

Dosing system for the nanolitre range, fabricated with the AMANDA process

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Abstract

We present a dosing system, which consists of four microvalves (presented by Rogge *et al* in the *Proceedings of Eurosensors XVI*, Prague, 2002, pp 109–110) for controlling and a movable membrane for flow measurement that divides a cavity into two chambers. The flow rate is determined from the straining of the membrane caused by the entering fluid. The displaced volume is detected by strain gauges integrated in the membrane. The overall dimensions of the membrane and the entire system are 2 mm and $10 \times 50 \times 6 \text{ mm}^3$, respectively. A simple control was coded for dosing a certain amount of volume. This simple system has already achieved an accuracy of about 10%. The dosing system is manufactured with the AMANDA process, which allows for low-cost production of microcomponents from polymers.

1. Introduction

Dosing of liquids in the nanolitre range is required for biomedical applications, such as dosing of medicine or fabrication of very small amounts of substances for pharmaceutical testing. The maximum time of operation of a miniaturized dosing system is often limited by the smallest quantity of liquid which can be dosed with the required accuracy, because the concentration of a medicine inside a reservoir of limited space can be raised, if smaller amounts can be dosed.

Unfortunately, many working principles for flow measurement cannot be scaled down to very small dimensions, e.g. flow measurements with heated wires fail when heat diffusion is of the order of flow velocity and rotating wheels stop when friction forces are larger than the forces generated by the flow. Besides this, leakage may be a problem, if for example highly concentrated medicine is to be dosed, as even a very small leakage may severely harm the patient.

Therefore, we have been developing a microdosing system, which may detect leakages, which are too small to be measured directly.

Microcomponents as well as other products can only be useful to most people if they are affordable. This is why nearly all components are made of polymers using the AMANDA

process [1] for fabrication. This process is suitable for low-cost production of large quantities of microcomponents. Construction principles of polymer devices have to take into account large thermal expansion and creeping of the material.

2. Design and operation

The dosing system consists of five components, four piezo-driven microvalves [2], which control the liquid flow and a measurement unit (cf figure 1). The valves were already realized before [2] and are not optimized for this usage. The measurement unit contains the measurement chamber, which is divided into two parts by a polyimide membrane (chambers 1 and 2). Each chamber has its own input and output orifices. Two inlet valves (valves 1 and 3) are connected to the two inlets of the chambers and two outlet valves (valves 2 and 4) are connected to the two outlets of the chambers. The inlet of the system is led by one of the two input valves either to chamber 1 or 2, and by the outlet valves from the opposite chamber, chamber 2 or 1, respectively, to the outlet of the system.

The membrane consists of two polyimide layers with the same thickness ($1 \mu\text{m}$) encapsulating gold strain gauges (100 nm) with a chromium adhesion layer (5 nm). The strain gauges are separated from the liquid by the two polyimide

layers. Besides this, the position of the strain gauges at the neutral surface eliminates bending of the membrane affecting the output signal. The signal is a function of only the straining of the membrane and is symmetrical with respect to the direction of membrane deflection. Anyway, the strain on the outer surface of the membrane will not differ very much from that in its middle layer because the membrane is very thin compared to its diameter (of the order of millimetres) and typical deflections are large compared to its thickness. We placed two strain gauges into the membrane, both form a spiral. One spiral is situated at the centre while the other is arranged at the rim of the membrane (cf figure 5). The two spirals are used as branches in a full Wheatstone bridge, which is completed by external resistances.

The membrane is strained isotropically in its centre when it is deflected by the flow into one of the chambers of the sensor. The central spiral is strained nearly in the same way as the membrane, because it is much thinner than the membrane and, therefore, the central spiral becomes wider and longer by the same factor. This would result in no change of the resistance of the centre spiral, but at the same time it becomes thinner by Poisson's ratio times the strain in radial plus the strain in tangential direction. This causes an increase in the resistance of the central spiral when the membrane is deflected.

At the rim, the membrane is not strained tangentially but only in the radial direction because it is fixed at the border of the chamber. This results in an increase in the width of the spiral and a (smaller) reduction of its thickness. As a result, the resistance of the spiral situated at the rim is reduced when the membrane is deflected.

As both spirals form one branch of a Wheatstone bridge, the deflection of the membrane results in an increasing signal. A temperature change affects the resistance of both spirals in the same way and, therefore, should show no effect on the output signal of the bridge. For our first trials we based the design of the strain gauges on these considerations. For a real optimal design finite element calculations would be necessary which take into account the fact that the spirals affect the deflection of the membrane.

To operate the system a control unit is necessary, which monitors the output signal of the Wheatstone bridge and switches the valves before the membrane touches the wall of a chamber. The switching may be triggered by the output signal reaching a pre-defined threshold. The control unit also needs to calculate the amount of fluid flown through the system and to close all valves when the desired amount of liquid has been dosed. Alternatively the control unit could calculate the flow rate and adapt the opening of the valves to a desired flow.

When dosing starts, two microvalves are opened (valve 1 opens the inlet to chamber 2 and valve 4 opens chamber 1 to the outlet of the system) and the flow is directed into chamber 2 as shown in figure 1. The incoming liquid pushes against the membrane and the liquid is displaced from chamber 1 to the outlet of the system. When the membrane approaches the wall of one chamber, the two valves (valves 1 and 4) are closed, while the other two valves (valves 2 and 3) are opened. Then the flow is directed into chamber 1 and the direction of the movement of the membrane is reversed. After the microvalves

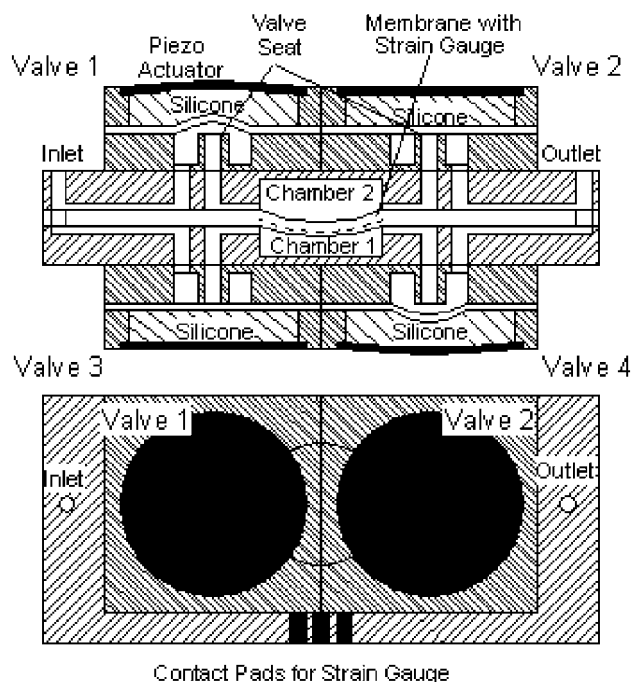


Figure 1. Schematic cross-section (top) and top view (bottom) of the microdosing system shown in figure 7.

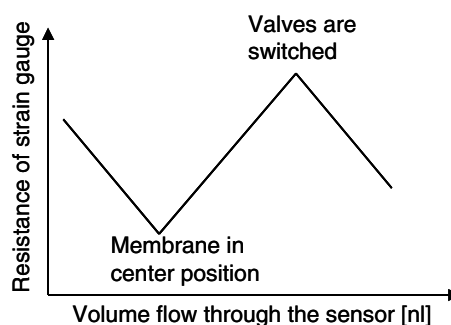


Figure 2. Schematic diagram of the theoretical resistance change of the strain gauge in the membrane.

are switched, a further fluid flow moves the membrane towards its flat position; the strain of the membrane is decreased and the output signal of the Wheatstone bridge is reduced. When the flat position of the membrane is passed, the resistance of the central strain gauge reaches a minimum and rises again (cf figure 2). Thus, it is possible to recalibrate the sensor signal when the centre position is passed. The strain gauges inside the membrane detect the membrane deflection and, thus, the displaced volume can be derived, i.e. by calculating the corresponding segment of a sphere.

In some applications the direction of flow may change. If the membrane is in the flat position, the dosing system is not able to recognize the flow direction. Therefore, it may be advantageous to include another strain gauge in the membrane, which is located at a position where, for example, chamber 1 does not extend to the edge of the membrane. Consequently, this strain gauge is only strained when the membrane is deflected towards chamber 2, and the system can recognize to which side of the measurement chamber the membrane is deflected.

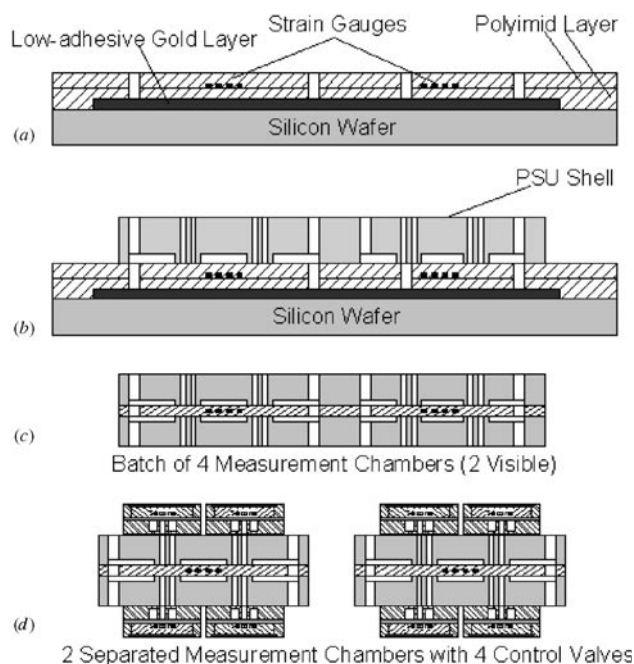


Figure 3. Schematic representation of the fabrication process of measurement cavities in batches and assembly with microvalves. Only two cavities are shown, although four are manufactured in a batch.

3. Fabrication process

The fabrication method for all components (four valves and the measurement unit) is known as the AMANDA process [1]. It is a batch process, a combination of molding, surface micromachining and membrane transfer, where several components, i.e. eight valves, are fabricated at the same time. All components consist of two polymer shells forming the upper and lower housings and a thin polymer membrane in between. The fabrication of the microvalves is published elsewhere [2]. They are equipped with a piezo actuator, which is coupled to the valve membrane with a flexible silicone gel. The gel transmits the small deflection of the piezo disc to a larger movement of the valve membrane of approximately $50\text{ }\mu\text{m}$. The diameter of the valve inlet is $200\text{ }\mu\text{m}$. The microvalves are fabricated separately and adhesively bonded to the measurement cavity. Here the manufacture of the measurement unit is described briefly.

The housings contain the measurement chambers and the channels, via which they are connected to the valves. They are fabricated from two shells made by hot embossing [3] of polysulfone (PSU). Four measurement units are manufactured in parallel on one batch (cf figure 4 right). Two measurement chambers with a diameter of 1 mm and two with 2 mm diameter are placed in one batch. The height of the chamber is $50\text{ }\mu\text{m}$ and $100\text{ }\mu\text{m}$, respectively. We designed units with different diameters, however all results presented here were achieved with a 2 mm measurement chamber.

A $1\text{ }\mu\text{m}$ thick polyimide (PI) layer is spin-coated on to a silicon wafer covered by a gold layer. Gold has low adhesion to silicon, which allows us to separate the finished membrane from the wafer easily. A 3 mm wide rim of the wafer is free of gold. There, the membrane sticks to the wafer during

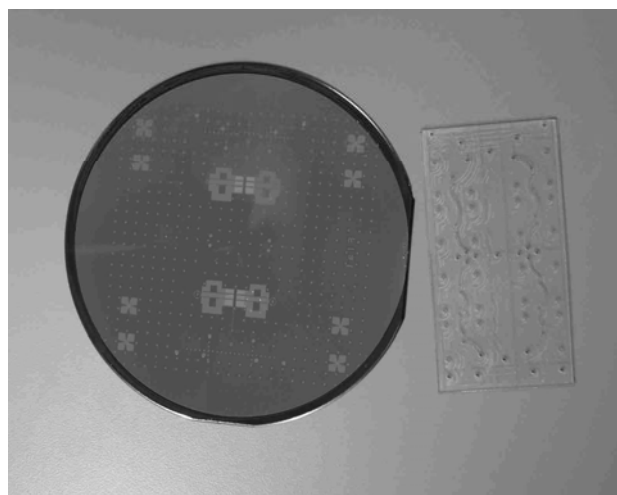


Figure 4. PI membrane on a silicon wafer ($\varnothing 100\text{ mm}$) (left) and hot-embossed PSU shell for the housings of four measurement units (right).

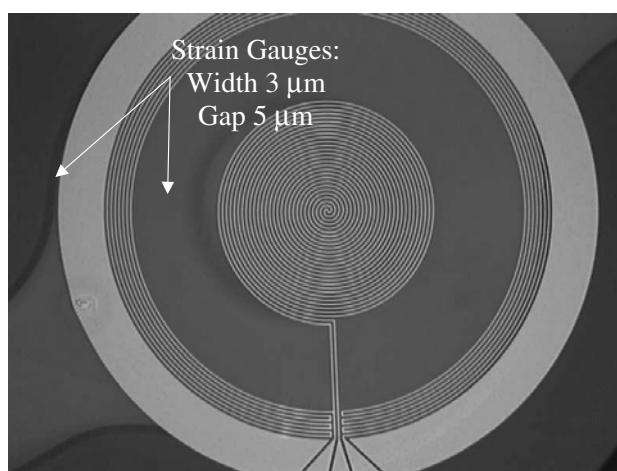


Figure 5. Strain gauges in the centre and at the circumference of the membrane.

the fabrication process (cf figure 3(a)) and this avoids the membrane coming off the wafer during processing. The PI is a photoresist (Durimide 7005 from Arch Chemicals) patterned by photolithography for generating all orifices required in the later PI membrane. A 100 nm thick gold layer is evaporated on to the PI and patterned by a lift-off process forming the strain gauges for the measurement of membrane displacement (cf figure 4 left). The spirals used as strain gauges are made of $3\text{ }\mu\text{m}$ wide gold paths with a gap width of $5\text{ }\mu\text{m}$ (figure 5).

One PSU shell is adhesively bonded to the PI by means of chamber adhesive bonding [4] (cf figure 3(b)) and the membrane is cut around the glued housing. The compound of the membrane and the PSU shell is separated from the substrate due to the low adhesion gold layer between the membrane and silicon wafer. The second PSU shell is adhesively bonded to the other side of the membrane in the same way (cf figure 3(c)).

The batch is diced with a saw and four valves (cf figure 6) are glued to each measurement chamber (cf figure 3(d)). Finally, a flat strip cable provides electrical contact to the strain

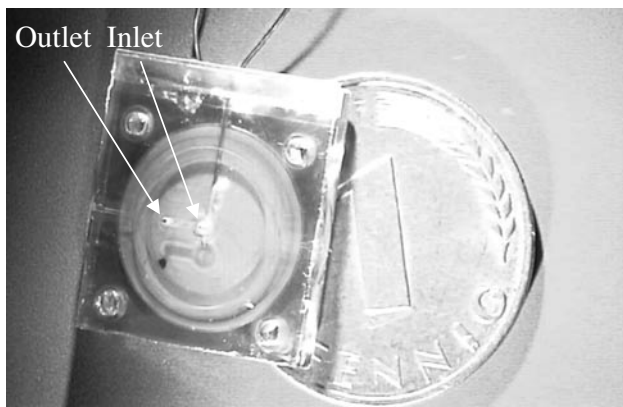


Figure 6. Controlling valve [2]. Rear with valve inlet and outlet, which is glued to the measurement unit. The overall dimensions are $16 \times 16 \times 3 \text{ mm}^3$.

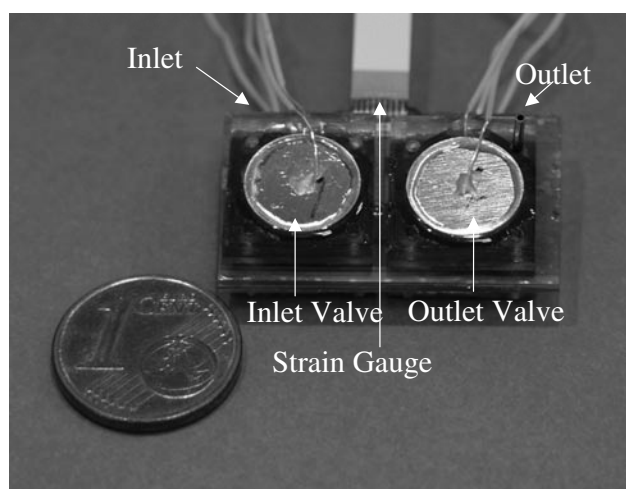


Figure 7. Complete dosing system.

gauges, and fluid connectors for inlet and outlet are mounted (cf figure 7).

4. Experimental details

4.1. Investigation of the valves

The dead volume of the valves is critical for the precision of the dosing system. When a valve opens, the dead volume needs to be filled and, while it closes, the dead volume needs to be emptied. Both result in a small flow which is not detected by the sensor. When the four valves are similar in performance and dead volume, it is possible that the dead volumes of different valves compensate each other.

Before they were mounted to the dosing system, both the flow rate and the dead volume of the microvalves were measured. The nitrogen flow rate at a pressure difference of one atmosphere was measured with a flow sensor as a function of the voltage applied to the piezo actuator. This is shown in figure 8, e.g. valve 1 starts to open at a voltage of 170 V and closes again after the voltage is lowered below 120 V. When the voltage is enhanced beyond 200 V, the flow through the valve no longer increases because the flow is restricted by the

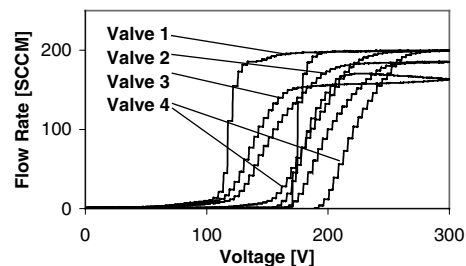


Figure 8. Nitrogen flow rate at 100 kPa through the four microvalves used to build up the dosing system.

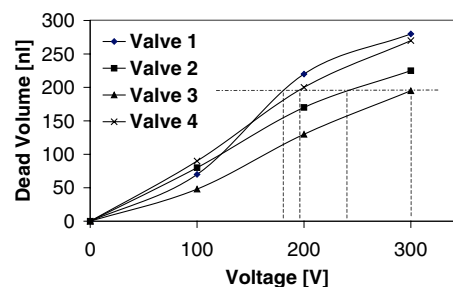


Figure 9. Dead volumes of the four microvalves used to build up the dosing system. The dotted lines indicate the voltages used to control the valves.

diameter of the inlet and not by the distance between the valve membrane and valve seat (see figure 1 for a cross-section of the valve). Figure 8 shows that the performance of the four valves is different. All valves could be opened with a voltage of 300 V and closed by discharging the piezo actuators, but this would result in different dead volumes which affect the accuracy of dosing.

Therefore, the dead volume of the valves was measured as a function of the voltage supplied to the piezo actuators. The higher this voltage is, the larger the distance between the valve membrane and valve seat and the more fluid is drawn into the valve.

A thin polymer tube with an inner diameter of 0.1 mm was connected to the outlet of the valve, the valve was filled with water, and the inlet was blocked. Then the valve was closed and opened while the end of the water column in the tube was observed with a microscope. The dead volume was calculated from the movement of the water column during valve switching. Figure 9 shows the measured dead volumes as a function of the voltage applied to the piezo actuator. The supply voltage of the microvalves was then adapted such that all valves showed the same dead volume of 200 nl. The resulting voltages are 180, 240, 300 and 200 V for valves 1–4, respectively.

4.2. Flow sensing and dosing

The resistance of each strain gauge is about 11 k Ω . Combined with two external resistors, a full bridge is assembled. With a 10 V power supply, the relation between the bridge signal and the pressure difference on the membrane is measured as shown in figure 10.

Investigations of the dosing system were carried out with water. A steady flow of the order of 500 nl min^{-1} was generated

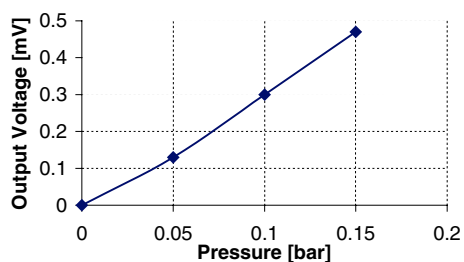


Figure 10. Relation between the strain gauge signal and the pressure.

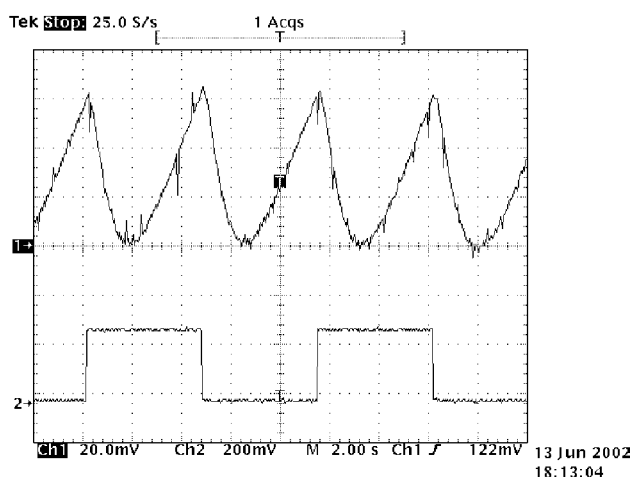


Figure 11. Upper signal: Output voltage of the Wheatstone bridge, including strain gauges, during operation of the dosing system. Lower signal: Control signal for switching the inlet and outlet valves.

and the two valve groups (valves 1 and 4 and valves 2 and 3) were switched with a frequency of 0.2 Hz. Figure 11 shows the output signal of the bridge, containing the strain gauges in the membrane, during the operation of the system with water. The different height of the peaks is due to the fact that the valves were switched with a constant frequency and an arbitrary phase. Peaks with constant heights can be reached by switching the valve when the strain gauge signal passes a threshold.

The observed signal reaches a minimum when the flat position of the membrane is passed. It is expected that the noise can be reduced with an improved electronic circuit.

The strain gauge signal is amplified and read by a DAQ board in a computer. With a LabView code as a control unit, we created a system to dose a pre-defined liquid volume.

The control unit reads in the strain gauge signal and detects the peaks of the signal. This simple control system uses the peak value to calculate the volume between the two deflected membrane states from one peak to the next. For the calculation we approximated the volume moved by the deflected membrane as a segment of a sphere with the height of the peak value. This volume corresponds to the amount of liquid which entered and also left the dosing system since the last peak. The overall amount of liquid, which has passed the system so far, is determined by adding these peak-to-peak values.

The user interface asks for the amount of liquid, which we want to dose, the target volume. The valves are closed,

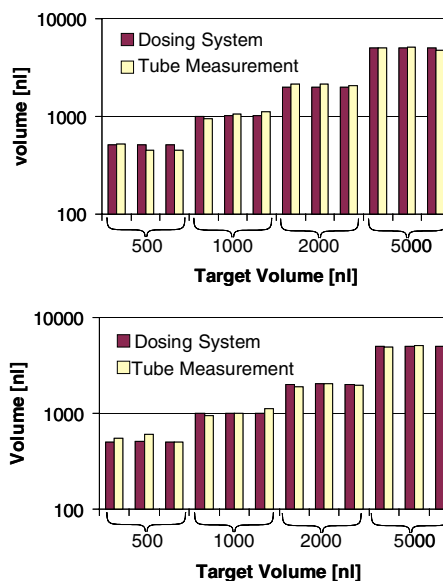


Figure 12. Dosed volume as measured with the system and the tube, respectively, as a function of the target volume at a pressure difference of 50 hPa (top) and 100 hPa (bottom).

when the sum of peak-to-peak volumes exceeds the target value. This is of course a preliminary approach with a limited accuracy. A continuous dosing will be possible, when the volume flow is derived from the resistance change during the movement of the membrane.

To measure the accuracy of the dosing system, we connected a tube with an inner diameter of 0.5 mm to the outlet of the dosing device; the real volume coming out of the device can be determined by measuring the length of the liquid column in the tube. In this way we obtained a resolution of 20 nl.

Figure 12 shows the dosing results at pressure differences of 50 hPa (top) and 100 hPa (bottom). The target volume was set to 500, 1000, 2000 and 5000 nl. It can be seen that the error of the dosing volume is less than 10%.

5. Discussion

The microdosing system presented here determines the volume flow from the measured displacement of a membrane. In this way, even the adding of an undetectable small flow rate can be measured. The measurement limit is given by leakage through the housings, through the valves, and by diffusion through the membrane.

Thermal expansion of the polymer housing and creeping of the polymer may result in a change of membrane stress. This may cause some flow, but the sensor also detects this flow.

We believe that scaling down of the sensor is limited by technical reasons. The smaller the membrane is, the more difficult it will become to obtain a reasonable resistance of the strain gauges. The smaller the diameter of the membrane is, the thinner it needs to be or the flow resistance of the dosing system will rise. However, diffusion will increase when the membrane is thinner.

The main drawback of the dosing system is the need for four microvalves with dead volumes of the order of the volume changes to be detected. Thus, scaling down is limited by scaling down of suitable valves. Therefore, we have been developing microvalves with much smaller dead volumes of less than 6 nl, which will be reported on in a forthcoming paper.

Dosing is independent of the properties of the medium used, such as heat capacity, viscosity, and density. It is also independent of particles, which may partially block flow channels. This may be the main advantage of the system introduced.

Considering biomedical applications, it may be a remarkable advantage that the fluid is not influenced by heat, which is used in almost all flow sensors in that flow range.

6. Conclusions

The deflection of a membrane due to the fluid volume entering a cavity is used to determine the volume flow. This concept allows for dosing independent of fluid properties such as density and thermal capacity and of particles in the flow.

A small counter pressure is the only effect of the system on the medium to be dosed.

The smallest amounts of fluid, which can be dosed, and the accuracy are expected to be given by technical limits such as the thinnest possible tight membrane and the smallest microvalves with sufficient small dead volume.

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