Feature

Liquid flow sensor for nano- and micro-flow ranges

Joost Lötters
R&D Manager, Bronkhorst High-Tech B.V., Nijverheidsstraat 1A, 7261 AK Ruurlo, The Netherlands

Abstract
Purpose – To present a new generation of liquid flow sensors that is capable of meeting the requirements as imposed by the life science, analysis, biotech and other markets.
Design/methodology/approach – A description of the design and development of low flow rate measuring system and typical applications.
Findings – The system described uses tubes made of silica, stainless steel or PEEK, and either constant power or constant temperature methods in conjunction with a heater and temperature sensor. The tested instruments were capable of measuring flow ranges between 25-500 nl/min (smallest flow range) and 100-2000 μl/min (largest flow range) water, with operating pressures up to 100 bar (up to 400 bar for flow meters with flow ranges below 100 μl/min).
Originality/value – Presents information on a new generation of liquid flow sensors.

Keywords Liquid flow, Sensors, Measurement

Paper type General review

Accurate measurement and control of tiny liquid flows of the order of nanolitres through millilitres per minute is becoming more and more important for a lot of applications in the life science, analysis (e.g. HPLC), biotech, synthesis (of e.g. pharmaceuticals) and nanotechnology markets. Accompanying demands to flow sensors suited for this low flow range are an extremely small internal volume and the use of for instance PEEK or fused silica as wetted material for the flow sensor tube as alternative to stainless steel. Furthermore the instruments should have a modular set-up, so they can be easily exchanged and adapted to a new need.

For example, the separation column at the detector side of analytical equipment is sometimes made of the material fused silica with an internal diameter typically of the order of 100 μm. In some cases, it is necessary to measure the – very small – flow at the detector side to improve the accuracy of the analysis. In this application, the internal diameter and wetted material of the flow sensor tube should preferably be the same as those of the separation column, to avoid disturbances in the flow and to minimise the internal volume.

Until recently, none of today's commercially available flow sensors were equipped with the above-mentioned features. In this paper, a new generation of liquid flow sensors is presented that is capable of meeting the requirements as imposed by the life science, analysis, biotech and other markets.

Sensor structure and basic operating principle

The actual flow sensor consists of a straight flow tube with two active elements around it, as shown in Figure 1(a) and (b). The wetted material of the flow tube is stainless steel, or as an option, fused silica or PEEK. The internal diameter of the flow tube may vary between 20 and 200 μm, depending on flow range. The corresponding internal volume of the mass flow meter is 1.5-20 μl.

Two measurement principles can be distinguished, namely, the constant power (CP) and the constant temperature (CT) method.

The CP measurement principle is used for the flow ranges below circa 100 μl/min. In this case, the two elements are used as both heater and as temperature sensor, as shown in Figure 1(a). Both elements are provided with an equal amount of constant power, the temperature difference (ΔT) between them is a measure for the flow.

The CT measurement principle is used for the flow ranges above circa 100 μl/min. In this case, the first element acts as temperature sensor, and the second element acts as a heater, as shown in Figure 1(b). The heater is heated to a certain constant temperature difference (ΔT) over the medium temperature. The heater power P_{heater} necessary to keep ΔT constant is a measure for the flow.

The above-mentioned flow sensor structures and operating principles have the following innovative features and advantages.

- The flow sensor comprises a short straight flow tube, with an internal diameter varying between 20 and 200 μm, thus having an extremely small internal volume, varying between 15 nl and 1.5 μl.

The Emerald Research Register for this journal is available at
www.emeraldinsight.com/researchregister

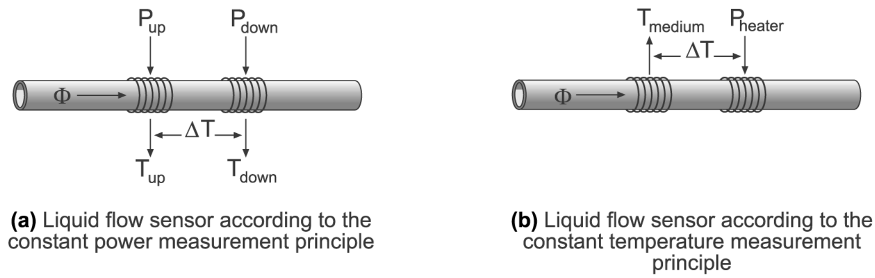
The current issue and full text archive of this journal is available at
www.emeraldinsight.com/0260-2288.htm

Sensor Review
25/1 (2005) 20–23

[ISSN 0260-2288]
[DOI 10.1108/02602280510577771]

Sensor Review
Volume 25 · Number 1 · 2005 · 20–23

Figure 1



- The smallest measurable flow range is 25-500 nl/min, the largest measurable flow range is 100-2,000 μl/min; the response time $t_{98\%}$ is of the order of 1 s.
- The measurable flow range can easily be adjusted, by varying the internal diameter, material and wall thickness of the flow tube and the measurement principle, so a wide flow range can be covered with the same type of instrument.
- The material of the flow tube can be either stainless steel, PEEK or fused silica; other materials may also prove to be feasible.
- The length of the flow sensor tube is the same for all flow ranges. This enables a modular set-up and exchangeability of the instruments.
- The active elements are placed outside the flow tube, so all wetted parts are either stainless steel, PEEK, or fused silica.

Electronic circuitry

Both measurement principles, CP and CT, need their own specific electronic circuitry, which is based upon a wheatstone bridge configuration. The electronic circuitry converts the output signals, namely ΔT for the CP and P_{heater} for the CT measurement principle, into an output voltage, showing a linear relation with the mass flow.

Flow control

Flow control is achieved by integrating a control valve onto the body of the liquid flow metre. This control valve has a purge connection on top of the sleeve that enables easy elimination of air or gas when starting up the system. The electronic control function forms part of the standard circuitry in the liquid flow metre, so the need for an external controller is eliminated.

Test procedure

For the experiments several liquid flow sensors were used, with flow tubes made out of stainless steel, PEEK and fused silica, and with internal diameters varying between 20 and 200 μm. Furthermore a liquid flow controller was used, comprising one of the sensors working according to the constant temperature measurement principle.

The output signal of the flow sensors was measured for flow ranges varying between 25-500 nl/min and 100-2,000 μl/min water. The mass flow controller was used for measurements of the dynamic behaviour. The following three stepwise variations in set-point were performed:

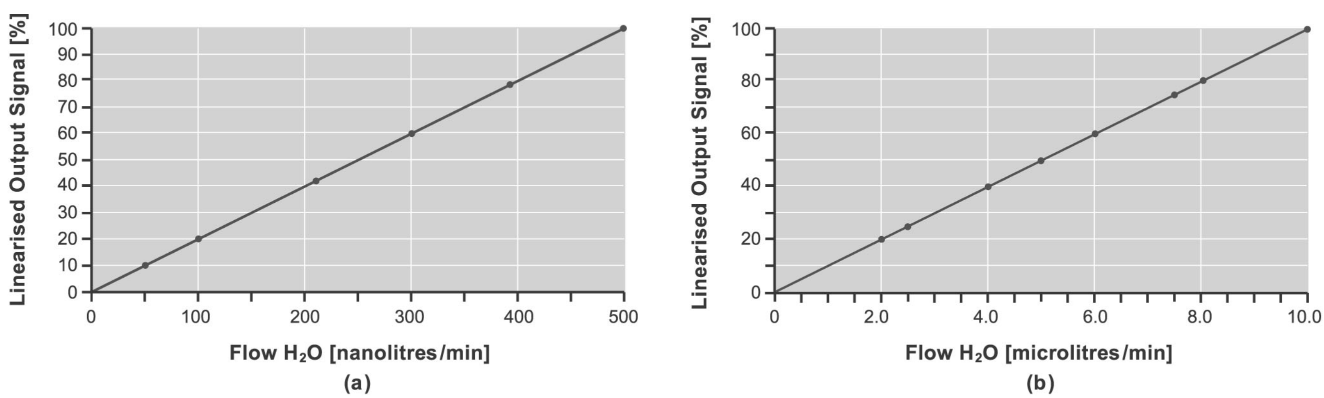
1. 0% ⇒ 100% ⇒ 0%;
2. 20% ⇒ 80% ⇒ 20%;
3. 20% ⇒ 40% ⇒ 60% ⇒ 80% ⇒ 100%.

A digital oscilloscope measured the resulting response of the mass flow sensor.

Test results

The measured output signals as a function of the mass flow are shown in Figures 2-4. The measured dynamic behaviour of the mass flow controller is shown in Figure 5.

Figure 2 Measurement results obtained with two liquid flow sensors for H_2O with flow range: (a) 500 nl/min=100%; and (b) 10 μl/min = 100% both working according to the CP measurement principle; the flow tubes are made of stainless steel



Sensor Review
Volume 25 · Number 1 · 2005 · 20–23

Figure 3 Measurement results obtained with two liquid flow sensors for H_2O both working according to the CP measurement principle with flow range 2,000 nl/min=100%; the flow tubes are made of: (a) fused silica; and (b) PEEK

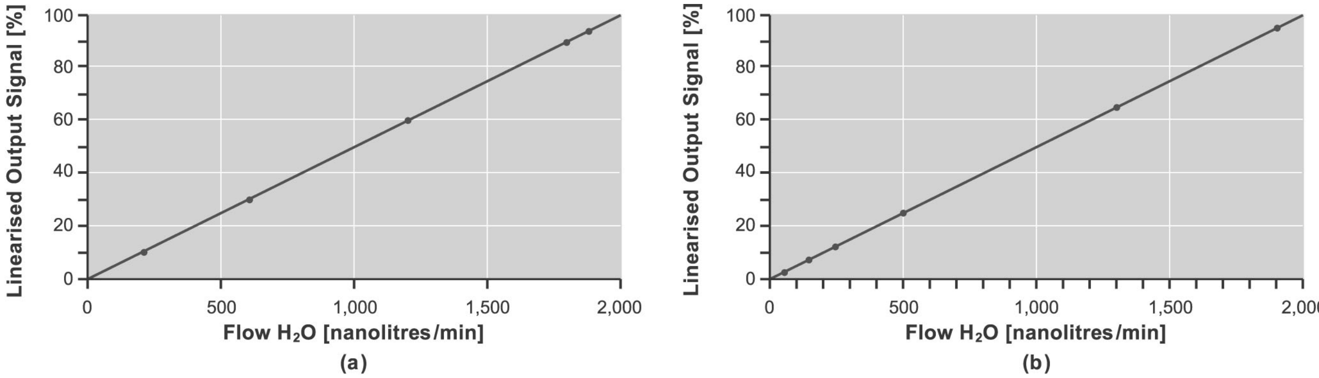
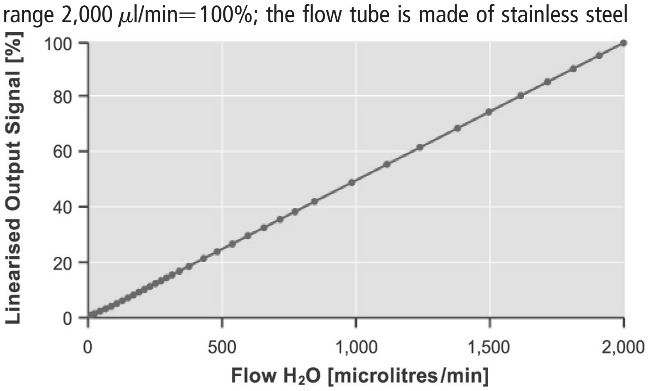


Figure 4 Measurement results obtained with a liquid flow sensor for H_2O working according to the CT measurement principle with flow range 2,000 μl/min=100%; the flow tube is made of stainless steel



The measured curves, as shown in Figures 2-4, correspond well with the theoretically expected values. The measured response times, as shown in Figure 5, are all within the value of $t_{98\%}=2$ s.

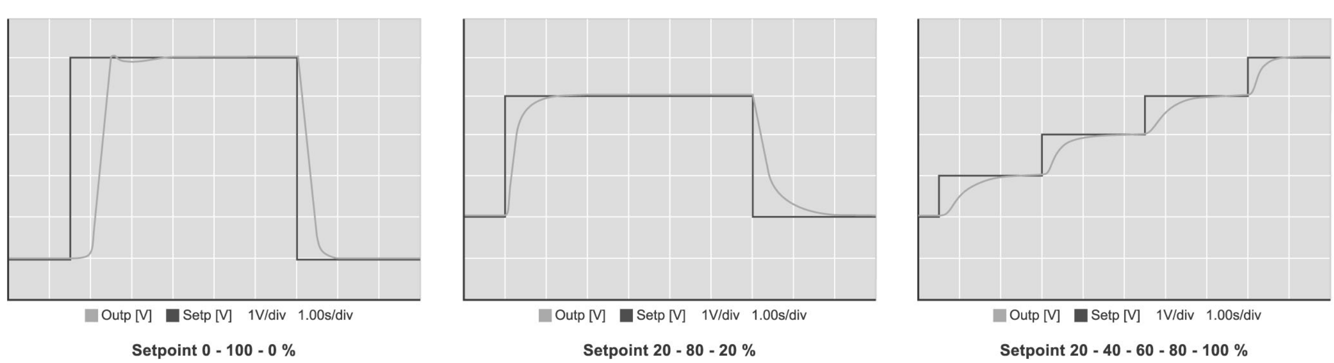
Conclusions

The new generation of liquid flow sensors presented in this paper is capable of meeting the requirements as imposed by the life science, analysis, biotech and other markets. The actual flow sensor consists of a straight flow tube with two active elements around it. The internal diameter of the flow tube may vary between 20 and 200 μm. The corresponding internal volume of the sensor tube is 15 nl and 1.5 μl, respectively, which is extremely small. Instruments with a flow tube made of stainless steel, fused silica, or PEEK, were tested successfully. The feasibility of other materials suitable for the above-mentioned markets will be further investigated. The flow sensors have been driven with two different measurement principles, namely the CP and the CT method. The CP measurement principle proved to be useful for flow ranges below circa 100 μl/min, the CT measurement principle was suited for flow ranges above circa 100 μl/min.

The tested instruments were capable of measuring flow ranges between 25...500 nl/min (smallest flow range) and 100...2,000 μl/min (largest flow range) water, with operating pressures up to 100 bar (up to 400 bar for flow metres with flow ranges below 100 μl/min).

The measured response time of the flow sensors is of the order of $t_{98\%} \cong 2$ s. The feasibility of the new measurement concept for low liquid flows has also been field-proven. Products based upon this new technology have been introduced on the market in 2003 and were found to be a reliable solution for many low liquid flow applications, for example in HPLC systems (Figures 6 and 7).

Figure 5 Measured response times of a liquid flow sensor adjusted for H_2O: 2,000 μl/min=5 V; the *X*-axis shows the time [s] with 1 s/div., the *Y*-axis displays the output voltage [V] with 1 V/div.; the flow tube is made of stainless steel; the sensor is working according to the CT measurement principle



Sensor Review
Volume 25 · Number 1 · 2005 · 20–23

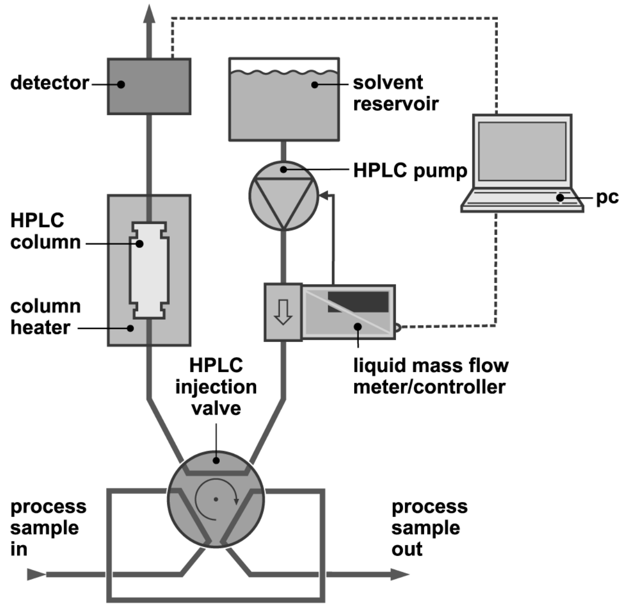


Figure 6 Example of application: liquid flow control in HPLC

Further reading

Bejan, A. (1993), *Heat Transfer*, Wiley, New York, NY.

Incropera, F.P. and DeWitt, D.P. (1996), *Fundamentals of Heat and Mass Transfer*, Wiley, New York, NY.

Figure 7 The Bronkhorst μ-flow metre and control unit