A precision thermal mass flow sensor for (very) small liquid flows.

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Introduction.

Measurement and control of small liquid flows is a delicate matter. Most conventional methods make use of moving parts in the flow. This disturbs the continuity of small flow ranges. The flow meter series LIQUI-FLOW® (see the picture) discussed here, measures and controls flows from 1000 g/h down to 0.25 g/h full scale stabile and continuous. The instruments, based on a thermal measuring principle, are rather new. Many applications of the instruments can be found in R&D laboratories in the chemical and pharmaceutical field, as well as chemical pilot plants and in research projects of fuel cells and catalysers for combustion.

The Bronkhorst High-Tech LIQUI-FLOW® controller.

Thermal flow metering: theory.

The measuring principle of a thermal mass flow meter is explained in Figure 1.
The flow is lead through a tube, sketched at the top of the picture. On this tube, three sensor elements are placed. A heater in the middle, and temperature sensors upstream T_{up}, and downstream T_{down}, of the heater. In the lower part of this figure, the temperature profile along the sensor tube is sketched. When there is no flow through the pipe, this temperature profile is symmetric around the heater. When fluid is flowing through the pipe from left to right, the temperature profile will shift to the right. The shift in temperature profile represents a temperature difference ΔT, between T_{up} and T_{down}.
The shift of the temperature profile and the temperature difference ΔT, is a result of the heat transport of the flowing fluid. Heat transport is proportional with mass flow and heat capacity.
So this thermal flow sensor measures the <u>mass</u> flow of the fluid.
The temperature difference is transformed into an electrical output signal.

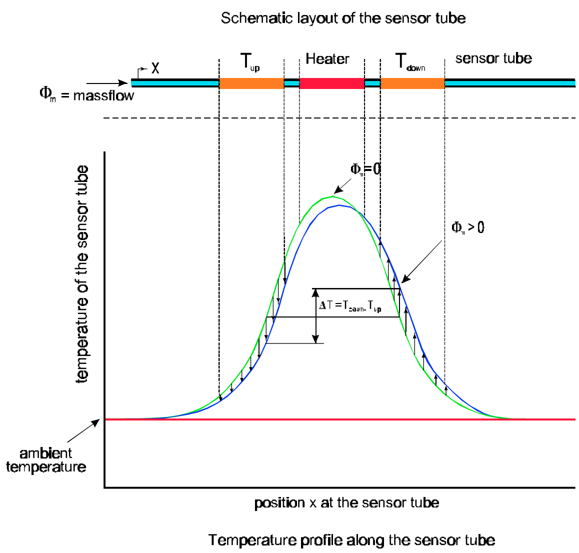


Figure 1: A tube-based thermal mass flow sensor.

Practical realization.

The measuring principle described in the paragraph above, can be applied to gases as well as liquids. In Figure 2, a schematic cross-section of the LIQUI-FLOW®, a flow meter for liquids is given. The same theory as described above can be applied.
The sensor tube is bent in a U-shape. In the middle of the tube, a heater is placed. The heater temperature is kept constant at some degrees above the base (housing), by means of two Pt-100 temperature sensors and an electronically controlled heater circuit. The heater temperature of the flow meter is held at approx. 5 °C above ambient.

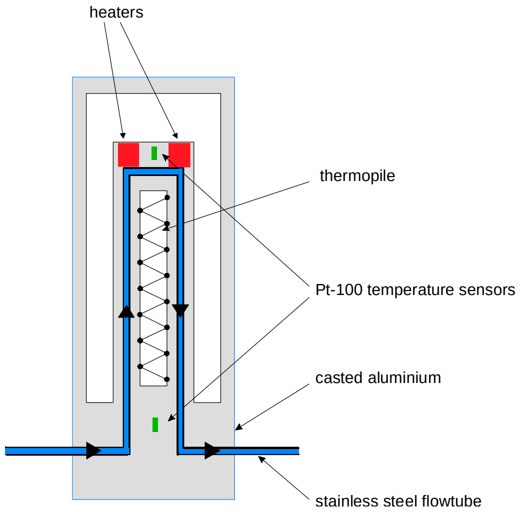


Figure 2: A schematic cross-section of the LIQUI-FLOW®

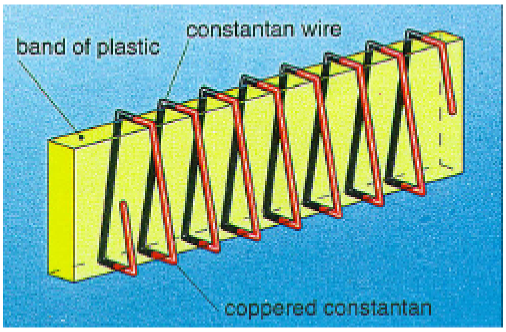
A ΔT sensor assembly, in stead of two individual temperature sensors, measures the temperature difference ΔT, between T_{up} and T_{down}. The ΔT sensor assembly is a thermopile, that consists of over 5000 individual Copper-Constantane thermocouples.
See "The thermopile" in frame.

The thermal signal, ΔT is only some tenths of degrees. With the thermopile sensor, this very low thermal signal is amplified to an electrical signal of approximately 20 mV.
The flow tube, which consists of Stainless Steel, is cast into Aluminum. The housing is cast in the same process and serves as an electronical and thermal shield for the sensor. In the housing, also the printed circuit board for the electronics is placed. The circuit has different functions, a.o. amplification and linearization of the thermopile signal, temperature control of the heater and the electronical circuit for flow control with a control valve.

This design leads to a very accurate and stable mass flow meter especially suited for small flow ranges.

The thermopile.

The thermopile applied in the LIQUI-FLOW® contains Copper-Constantane thermocouples.
This combination gives a Seebeck coefficient of 42 uV per °C.
In a special technique, over 5000 thermocouples are gathered in one sensor. The basic material is thermopile tape (see Figure). A very thin Constantane wire is wound (4 windings per mm) around the tape (2.4 mm wide). One half of the tape is covered (Electrochemically) with a thick layer of copper. In this way, Copper-Constantane junctions are made. A length of 150 cm of thermopile tape is wounded in a sensor. So a thermopile sensor contains almost 5000 junctions.



A cross section of the thermopile tape

Comparing this flow meter with other instruments suitable for flow metering in low flow ranges, gives the following result:
Most flow metering principles make use of moving parts in the flow. These moving parts for instance in the turbine flow meter or in a positive displacement flow meter induce instability in the flow at low flow ranges and are sensitive to clogging and aggressive fluids.
The electronics of the liquid flow sensor is integrated in the housing, the electrical output signal can be used to monitor the flow by computer or plotter.
A very strong feature of the instrument is the fact that the control function is also integrated on the PC-board of the flow meter. This means that the flow meter very easily can be combined with a control valve, integrated in the body, to form a liquid flow controller. No external PID controller is needed.
The sensor always needs power; some hundreds mWatts.

Most other types of flow meters in these low flow ranges, measure volume flow instead of mass flow, except flow sensors that make use of the Coriolis metering principle. These mass flow meters, however, are not suited for low flows.
Using scales in combination with a clock is a very accurate method of mass flow metering, but it is a non-continuous measurement, not on-line.
In fact, precision scales and an accurate clock both connected to a computer are used as the mass flow standard to calibrate the precision thermal liquid mass flow meters and controllers. This calibration method leads to an accuracy of 0.2 % rd.
The wetted parts of the flow meter are Stainless Steel. The sensor is very robust, fluid pressures up to 400 bar can be applied.

Applications.

The LIQUI-FLOW® was introduced in 1993, as a first instrument metering and controlling these very small flow rates of liquid. The instrument has since been very successfully applied in European companies like: BAYER, AGFA, DSM, POLYOLYFINE, DAIMLER BENZ, SIEMENS, DEGUSSA, HOECHST, AKZO, DOW CHEMICALS, BALZERS, DRäGER, MERCEDES BENZ, GENERAL MOTORS, BASF, FRAUNHOFER INSTITUTES, ECN, SHELL and TNO.
Most of the applications of the liquid flow meters and controllers for small flow rates, can be found in chemical research environment in laboratories and (process-) industry e.g. pilot plants for down scaling of chemical processes.

Thermal flow instruments are successful because of some characteristic properties.

- Very small flow rates can be metered and controlled precise and stable.
- The instrument has an electrical output signal.
- All wetted materials are Stainless Steel, and it has all-metal seals to the outside, the leak tightness is very high.
- The instrument is very robust, there is almost no limitation to the applied fluids or pressures.

Most of these specific features are discussed in typical examples of applications below.
Some main fields of application can be distinguished.

1. Catalysers.

Development and testing of Catalysers for chemical reactions.
The instruments that are used here are liquid flow controllers, applied often at high pressures. One ore more reactants are controlled in a test facility or pilot plant. The flow rates can be varied individually or in a master-slave mode.
The fluids applied are a.o. Ethylene, Propylene, Ethylene-Oxide Propylene-Oxide. The flow rates used are up to 500 g/h, the leak tightness is very important, because the fluids are flammable or toxic.
Another category of Catalysers that is tested with LIQUI-FLOW® is Catalysers for combustion gases from automobiles and burners for central heating systems. See also: Vapor control.

2. Process control.

The liquid flow controller is very successfully applied in Process control. Liquids can be dosed very accurate, stable and repeatable. For controlling blends of fluids, a master slave option can be useful.
Additives for smell- and taste components are dosed with the flow meters. In these applications the capability of dosing very small flow rates is an important feature. Flow rates down to 0.1 g/h can be dosed.

Example:
Dosing of Nicotine for Nicotine plasters. The liquid flow controller, the µ-flow type, is used here because of three main reasons, the high accuracy and reproducibility, the control function of the instrument and the good leak tightness of the metal seals of the flow meter, Nicotine is a very poisonous liquid!
The flow controller controls a flow rate from 0 to 250 milligram per hour.
The liquid flow meter is often used to replace batch-blending processes by inline blending.
The instrument is suited for this because of the possibility to control small flow rates very accurately, if necessary in a master slave mode.

Example:
Ultra pure water is dosed at a flow rate of 100 g/h in a process where long term stability and reproducibility are very important.

Example:
A pilot plant for hydration of Propylene for the production of poly-styrene at pressure 20 bar, flow rates from 5 g/h to 2 kg/h liquid Propylene are controlled with the liquid flow controller.

3. Fuel cells.

Research of fuel cells and up-scaling of the fuel cell modules.
The liquids that are used in these applications are mainly Methanol and Water.
Methanol is the fuel for the fuel cell. The water is controlled and subsequently evaporated and used to humidify H2. (see also: Vapor control).
The goal of the tests is up-scaling the fuel cell and test of long term performance.
The flows applied vary from very small, 10 g/h to high, 10 kg/h Methanol.
See Figure 3.

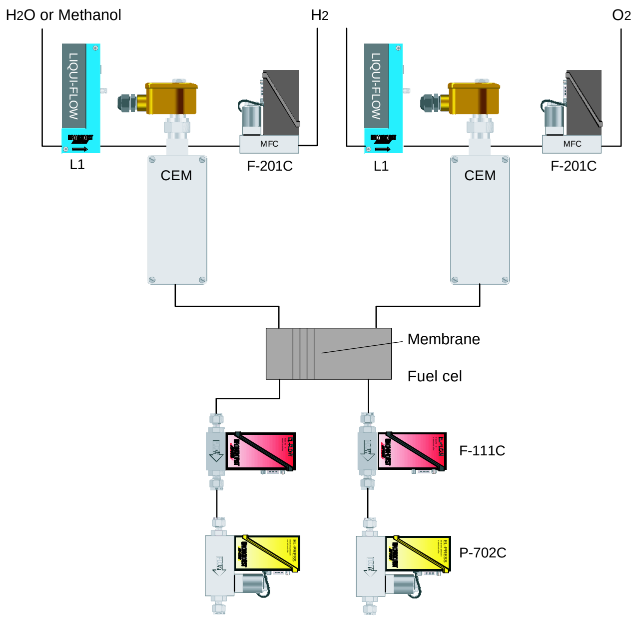


Figure 3: Liquid, gas delivery for a fuel cell.

4. Vapor control.

In many applications in research or production projects, the liquid is needed in vapor phase.
For these applications, the liquid flow controller can be combined with an evaporation device (CEM) [1].
See Figure 4: CEM. The liquid, which is controlled with the liquid flow controller, is mixed with a precisely controlled amount of gas (MFC), and subsequently evaporated.
The systems are used for generating mixtures with very small, very precise concentrations of vapor.
These test gases can be used for calibrating gas chromatographs or mass spectrometers.

Example:
A mass spectrometer can be calibrated with Acetic Acid before use with a 50 g/h liquid flow controller.
Test gases are also used for environmental research. Small concentrations of Toluene or Xylene in air are generated for testing sensors for toxic gases.

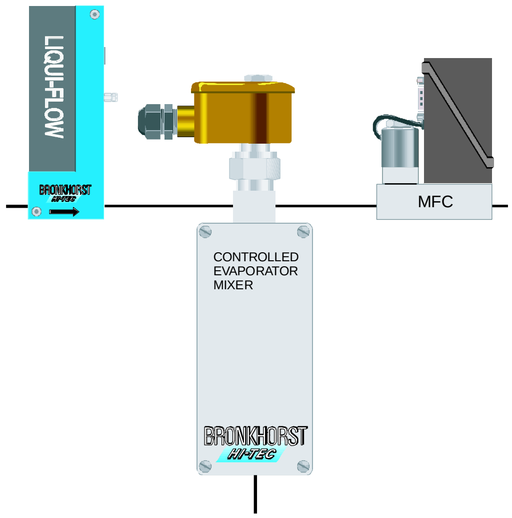


Figure 4: CEM; Controlled, Evaporation and Mixing system.

Example:
Generation of test gases for testing carbon filters with toxic gases for gas masks.
The CEM system is used in many cases to humidify gases, e.g. generation of a certain degree of relative humidity, for example in air.

Example:
Generating artificial combustion gases for testing Catalysers for cars. Water vapor, from the CEM system is mixed with N_2, CO_2, NO, etc. In this way the combustion gases of the motor can be simulated.

Example:
Humidifying H_2 for fuel cells. (See Figure 3)

5. Quality control.

Calibration of HPLC pumps. The thermal mass flow meter is especially well suited for calibrating HPLC pumps, because of the high accuracy at these low flow rates. The high pressure applied in HPLC pumps is no problem for a LIQUI-FLOW®.
The liquid flow controller is often used as a replacement of the HPLC pump. The flow controller is very well suited to control the liquid at high pressures at a very constant flow rate.
The liquid flow meter can also be combined with a pump, in stead of a valve for a flow control unit. The PID controller on the p.c. board of the flow meter is used to control the flow.
For the calibration of infusion pumps these liquid flow instruments are used. A certain test procedure for the pump, controlled by a computer, is run. The test runs overnight. The data from the electronic output of the Liquid flow meter are collected and a calibration certificate is printed at the end of the cycle.

6. Liquefied gases and supercritical fluids.

The liquid flow controller is well suited for metering and controlling liquefied gases thanks to the high pressure rating that can be applied, and the leak tightness.
Sealing material is often a problem for liquefied gases and especially for supercritical fluids.
The properties of supercritical water for instance are very special, many elastomers dissolve in supercritical water. For liquefied CO_2, Viton acts like a sponge. The sealing material of the flow controller is all-metal, so these problems do not occur.

Example:
At the FRAUNHOFER research institute in Karlsruhe, Germany, at 400 bar, room temperature, 500 to 1000 g/h CO2 (and other fluids) are dosed in a supercritical reaction test.

Conclusion.

In the flow sensor a unique thermopile configuration is applied. Thanks to this very sensitive ΔT sensor, a very low hot spot temperature in the flow results in a very stable output signal.
The most important properties of the instrument are high accuracy and stability at relatively low flows, the instrument is all metal sealed, very robust, so high pressures can be applied. The instrument opens new fields of applications, and new possibilities, such as stable control of small vapor flows. This unique combination of properties leads to a broad field of applications and a wide variety in usage.
The thermal liquid flow controller is gaining a permanent place in modern R&D projects but also in process control.

[1] H.J. Boer. "Mass Flow Controlled Evaporation System", Journal De Physique IV, Colloque C5, supplement au Journal de Physique II, Volume 5, juin 1995. pp C5-961 - C5-966.

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