A Gentle Method for Handling Chemically Aggressive Substances

Non-invasive ultrasonic concentration measurement for use in the production of high purity sulfuric acid

Translation of a Reprint from CHEMIE TECHNIK 8/2005 (Huethig Verlag, Germany)

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Measuring flow with ultrasonic techniques is a well established method in measurement and control engineering. Acoustical methods for concentration determination are, in contrast, still the exception – although the advantages of the convenient clamp-on technique for concentration measurement are obvious, especially with chemically aggresive media or in the production of high purity substances.

Sulfuric acid represents one of the most essential basic substances in the chemical industry. A historic milestone that paved the way towards large scale chemical production was the development of industrial synthesis methods. The synthesis of sulfuric acid plays a major role in the highly complex production network of the world's largest chemical production site, BASF AG in Ludwigshafen, Germany. During the production of highly concentrated sulfuric acid, reliable and continual concentration measurement is indispensable for process control and quality insurance.

In the past, process samples were analyzed in the laboratory. The concentration was determined using a combined measurement of density and sound velocity. Inline measurement of the electrical conductivity had proven unreliable. The *Fachzentrum Prozessanalysentechnik* at BASF AG then installed the PIOX® S Ultrasonic Measuring System produced by the Berlin based company FLEXIM (see Fig. 1). As a result, the acid concentration can now be measured continuously during the entire operating time of the facility. Random or regular spot sampling in the lab is now obsolete. A constant quality control and a time and material saving process management are now possible.

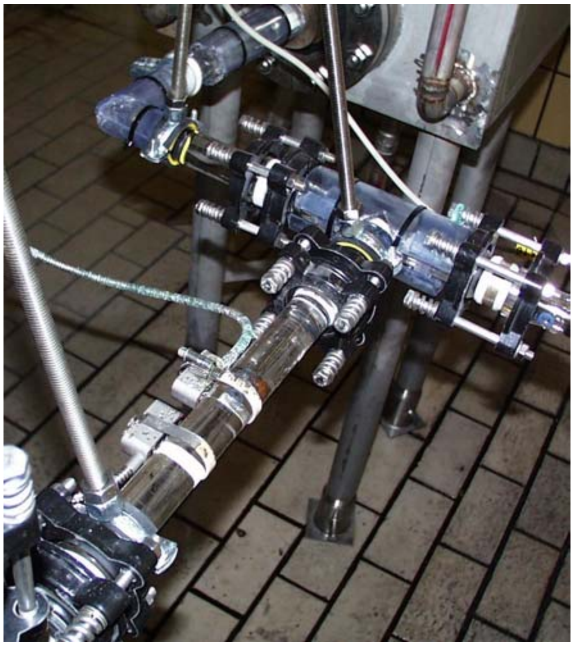

Fig. 1: Pair of transducers installed in the production process at BASF AG

Sound velocity as monitoring quantity

In measuring the concentration of a substance using ultrasound, the medium's sound velocity is determined by measuring the ultrasonic transit time (see Fig. 2). The sound velocity is a suitable characteristic product quantity for tracking all kinds of changes in liquid media. Unlike density, the sound velocities of many liquids have not been fully documented. Nevertheless, in many applications, sound velocity measurement is now a proven method. The concentration measurement of highly concentrated sulfuric acid is one of them.

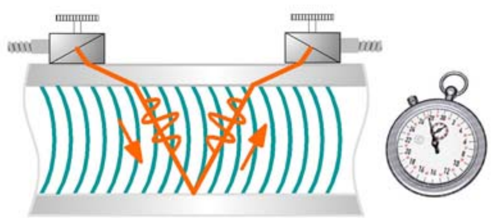

Fig. 2: Measuring principle - Determining the medium's sound velocity by measuring the transit time of ultrasound

The sound velocity and the concentration of a liquid medium are related via its density and adiabatic compressibility. In the most simple case, this dependence is linear. In the majority of cases, however, both sound velocity and density curves are non-linear. In order to measure the concentration, the characteristic concentration curves of the measured medium must first be determined, if they are not already known. In doing this, it must be taken into account that the sound velocity correlates with the temperature via the density of the medium. At the applications laboratory of the *Fachzentrum Prozessanalysentechnik*, the concentration characteristic curves of sulfuric acid have been determined using a calibrated clamp-on measuring rig. In order to calibrate the measuring rig, a documented reference medium was measured first. Afterward, the sound velocity of the medium to be measured in the production process was determined as a function of concentration and temperature.

Particularly in the case of high-percentage sulfuric acid (90-100%), sound velocity measurement is an outstanding method to determine concentration (see Fig. 3). It produces excellent results, whereas the density of the acid hardly changes any further in this concentration range. Thus, the sound velocity measurement in this range is 10 times more accurate than density measurements. Similar results are found in other concentration measurement applications, for example in alcoholic solutions.

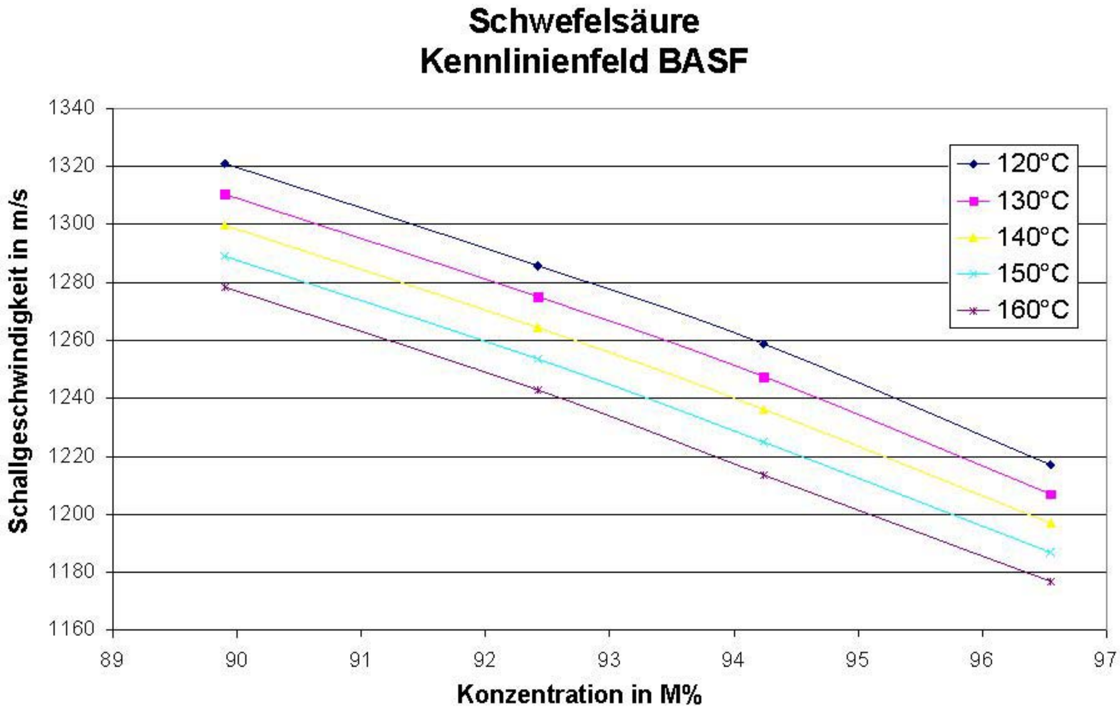

Fig. 3: Characteristic curve for the concentration dependence of sound velocity in sulfuric acid

Having determined the characteristic curves for the concentration, the measuring rig has to be calibrated. To do this, the deviation of the measured sound velocity from the documented reference values is recorded at different temperatures (see Fig. 4). The differences between measured and reference values result from uncertainties in determining the measuring point's physical parameters. Due to the exceptionally short transit time, even a minor uncertainty in measuring, for example, the thickness of the pipe's wall may result in a discrepancy between measured and documented values. This calibration was also done in the BASF applications laboratory prior to initial operation of the new measuring system (see Fig. 5). In this way, the otherwise necessary process calibration could be avoided.

All measured data - the characteristic diagram of concentration and the correction values - are saved in the transmitter's material data base for each specific measuring point. It is automatically taken into account during future measurements.

High process safety for ultra pure substances

The ultrasonic measuring system enables simultaneous measurement of flow and concentration. Since the measurement is done using the clamp-on technique, contaminating substances cannot enter. Consequently, this method is ideally suited for measuring products with high purity standards. Determining the concentrations of alcoholic and sugar solutions are typical applications in the food industry. In the chemical industry, ultrasonic methods are especially useful for measuring aggressive compounds for which a change in concentration is correlated with a significant change in sound velocity. This applies to, for example, highly concentrated sulfuric acid, but also to sodium hydroxide, liquid ammonia, acetic acid and highly concentrated hydrofluoric acid.

Using ultrasonic concentration measurement, BASF´s high purity sulfuric acid production facility has implemented a closed control loop for quality control. The risks and costs of product sampling have been eliminated. The process can be managed continuously and optimally.

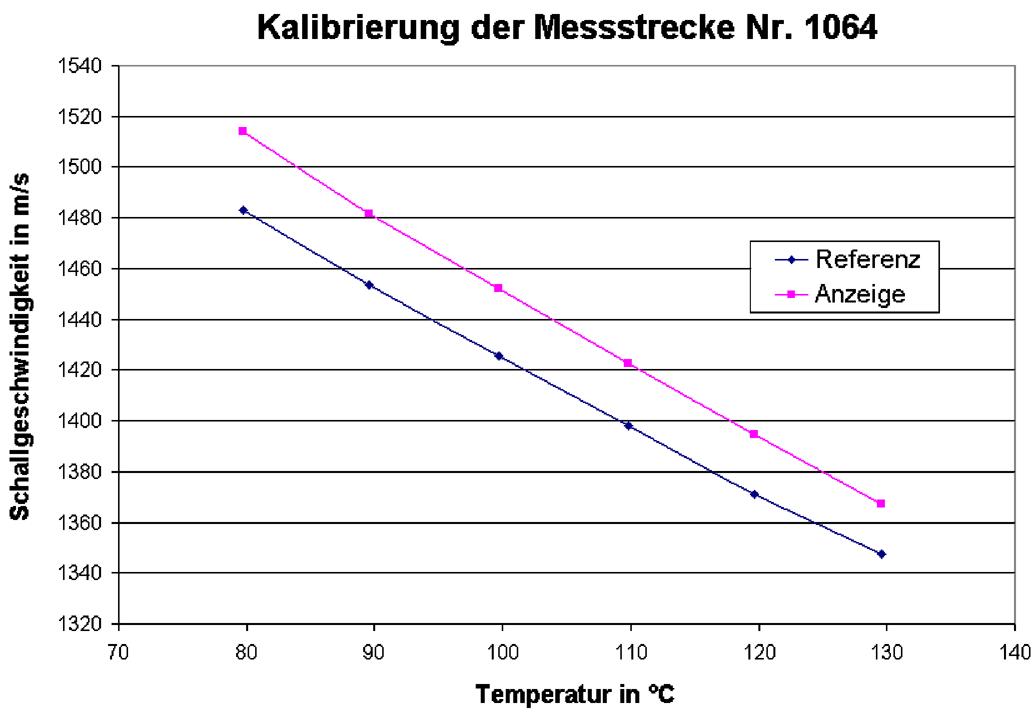

Fig. 4: Calibration of the measuring rig

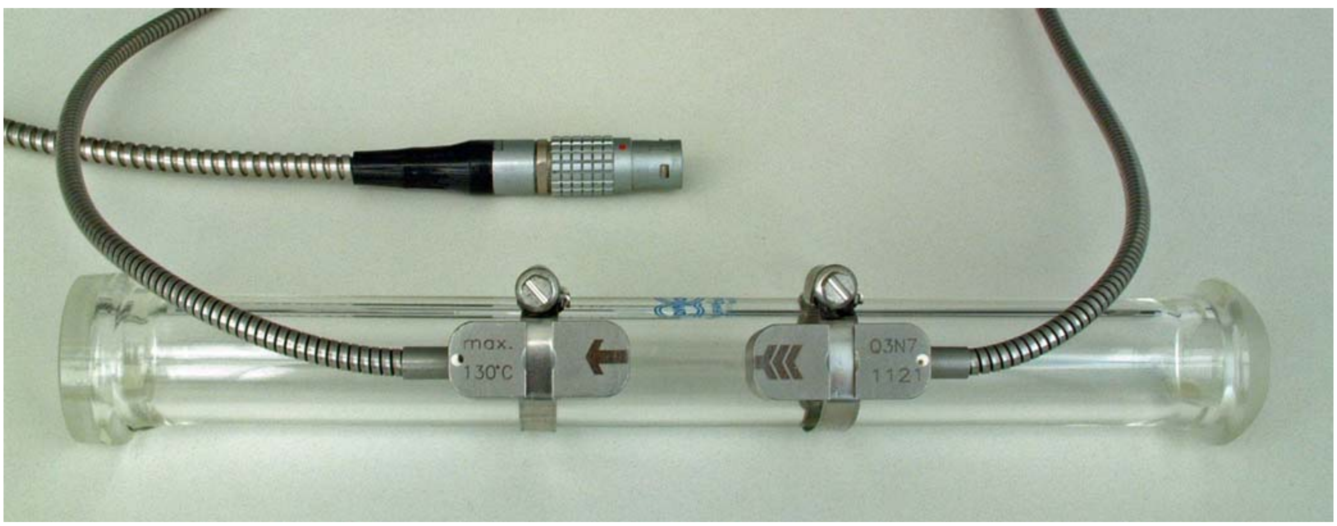

Fig. 5: Part of the measuring rig in the applications laboratory at BASF AG

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Ultrasonic Technology for Concentration Measurement

The ultrasonic measuring system enables simultaneous measurement of flow and concentration. The measurement is done using the clamp-on technique. The transducers are attached to the outside of the pipe and do not come in contact with the medium (see Fig. 1). Because the sound velocity depends on the medium's temperature, this temperature is determined at the measuring point by a Pt 1000 probe. Using the previously acquired data, the transmitter calculates the temperature compensated sound velocity and the resulting concentration. Since the measurement is done using the clamp-on technique, contaminating substances cannot enter.