the liquid from a transmitter (S) to a receiver (E), and the time it takes for the sound to travel from the transmitter to the receiver is measured (Fig. 1B).

If the distance between the transmitter and the receiver is assumed to be constant, the sound velocity can be directly measured.

$$v = \frac{s}{t - t_e}$$

v	=	*sound velocity*
s	=	*distance between transmitter and receiver*
t	=	*total travelling time of signal*
t_e	=	*travel time through electronics*

To achieve a high sound velocity resolution, the distance between the transmitter and the receiver should be large.

Factors influencing measuring accuracy

Measurement accuracies

For the accuracy of measuring instruments, definite values are specified. For densimeters, they usually apply to temperatures from 0 to 50 °C and a pressure of 1 bar. High-quality process densimeters achieve a density measurement accuracy of $\pm$ 0.1 kg/m^3 (10^{-4}).

For ultrasound instruments, accuracy values usually are specified for the range from 0 to 100 °C and pressures up to 10 bar. High-quality ultrasound measuring instruments achieve an absolute accuracy of ± 0.1 m/s.

In practice, however, these accuracy values are affected by various conditions.

Below, the impact of practical conditions on the accuracy of density or sound measurements will be considered.

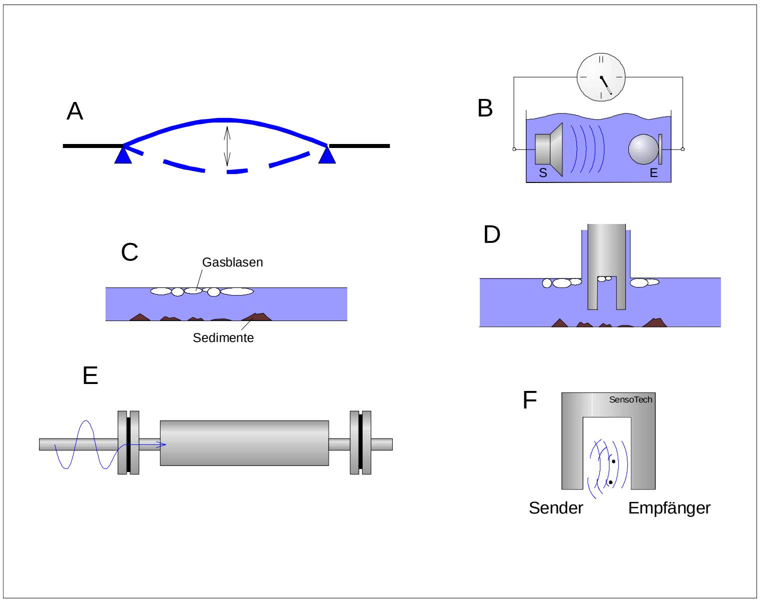


Fig. 1: Factors influencing density and sound velocity measurements

Gas bubbles in the liquid

For a density sensor, if gas bubbles are present in the liquid, both the mass and the volume measured in the tube will change (Fig. 1C). The density of the liquid-gas mixture is measured. As the gas has a much lower density, just a few gas bubbles will suffice to cause very large measuring errors when determining the density of the liquid.

The impact on the measurement of sound velocity will be much lower. On the one hand, only those gas bubbles which are located directly between the transmitter and the receiver will influence the measurement (Fig. 1D).

On the other hand, the ultrasonic waves will be „diffracted" around the gas bubbles or reflected (Fig. 2). This means that, for small gas fractions, the correct sound velocity of the liquid will be measured (the sound does not pass through the gas bubbles, but travels around the gas bubbles without any delay). If too many gas bubbles are present in the liquid, the ultrasound will be strongly reflected, and less sound will reach the receiver. By monitoring the amplitude of the signal received, this may be interpreted as an error.

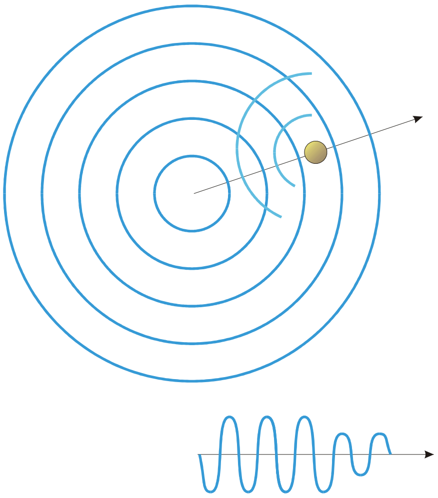


Fig. 2: Diffraction of ultrasonic waves

Special material of oscillator tube or sound transducers

If the liquids to be measured are aggressive, it is often necessary to use special materials such as Hastelloy, tantalum or titanium.

There are two disadvantages, if the oscillator tube of the density sensor is made of special materials:

1. Due to the higher material density of the tube (Hastelloy, tantalum), the influence exerted by the tube mass will be greater and, consequently, the accuracy of the density measurement will be lower.
2. The expansion properties of special materials such as Hastelloy or tantalum are inferior to those of titanium. The continual mechanical oscillations of the tube result in fatigue of the material.

For an ultrasonic sensor, the special material has no influence on the accuracy of the time measurement and is not subjected to mechanical loads. The material only serves for „holding" the ultrasound transducer.

Deposits

In some processes, the liquids tend to cause deposits and clogging.

Deposits on the tube wall of the density sensor lead to changes in the mass and volume and, thus, to erroneous measuring values. Moreover, the oscillation behaviour of the tube will change (spring constant). Even small deposited layers may cause major measuring errors.

Deposits forming on the ultrasound transducers will manifest themselves as a measuring error only in proportion to the total distance, because the sound velocity is measured over the entire distance between the transmitter and receiver. If, for example, the layer thickness amounts to 1 % of the transducer distance, the measuring error will be approximately 1 %.

Climatic influences

With cold liquids, the mass of the tube of a density sensor may change as a result of the formation of dew or condensate. The mass of the water droplets will be incorporated in the calculation and result in a seemingly higher density.

This may be prevented to a large extent by the use of a protective gas.

Climatic factors do not have any effect on ultrasound sensors. The ultrasound transducers usually are completely sealed in.

Mechanical vibrations and stresses

A density oscillator is a mechanical system oscillating at frequencies of a few hundred Hertz. If vibrations in the pipeline are caused by pumps or by switching valves, the vibrations will superimpose and lead to measuring errors (Fig. 1E). This will be particularly pronounced in the case of major pressure surges. If mechanical stresses (tensile and compressive forces) occur in the pipeline, they also influence the vibration behaviour.

To reduce the impact of vibrations and stresses, manufacturers demand that connections be firmly supported or clamped on both ends. They are to prevent the transmission of vibrations and stresses to the measuring instrument.

Ultrasonic instruments operate in the Megahertz range. Mechanical vibrations, which usually are in the range from 1 to 5 kHz, do not have any influence on the time measurement. The same is true of mechanical stresses.

Flow rate of liquid

The flow rate does not have any influence on the density measurement.

Neither does the flow rate have any influence on the measurement of the sound velocity, because it is measured perpendicularly to the direction of flow. (Contrary to ultrasound flow meters where measurement is performed obliquely to the direction of flow.)

Temperature variations

Density sensors normally are temperature compensated devices. This means that the tube temperature is measured and the temperature-dependent change of the spring constant as well as the volume change are incorporated in the calculation. Temperature compensation is effective in case of slow temperature variations.

The temperature is also measured for ultrasonic instruments. The temperature-dependent change of the distance between the transmitter and receiver is compensated for.

Pressure variations

With densimeters, pressure variations will result in a change of the force acting on the measuring tube. The influence differs in dependence on the design (tube thickness, clamping). Accuracies of densimeters are usually specified only for a pressure of 1 bar.

Pressure variations of up to 50 bar do not have any effect on ultrasonic devices. The force produced will not cause any relevant change in the distance between transmitter and receiver.

Operating conditions

Installation

Bending oscillators are mostly installed in a bypass. The liquid is passed through the sensor via orifice systems (pressure differences) or by means of pumps. A bypass system using a pump offers the advantage that the „current“ medium is passed through the density sensor and that the temperature is better compensated for.

Coriolis force sensors are installed in a bypass and in the main line. They must be fixedly installed so that the transmission of mechanical vibrations will be prevented. As the sensors will be quite big (up to 2000 mm) and heavy (up to 300 kg) for nominal diameters greater than DN25, special measures will have to be taken in this respect.

In the installation, the total costs must be taken into account (cost of ownership). Especially for large nominal diameters, components of the bypass system such as pumps and valves play a decisive part.

Fig. 3: Density measurement system LiquiDens

No requirements are specified for the installation of ultrasound sensors. They are usually installed in pipelines or vessels without any specific mounting. The installation length is usually quite short (30 mm), and the weight ranges between 4 and 8 kg, depending on the type (Fig. 4).

Fig. 4: Ultrasound measurement system LiquiSonic

	Density		Sound velocity
	Bending oscillator	**Coriolis force sensor, mass flow sensor**	**Ultrasound sensor**
Concentration characteristic / calibration curve	Specific density of many liquids is well documented		Specific sound velocity is little documented
Calibration liquid	Reference liquid (reference density)		Water
Configuration	Bypass with ND 6..10	Inline, large and heavy	Inline, relatively light and robust
Installation	Bypass	Inline, fixed support points	No requirements
Installation in tank	Only via external bypass		Immersion probe
Tranquilizer installation	Required		Not required
Wear	Material fatigue depending on material (special material) because of mechanical oscillator system		No wear
Gas bubbles	Cause erroneous measuring values		Hardly influence measuring accuracy, error message
Deposits	Influence vibration behaviour		Have an impact in proportion to total measuring length
Pipeline vibrations	Influence measuring accuracy (operating frequency: a few hundred Hz)		Do not influence measuring accuracy (operating frequency: 1 MHz)
Pressure surges	Influence measuring accuracy		Do not influence measuring accuracy
Special material (Hastelloy, tantalum, monel)	Influence measuring accuracy		Do not influence measuring accuracy
Pressure loss	Low, because of bypass	High	Low
Weight	2 to 4 kg	10 to 300 kg	2 to 6 kg
Installation length	Via bypass	500 to 2000 mm	30 mm

Table 1: Comparison of density and sound velocity sensors

Density – sound – concentration

The previous sections have dealt with the determination of density and sound velocity. Below, it will be considered how density and sound velocity change with the concentration of the liquid.

By concentration of a liquid, we understand the ratio of the masses or volumes of a component to the total mass or total volume of the liquid. Concentrations are indicated in ppm, g/l or m% and vol%.

$$c_1 = \frac{m_1}{m_g} = \frac{m_1}{m_1 + m_2 + K}$$

c_1 = *concentration of component*
m_1 = *mass of component*
m_g = *total mass*

For measuring concentrations, it must be known how density or sound velocity change with concentration. The greater the change the more accurate the concentration can be determined.

Density and concentration

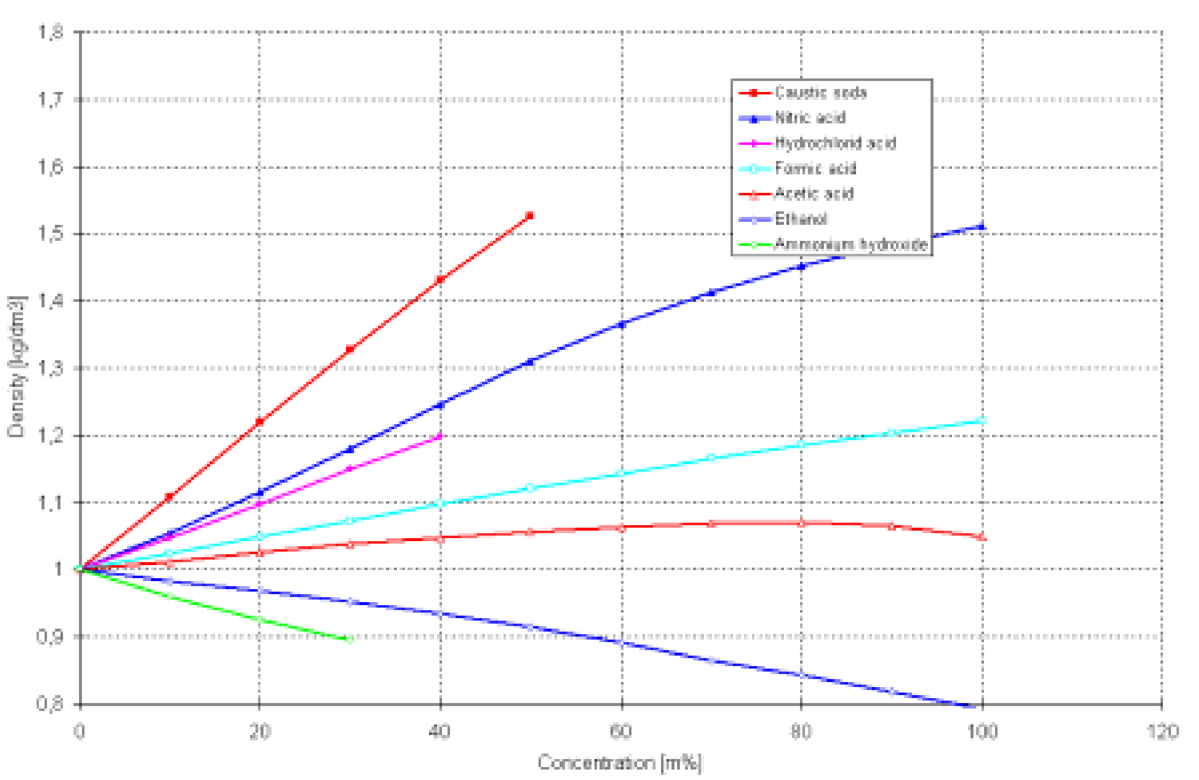


Fig. 5: Specific density of some liquids

Water has a density of approx. 1 kg/dm^3. With many liquids, density changes with a change in the concentration in the range from 0.8 to 1.8 kg/dm^3 .

The density of many liquids at different temperatures and the associated concentrations are documented in literature. Concentration curves or concentration characteristics can be relatively quickly obtained for the various liquids.

Sound velocity and concentration

The sound velocity is dependent on the density and adiabatic compressibility.

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

v	=	*sound velocity*
ρ	=	*density*
β_{ad}	=	*adiabatic compressibility*

The sound velocity of water at 20°C is 1,483 m/s. With many liquids, velocity changes with the change in concentration in the range from 1,200 m/s to 1,800 m/s.

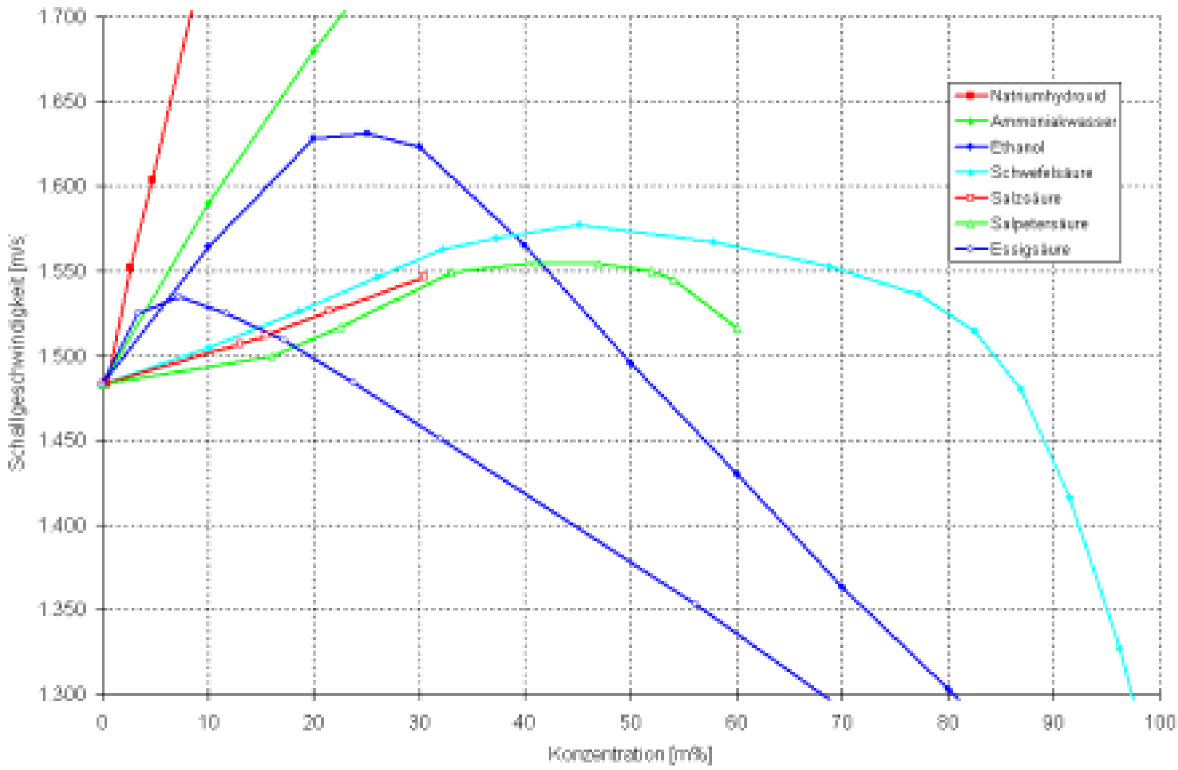


Fig. 6: Sound velocity of some liquids

The sound curves of water-soluble substances often are non-linear. This is due to the non-linearity of the temperature coefficient of the sound velocity of water (Fig. 6).

With all other liquids, the temperature coefficient is approximately linear, so that the concentration curves are also linear.

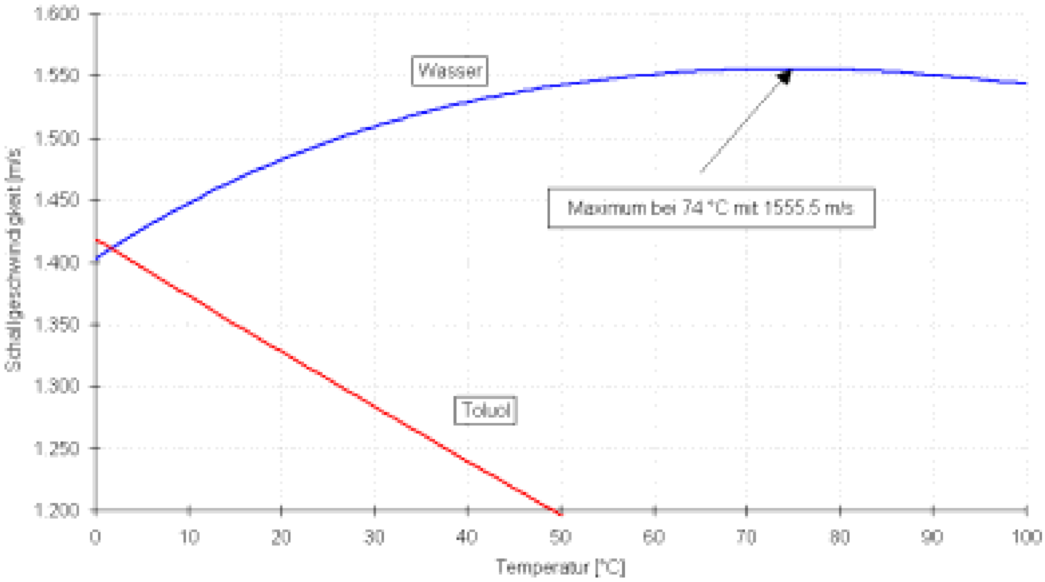


Fig. 7: Sound velocity of water and toluene

The sound velocity of the various liquids has been documented only little so far. This is primarily due to the fact that it has become possible only in recent years to determine sound velocity with a high accuracy. It is certainly only a question of time, however, till sound velocity, too, will have been comprehensively documented.

Achievable accuracy

The achievable accuracy of concentration measurements is dependent on the slope of the density or sound velocity curve. There are ranges where density will change more than sound velocity with concentration, and ranges where it is the other way round. The more quantization stages exist in the measuring range, the greater the degree to which concentration can be resolved.

If it is assumed that a densimeter allows for achieving an accuracy of 0.1 kg/m^3 and ultrasound measuring instruments an accuracy of 0.1 m/s, the following table of examples results:

	Principle	Change	Accuracy of instrument	Total no. of quantization stages in measuring range	Achievable accuracy of concentration
Ethanol 0 .. 20 m%	Density	-32 kg/m^3	0.1 kg/m^3	320	0.06 m%
	Sound	+145 m/s	0.1 m/s	1450	0.01 m%
Sulphuric acid 10 .. 20 m%	Density	+73 kg/m^3	0.1 kg/m^3	730	0.01 m%
	Sound	+16.5 m/s	0.1 m/s	165	0.06 m%
Sulphuric acid 90 .. 100 m%	Density	+16 kg/m^3	0.1 kg/m^3	160	0.06 m%
	Sound	-208.9 m/s	0.1 m/s	2089	0.005 m%

Table 2: Accuracy achievable for some liquids

Temperature dependence of physical quantities

Both, the density and sound velocity of a liquid are dependent on temperature.

With many liquids, density changes absolutely with a temperature coefficient of 2 kg/m^3 per °C. Depending on the liquid, sound velocity changes at a rate of 2 to 3 m/s per °C. In relative terms, density changes at 10^{-3} per °C and sound velocity at 1 to 2 10^{-3} per °C. This means that the temperature dependence of both quantities is approximately the same.

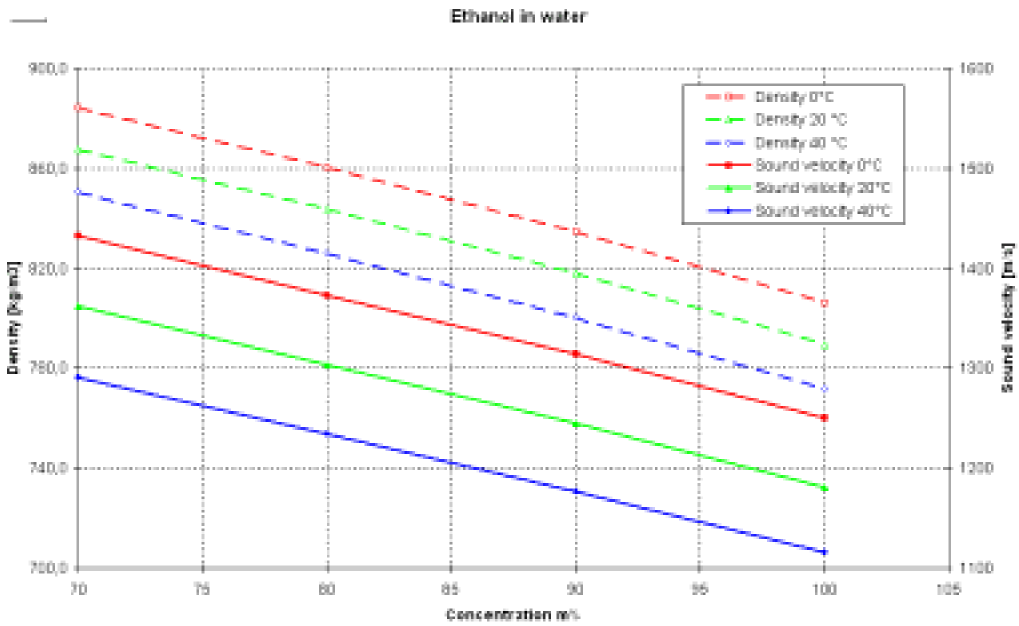


Fig. 8: Density and sound velocity of an ethanol-water mixture at different temperatures

What does this mean for practical applications?

1. If the density and sound velocity of a liquid are to be determined to within 0.1 kg/m^3 and 0.1 m/s, respectively, the temperature at the densimeter must be measured to within 0.1 °C and at the sound measuring instrument to within 0.05 °C.
2. If the concentration of a liquid is to be precisely determined, the temperature must be very precisely measured and compensated for in respect of the liquid concerned. Each liquid has its own temperature compensating function. This temperature compensation is non-linear over a greater concentration and temperature range.
3. Liquid-specific temperature compensation normally is performed using an efficient controller.

Note:
If a temperature-compensated sensor is used, this means that the impact of temperature on the measurement itself will be compensated for.

This compensation does not include any density or sound velocity compensation for the liquid, because it differs from liquid to liquid. Compensation is performed in the form of a liquid-specific calibration curve or characteristic.

Calibration and calibration liquids

Calibration liquid

For calibrating measuring instruments, calibration liquids are used. Various institutions offer density calibration liquids (DKD, NAMAS). They have an accuracy of 0.05 kg/m^3 and are offered together with a temperature correction formula.

Regrettably, there is no „density buffer“ such as a pH buffer solution. Density is dependent on temperature. As density changes at, e.g., 1 kg/m^3 per °C, temperature must be measured to within 0.05 °C during density calibration.

As concerns sound velocity, calibration can be easily performed using distilled water. Various sound velocities can be derived for calibration purposes from the „water curve“.

Temperature	Sound velocity	Temperature coefficient	Required accuracy of temperature measurement
0 °C	1402.74 m/s	4.97 m/s per °C	002 °C
10 °C	1447.59 m/s	4.01 m/s per °C	0.02 °C
20 °C	1482.66 m/s	3.11 m/s per °C	0.03 °C
50 °C	1542.87 m/s	1.13 m/s per °C	0.09 °C
74 °C	1555.47 m/s	0.03 m/s per °C	3.33 °C

Table 3: Sound velocity of water

Instrument calibration

Normally, the liquids whose concentration is to be determined in a process analysis via the density or sound velocity, are not very pure binary liquids.

Other components present in the liquid have an influence on the density and sound velocity.

This influence can be compensated for only by a medium-specific calibration.

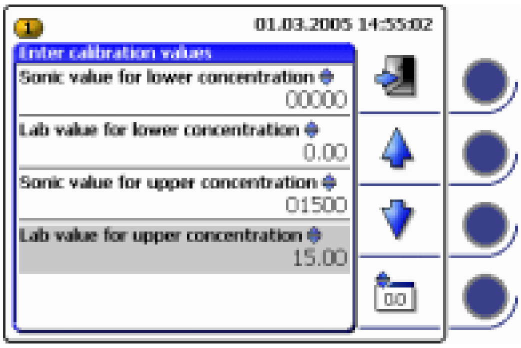


Fig. 9: Calibration dialogue

Combination of measuring methods

Using a combination of measuring methods is an appropriate way for determining the concentration of media consisting of several components.

For example, the concentration of two components in a solvent is to be determined. For a large number of liquids, the changes in the concentration of individual components have a different impact on the physical quantities to be measured.

One interesting application is the in-line measurement of alcohol and extract (wort) in beer. With increasing alcohol content, density decreases and the sound velocity increases. Increasing the extract content will increase density and the sound velocity.

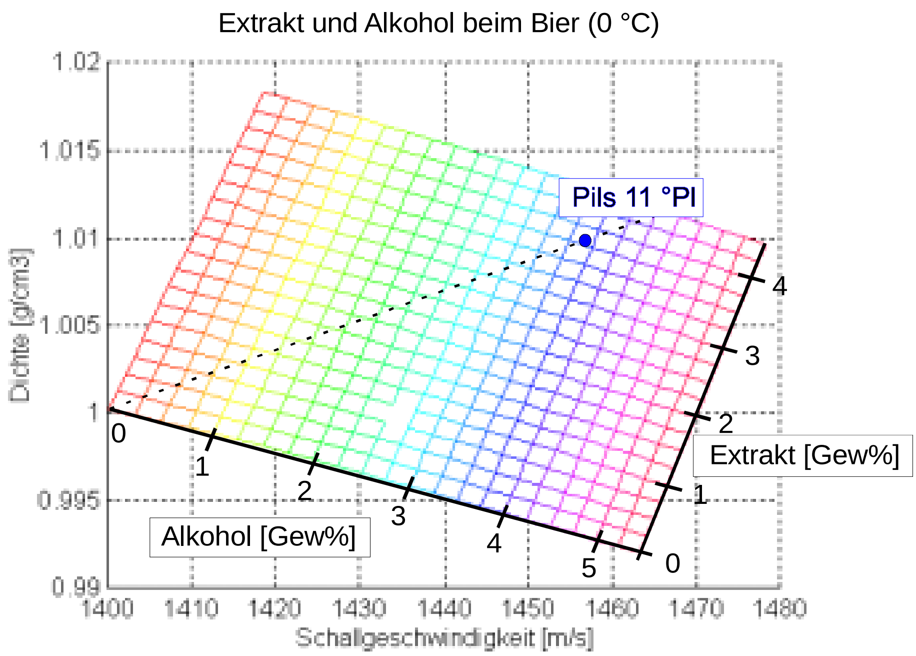


Fig. 10: Change in density and sound velocity with changing concentration of extract and alcohol

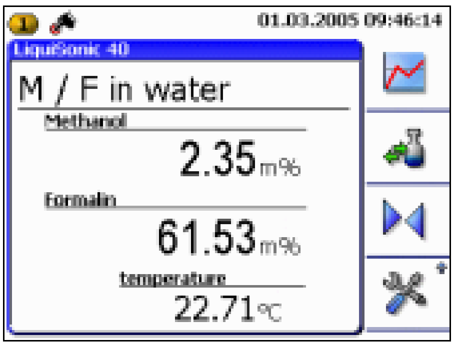


Fig. 11: Main view of multi-component measuring instrument.

SensoTech

For almost 20 years **SensoTech** has been primarily focused on the development, production, marketing. and support of high-performance in-line analyzers for concentration, density, or the monitoring of complex chemical reactions in liquid systems. During this time, **SensoTech** has an overall installation base of over 2000 devices worldwide. The unique products offer optimized and cost saving solutions for virtually every kind of application and process.

Providing solid solutions to complex problems has been both the challenge and the cornerstone of the **SensoTech** business. With the global installations and an extensive range of innovative products, **SensoTech** offers affordable, efficient solutions that meet our clients' exacting needs in food and beverage production and in chemical, pharmaceutical, biotechnical, semiconductor, iron and steel industries.

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