An additional uncertainty of the throughput generated by the constant pressure gas flowmeter

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Abstract. The lower range limit of constant pressure gas flowmeters is about \(10^{-8}\) Pa$\times$m$^3$/s. Detrimental gas throughputs caused by leaks and gassing from surfaces prevent from its decrease. Even if the flowmeter is entirely vacuum tight the throughput caused by the outgassing from surfaces can be sufficiently reduced only by pumping at elevated temperature. It can be performed with the flowmeters using directly driven bellows or diaphragm bellows in the volume displacers. Despite it, the lower range limit can hardly be decreased more than several ten times with up to now known designs. An additional uncertainty caused by the difference in pressure at the initial and final instant of measurement will increase at generating small throughputs to the extent that it will kill the measurement.

1. Introduction

Constant pressure gas flowmeters are widely used in metrology for generating gas throughputs in the range from many Pa$\times$m$^3$/s down to \(10^{-8}\) Pa$\times$m$^3$/s. The lower range limit is given mainly by increasing uncertainty of the generated throughput measurement. This uncertainty can be reduced and the lower limit decreased in a measure.

The principle of operation of the constant pressure flowmeter is common. The volume of the flowmeter is \(V_1\) at the initial instant \(t_1\). The gas pressure is \(p_0\) and thus, a given amount of gas \(p_0\times V_1\) is in. The gas escapes through an output element. In order to keep the pressure \(p_0\) constant the volume is reduced by means of a volume displacer. That is why the volume of the flowmeter is \(V_2\) and the amount of gas in the flowmeter is \(p_0\times V_2\) at the final instant \(t_2\). The generated gas throughput \(Q\) is calculated as

\[
Q = p_0 \frac{V_1 - V_2}{t_2 - t_1} = p_0 \frac{\Delta V}{\Delta t}. \tag{1}
\]

Despite both \(p_0\) and \(\Delta V\) and \(\Delta t\) can be measured with low uncertainties the uncertainty of the throughput \(Q\) can be unacceptably large if the following prerequisites are not met:

1) No gas apart from that escaping the output of the flowmeter either appears in the volume of the flowmeter or disappears from it.
2) The gas pressure at the final instant \(t_2\) is really exactly the same as that at the initial instant \(t_1\).
2. Undesirable change of the gas amount in the flowmeter

Apart from the gas escaping the output of the flowmeter some gas can flow in or out through leaks and the amount of gas in the volume can increase owing to the desorption from the surfaces. Modern sealing technique enables to achieve negligibly small leakage in all static joints. The only “suspicious” part of the flowmeter was earlier the volume displacer with a dynamic seal. The constant pressure flowmeters with bellows or diaphragm bellows driven either hydraulically ([1, 2]) or directly ([3, 4]) were developed in recent decades. All parts of the flowmeter are welded, brazed or metal sealed, the leakage is negligible.

Nevertheless, the desorption throughput $Q_{DES}$ flows from the surfaces in the volume of the flowmeter. Then the really generated throughput is in place of Eq. (1) given by

$$Q = p_0 \cdot \frac{\Delta V}{\Delta t} + Q_{DES}. \quad (2)$$

Apparently the minimum generated throughput has to be at least several times greater than the desorption throughput $Q_{DES}$. The achievable value of $Q_{DES}$ can be readily roughly estimated. The surface of a flowmeter with bellows or diaphragm bellows, a capacitance diaphragm gauge, a variable leak valve at the output, a small valve and necessary pipelines designed nowadays available technology altogether can hardly be less than 100 cm$^2$. The outgassing rate from stainless steel surface achievable without bake out is about $1 \times 10^{-7}$ Pa\cdot m/s [5]. Thus, $Q_{DES}$ can be approximately $1 \times 10^{-9}$ Pa\cdot m$^3$/s and the minimum applicable generated throughput can be of the order of $10^{-7} - 10^{-8}$ Pa\cdot m$^3$/s.

Much less outgassing rate can be achieved with preliminary pumping the flowmeter at elevated temperature. But, only the flowmeters with directly driven bellows or diaphragm bellows may be baked-out this way. The volume displacers employing liquid would be probably damaged. The estimations of achieved and expected results are summarised in table 1.

| Table 1. Achievable and expected values of outgassing rate and $Q_{DES}$. |
|---------------------------------------------------------------|
| Quantity | Achievable value | Remark |
|------------------|-----------------|--------|
| Inner surface of the flowmeter | 100 cm$^2$ | 1) |
| Outgassing rate I (now achieved) | $\approx 1 \times 10^{-7}$ Pa\cdot m/s | 2) |
| $Q_{DES}$ at outgassing rate I | $1 \times 10^{-9}$ Pa\cdot m$^3$/s | |
| Outgassing rate II (expected) | $1 \times 10^{-9} - 1 \times 10^{-8}$ Pa\cdot m$^3$/s | 3) |
| $Q_{DES}$ at outgassing rate II | $1 \times 10^{-11} - 1 \times 10^{-10}$ Pa\cdot m$^3$/s | |

1) … Minimum estimation.
2) … Achieved by the authors with clean stainless steel surface after some hour of pumping and several times repeated flushing with pure argon gas, without baking-out.
3) … Stainless steel surface after “reasonably practicable” baking-out.

3. Uncertainty of the displaced gas amount

The uncertainty of the displaced gas amount measurement can be high because this amount is de facto measured as a difference between the initial and final amount of gas in the flowmeter. If this difference is small in comparison with both minuend and subtrahend a high uncertainty rises.

The calculated gas amount $p_0 \times \Delta V$ is actually displaced only under the assumption that the pressure in the initial instant of measurement $p(t_1)$ is punctually the same as the pressure in the final instant of measurement $p(t_2)$. If they are different

$$|p(t_1) - p(t_2)| = \delta p > 0 \quad (3)$$

an additional relative uncertainty of the displaced gas amount measurement rises [6]:

2
\[ u_a = \frac{\delta p}{p_0} \cdot \frac{V_2}{\Delta V}. \] (4)

\( \delta p \) is only random part of the pressure measurement uncertainty (fluctuation of the pressure, changes in gas temperature, random errors of gauge reading). Systematic error (constant shift) of both \( p(t_1) \) and \( p(t_2) \) does not increase their difference. In order to give an idea of the orders of this uncertainty magnitude at small throughput generation a rough estimation will be done again. The achievable values (more or less boundary with nowadays used technique) are presented in table 2.

| Table 2. Achievable values of relevant quantities at generating very low throughputs. |
|---------------------------------------------|-----------------|--------------------------|
| Quantity | Achievable value | Remark  |
| Minimum gas pressure \( p_0 \) in the flowmeter | 10 Pa (minimum) | 1) |
| \( \delta p / p_0 \) | =1\times10^{-3} | (minimum) 2) |
| Time interval of measurement \( \Delta t \) | =10^4 s (maximum) | 3) |
| Final volume of the flowmeter \( V_2 \) | 10 cm^3 (minimum) | 4) |

1) … Capacitance diaphragm gauge is used for pressure measurement.
2) ... The authors of this paper can confirm the experience from [6]. Exactly unexplained random pressure fluctuation of the order of one per mille can hardly be avoided at stabilising low pressure by means of the volume displacer.
3) ... The time interval of measurement is limited by the temperature stability requirement.
4) ... The final volume of the flowmeter involves the inner volume of a capacitance diaphragm gauge (=3.5 cm^3 - estimation according to a front-end manufacturer’s specification), inner volume of an output variable leak valve (=5.4 cm^3 - estimation according to the authors measurement), inner volume of a by-pass valve, necessary pipelines and the volume of the displacer at the instance \( t_2 \).

The consequence is exemplified in table 3.

| Table 3. The increase of the additional uncertainty \( u_a \). |
|-----------------------------|-----------------|-----------------|
| Throughput to be generated [Pa×m^3/s] | Volume \( \Delta V \) to be displaced within 10^4 s [cm^3] | Additional uncertainty \( u_a \) |
| 1×10^{-7} | 100 | =1×10^{-4} |
| 1×10^{-8} | 10 | =1×10^{-3} |
| 1×10^{-9} | 1 | =1 % |
| 1×10^{-10} | 0.1 | =10 % |
| 1×10^{-11} | 0.01 | =100 % |

4. Discussion
The detrimental gas throughput caused by leakage and desorption is an obvious reason for the increase of uncertainty at the lower range limit of the constant pressure flowmeter. The leakage can be avoided at suitable design of the flowmeter using bellows or diaphragm bellows in the volume displacer. Thus, the outgassing from the surfaces is the main reason of the detrimental throughput. It cannot be reduced sufficiently neither by means of long term pumping at room temperature nor by means of pure inert gas flushing. One possible way to reduce gas evolution is the baking-out i.e. pumping at elevated temperature.

Only the flowmeters with directly driven bellows or diaphragm bellows could be baked out. The outgassing rate from the used materials may be reduced substantially this way, but the long-term
keeping of the bellows or diaphragm bellows at the elevated temperature could change their elastic properties. That is why the influence of this procedure on the achievable accuracy of the displaced volume measurement has to be carefully tested.

To use a high temperature for very long time at baking-out in order to reduce the outgassing rate as much as possible (as it was tested in many papers – overview for example [7]) is not necessary. Reduction of the detrimental throughput beyond a certain extent loses its meaning. Even if the detrimental throughput were entirely zero the additional uncertainty caused by the difference in the initial and final pressure in the flowmeter would kill the possibility to generate very small throughputs sufficiently accurately.

To discover and to put away the reasons of apparently random changes of pressure during the measurement is difficult. If a higher gas pressure is used also a longer time interval of measurement $\Delta t$ will have to be used and $\delta p$ will increase on account of long term temperature instability. The only way is to make smaller the ratio $V_2/\Delta V$ which is the sensitivity coefficient associated with the uncertainty $\delta p/p_0$, thus, to minimize the volume $V_2$. This volume $V_2$ can hardly be less than about 10 cm$^3$ with the design of the pressure gauges, valves and volume displacers used on the present.

5. Conclusions

The lower range limit of the constant pressure gas flowmeters is determined by the uncertainty caused by the detrimental gas throughput and by the uncertainty caused by the difference in the initial and final pressure in the flowmeter.

The constant pressure flowmeters using directly driven bellows or diaphragm bellows in volume displacers can be entirely vacuum tight and they could be outgassed at elevated temperature in order reduce the uncertainty caused by the gas desorption from the surfaces.

Only small volume of gas $\Delta V$ can be displaced during suitable time interval of measurement at generating small gas throughputs. Thus, the uncertainty caused by the difference in the initial and final pressure in the flowmeter is high, because the associated sensitivity coefficient $V_2/\Delta V$ is high.

In order to decrease the lower range limit of the constant pressure flowmeter it is necessary to make the final volume $V_2$ as small as possible. The final volume $V_2$ cannot be less than several cm$^3$ with the basic parts (gauges, valves, displacers) used at present. Thus, the lower range limit cannot be less than approximately $10^{-9}$ Pa$\times$cm$^3$/s even after outgassing at elevated temperature.

6. References

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