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# Liquid properties effects on Coriolis and thermal mass flow meters at very low flow rates

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**Abstract**

Calibration of flow devices are important in several areas of pharmaceutical, flow chemistry and health care applications where volumetric dosage or delivery at given flow rates are crucial for the process. Although most of the flow devices are measuring flow rates of process-oriented liquids their calibrations are often performed with water as calibration liquid. It is recommended to perform the calibrations of the flow devices with process-oriented liquids as the liquid itself might influence the performance of the flow devices. Therefore, METAS has developed facilities with METAS flow generators to address the issue of measuring with process-oriented liquids for flow rates from 400 ml/min down to 50 nl/min with uncertainties from 0.07 % to 0.9 %.
The effects of liquids with different viscosities and thermal properties on the measurement accuracy of Coriolis and thermal mass flow meters have been investigated at these very low flow rates. Calibrations with water and several reference oils with traceable viscosity were performed to study the viscosity effect on the flow meter performance and the dependency on the thermal properties for the thermal mass flow meter. The results will be discussed in this paper.

## 1. Introduction

Calibration of flow devices are important in several areas of pharmaceutical, flow chemistry and health care applications where volumetric dosage or delivery at given flow rates are crucial for the process. Although most of the flow devices are measuring flow rates of process-oriented liquids, their calibrations are often performed with water as calibration liquid. It is recommended to perform the calibrations of the flow devices with process-oriented liquids as the liquid itself might influence the performance of the flow devices. In this paper, we present calibrations results of two Coriolis flow meter with reference oils of different viscosities ranging from 1.0 mPa·s to 7.5 mPa·s. The Coriolis flow meters are known to have a very small dependency of viscosities. On the other hand, calibration results of a thermal mass flow meter with the same liquids are presented in this paper. However, the scaling behaviour is much more difficult as the heat capacity and the heat conductivity play an important role and the scaling behaviour is not linear with respect to the properties of water. In most cases, the manufacturer of liquid thermal mass flow meters propose calibrations with water and IPA (Isopropanol alcohol) with corresponding calibration parameters stored in the flow sensors. For liquids consisting of hydrocarbon chains the behaviour of the thermal mass flow sensor is linearly scalable with the calibration parameters obtained with IPA as it is a good thermal representative for most hydrocarbons [1, 2].

After developing a facility for the micro-flow range [3, 4], METAS has developed facilities with METAS piston provers to address the issue of measuring with process-oriented liquids. The METAS piston provers are homemade and allow measurements with liquids other than water in the range from 400 ml/min down to 50 nl/min [5, 6]. Traceability is guaranteed through the calibration of the generated flow rates of the METAS piston provers by means of the dynamic gravimetric method where a liquid of well-known density and a well-controlled evaporation rate is used. In a later stage, it will be directly traceable to length and time as it is usually done for piston provers. As the METAS piston prover is a volumetric flow generator, it can be operated with any liquid acting as a transfer standard to perform calibrations of flow devices. The advantage of traceable calibrations of a flow device with the process-oriented liquid is to enhance the quality of the measurements results of the flow device during the production process. Different types of flow devices are stated from the manufacturer with accuracies between 0.5 % and 10 % depending of the flow rate range and the working principle of the flow devices. Obviously, a traceable calibration with uncertainties ranging from 0.07 % to 0.9 % for steady flow rates ranging from 400 ml/min to

50 nl/min will enhance the accuracy of the measurements as the stated deviations of the flow devices are corrected in the application and the measurement uncertainties are known and much smaller than the accuracies stated by the manufacturer.

## 2. The METAS piston provers

The METAS piston provers consist of a high precision linear stage with a fixed linear measuring system, mounting parts to fix commercially available syringes or homemade prover cylinders in front of the table and mounting parts to fix and move the piston in the prover cylinder in order to generate the flow rate (see Figure 1 and Figure 2), [5]. The position of the linear stage is determined by counting the pulses sent by the linear measuring system by means of an FPGA, which is a Field Programmable Gate Array with hard coded program code running on a defined constant cycle time of the order of 25 ns (40 MHz). For each additional pulse in any direction, a time stamp of the FPGA is recorded and a pair with the position and the timestamp is formed. This pair of values is then read from the main software and the real time position can be recorded. The real time speed is then determined by a linear fit of several pairs of position data with the corresponding time stamps as the slope corresponds to the speed. Multiplying the speed with the cross section of the syringe gives the volume flow rate. The speed range for the METAS piston prover of the Microflow facility resp. of the Milliflow facility is from 0.1 mm/s to 0.1 µm/s resp. from 4.0 mm/s to 4.0 µm/s.

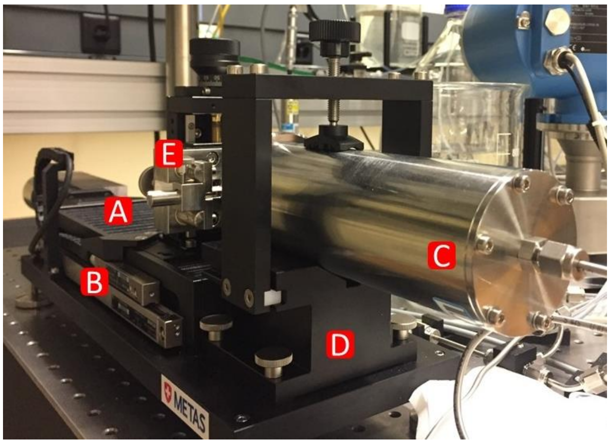


**Figure 1:** METAS piston prover of the Milliflow facility, (A) high precision linear stage, (B) linear measuring system, (C) piston cylinder, (D) mounting piston cylinder, (E) mounting and positioning for the piston.

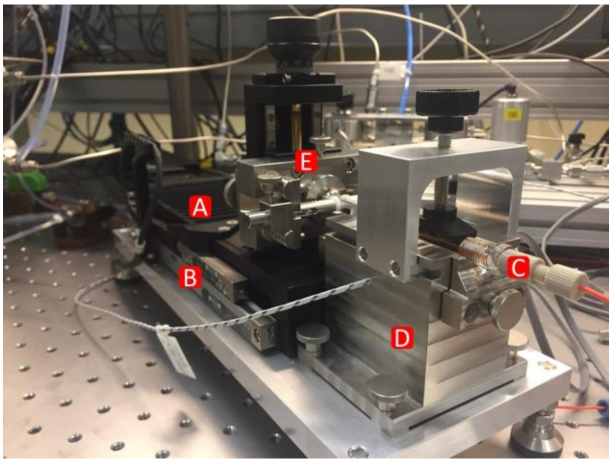


**Figure 2:** METAS piston prover of the Microflow facility, (A) high precision linear stage, (B) linear measuring system, (C) syringe, (D) mounting syringe body, (E) mounting and positioning for syringe plunger.

## 3. Liquid properties

The relevant properties of the reference oils that are used for the measurements with the Coriolis mass flow meters and a thermal mass flow meter are listed in Table 1.

**Table 1:** Properties of the reference oils and water at approx. 21.4 °C.

| **Property** | **Water** | **Reference oil 2BW** [1] | **Reference oil 5BW** [1] | **Reference oil 10AW** [1] |
|---|---|---|---|---|
| Dyn Viscosity $\eta$ ($kg{\cdot}m^{-1}{\cdot}s^{-1}$) | $0.9624 \cdot 10^{-3}$ | $2.361\ 10^{-3}$ | $5.903\ 10^{-3}$ | $8.419\ 10^{-3}$ |
| Spec. Heat capacity $c_p$ ($J{\cdot}kg^{-1}{\cdot}K^{-1}$) | 4184 | 2130 | 2067 | 2046 |
| Th. con-ductivity $\lambda$ ($W{\cdot}m^{-1}{\cdot}K^{-1}$) | 0.600 | 0.143 | 0.151 | 0.147 |
| Density $\rho$ ($kg{\cdot}m^{-3}$) | 997.9 | 767.5 | 796.4 | 805.9 |

[1] the reference oils are commercially available at https://zmk-wolfen.de/, ZMK & ANALYTIK GmbH in Germany.

## 4. Coriolis and thermal mass flow meters

The Coriolis and thermal mass flow meters are calibrated with the liquids water and the reference oils 2BW, 5BW and 10AW, where the properties are listed in Table 1. The METAS piston provers are used as traceable volumetric flow generators and are filled with each of these liquids. As the Coriolis mass flow meters measure mass flow rates, the volumetric flow rate of the piston prover is converted into mass flow rate by multiplying with the density of the liquid. The thermal mass flow meter indicates the volumetric flow rate.

### *4.1 Coriolis mass flow meters*

The following Coriolis mass flow meters were calibrated with these liquids: Cubemass DCI DN01 from Endress+Hauser AG and miniCori M12 from Bronkhorst High-Tech B.V. For both Coriolis flow meters the zeroing procedure was applied prior to the measurement with water. Afterwards, for the measurements with the other liquids, no zeroing procedure was performed, but the zero flow was measured in all cases.

*4.2 Cubemass DCI DN01 from Endress+Hauser AG*

The calibration results of the Cubemass DCI DN01 from Endress+Hauser AG are shown in Figure 3, Figure 4 and Figure 5, where the deviation is shown as a function of the reference mass flow rate, the Reynolds number and for the last figure with the zero flow correction applied after the measurements. It is common to use the Reynolds number instead of the mass flow rate as values for the x-axis as it takes into account the mass flow rate and the dynamic viscosity of the liquid.

In Figure 3, the results show a constant deviation for mass flow rates larger than 30 g/min for all liquids. For the mass flow rates around 10 g/min, the deviations seem to be widely spread for the different liquids. However, if we plot the deviations as a function of the Reynolds number as shown in Figure 4, we can observe a trend in the data, which is highlighted by black dashed trend-line drawn by hand.

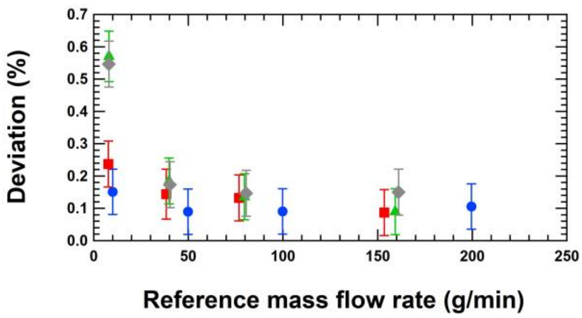


**Figure 3:** Cubemass DCI DN01. Deviations as a function of reference mass flow rate for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds).

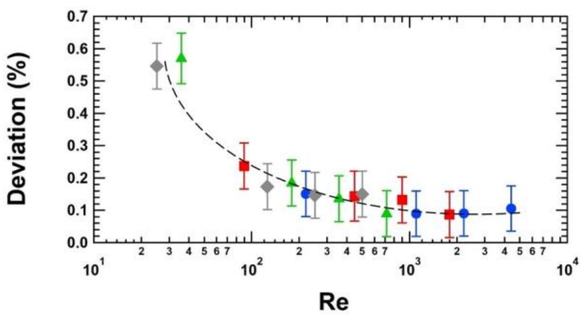


**Figure 4:** Cubemass DCI DN01. Deviations as a function of Reynolds number for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds). The black dashed line is only a trend-line.

Applying the zero flow correction by subtracting the measured zero flow (Table 2) from the measured flow rate, we obtain the deviations presented in Figure 5, which are close to a constant deviation for all liquids (except for the liquid 2BW at Re ~ 100). No obvious dependency of the various liquids with different viscosities could thus be observed in this case.

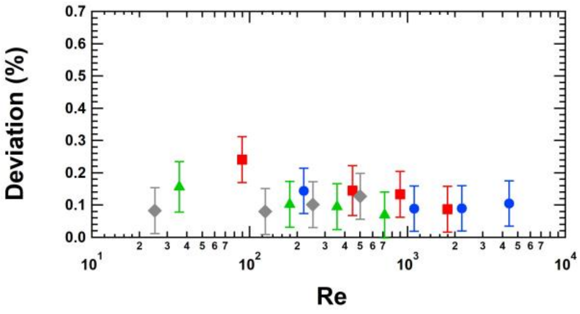


**Figure 5:** Cubemass DCI DN01. Deviations of the zero-flow corrected flow rates of the Coriolis mass flow meter as a function of Reynolds number for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds).

**Table 2:** Measured Zero flows of the Coriolis mass flow meters with the reference oils and water.

| Coriolis flow meter | Water | Reference oil 2BW | Reference oil 5BW | Reference oil 10AW |
|---|---|---|---|---|
| Cubemass DCI DN01 (mg/min) | +0.65 | -0.30 | +33.0 | +37.3 |
| miniCori M12 (mg/min) | +0.2 | +0.0 | +0.0 | +0.0002 |

*4.3 miniCori M12 from Bronkhorst High-Tech B.V.*

The calibration results of the miniCori M12 from Bronkhorst High-Tech B.V. are shown in Figure 6, Figure 7 and Figure 8, where the deviation is shown as a function of the reference mass flow rate, the Reynolds number and for the last figure with the zero flow correction applied after the measurements.

Also for these measurements, the results look more consistent when the deviations are plotted against the Reynolds number as shown in Figure 7. However, applying the zero flow correction by subtracting the measured zero flow (Table 2) from the measured flow rate does not change the results as the measured zero flow rates are already negligible (see Figure 8). We observe rather constant deviations for Reynolds numbers larger than 20 and a wider spread of the deviations for smaller Reynolds numbers. No systematic dependency on the various liquids with different viscosities could be observed for this Coriolis flow meter. The spread of the data for Reynolds numbers smaller than 10 are due to the larger inaccuracy of the

Coriolis measurement principle at lower flow rate rather than due to the any viscosity effect.

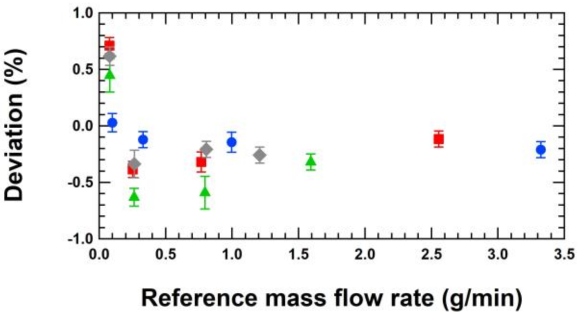


**Figure 6:** miniCori M12. Deviations as a function of reference mass flow rate for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds).

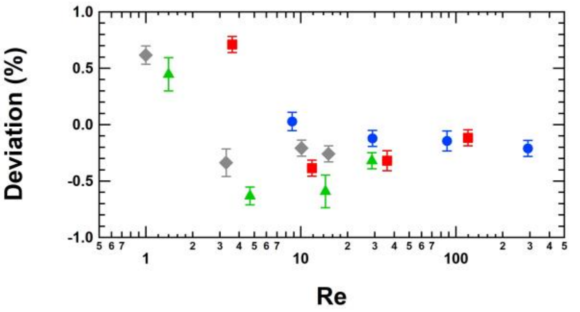


**Figure 7:** miniCori M12. Deviations as a function of Reynolds number for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds).

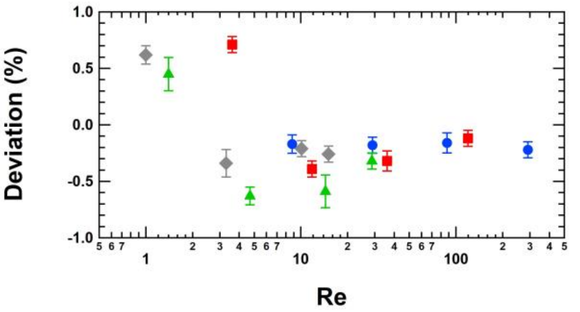


**Figure 8:** miniCori M12. Deviations of the zero-flow corrected flow rates of the Coriolis mass flow meter as a function of Reynolds number for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds).

*4.4 Thermal mass flow meter SLI-0430 from Sensirion*

The thermal mass flow meter SLI-0430 from Sensirion AG was calibrated with the liquids water and the reference oils 2BW, 5BW and 10AW (properties listed in Table 1).

As mentioned above, the manufacturer of liquid thermal mass flow meters propose calibrations with water and IPA with corresponding calibration parameters stored in the flow sensors. The calibration parameters of water were set in the flow sensor for the measurements showed in this paragraph. The results are presented in Figure 9 and Table 3. The indicated flow rates of the SLI-0430 are much smaller than the reference flow rate for the reference oils 2BW, 5BW and 10AW. The ratios between the indicated flow rates of water and any of the reference oils is dependent on the flow rate. Therefore, it is very difficult to find a scaling behaviour depending only on thermal properties of the liquids. The fact that the Zero flow of the SLI-0430 with the different reference oils indicates already non-negligible contributions (see Table 4) emphasizes these scaling difficulties.

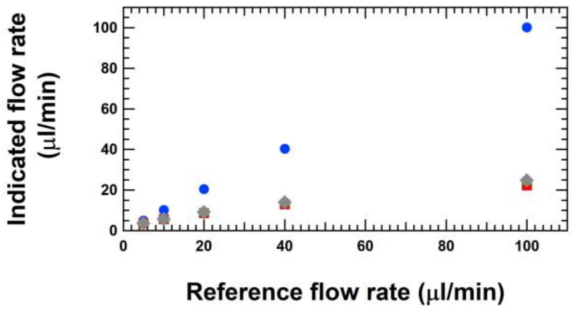


**Figure 9:** SLI-0430. Indicated flow rate as a function of the reference flow rate for the different liquids: water (blue circles), liquid 2BW (red squares), liquid 5BW (green triangles), liquid 10AW (gray diamonds). Uncertainties are smaller than the data symbols.

**Table 3:** Measured flow rates for the SLI-0430 with the different liquids and the corresponding uncertainties.

| Ref. flow rate (µl/min) | Indicated flow rate of the SLI-0430 (µl/min) | | | | Uncert. (k=2) (µl/min) |
|---|---|---|---|---|---|
| | **Water** | **Ref. oil 2BW** | **Ref. oil 5BW** | **Ref. oil 10AW** | |
| 99.96 | 100.07 | 22.277 | 24.287 | 24.844 | 0.17 |
| 39.984 | 40.266 | 12.937 | 13.808 | 14.012 | 0.089 |
| 19.990 | 20.425 | 8.666 | 9.061 | 9.162 | 0.066 |
| 9.995 | 10.175 | 5.598 | 5.738 | 5.785 | 0.028 |
| 4.998 | 4.963 | 3.542 | 3.602 | 3.619 | 0.021 |

**Table 4:** Measured Zero flows of the Thermal mass flow meter with the reference oils and water.

| Thermal mass flow meter | Water | Reference oil 2BW | Reference oil 5BW | Reference oil 10AW |
|---|---|---|---|---|
| SLI-0430 (µl/min) | +0.030 | +0.859 | +0.868 | +0.870 |

Therefore, it would have been advisable to load the calibration parameters of the IPA calibration in the SLI-0430. Thus, measurements with IPA and the reference oils would probably have led to a scaling behaviour between these liquids. However, this points out the difficulty to perform measurements with different liquids of very different thermal properties and thermal behaviour. This also underlines the fact that the flow devices measuring flow rates of process-oriented liquids should be calibrated with the process-oriented liquids. This would limit the influence of the theoretical scaling behaviour on the measurement uncertainty and increase the accuracy of the measurements.

*4.5 Thermal mass flow meter SLI-1000 from Sensirion*

To illustrate a possible scaling behaviour the thermal mass flow meter SLI-1000 from Sensirion AG has been calibrated with water and liquids that are aqueous solutions: 0.0111 g Ethanol per 1 g water (solution 1) and 0.0222 g Ethanol per 1 g water (solution 2). The thermal properties have been taken from the literature at a temperature of 20 °C and are summarized in Table 5. The specific heat capacity and the thermal conductivity have been calculated according to the mass weighted mean of the individual components of the solutions according to Equation (1) and (2). These values are approximate, but they are useful to explain the calibration data obtained with different liquids for the SLI-1000.

$$c_{p,sol} = \frac{m_{H2O} \cdot c_{p,H2O} + m_{Ethanol} \cdot c_{p,Ethanol}}{m_{H2O} + m_{Ethanol}} \quad (1)$$

$$\lambda_{sol} = \frac{m_{H2O} \cdot \lambda_{H2O} + m_{Ethanol} \cdot \lambda_{Ethanol}}{m_{H2O} + m_{Ethanol}} \quad (2)$$

**Table 5:** Properties of water and the aqueous solutions at 20.0 °C.

| Property | Water | Etanol | Solution 1 (1.1 %wt) | Solution 2 (2.2 %wt) |
|---|---|---|---|---|
| Dyn Viscosity $\eta$ [7] ($kg \cdot m^{-1} \cdot s^{-1}$) | $1.002 \cdot 10^{-3}$ | $1.189 \cdot 10^{-3}$ | $1.040 \cdot 10^{-3}$ | $1.078 \cdot 10^{-3}$ |
| Spec. Heat capacity $c_p$ ($J \cdot kg^{-1} \cdot K^{-1}$) | 4184 | 2430 | 4164.7 | 4145.9 |
| Th. conductivity $\lambda$ ($W \cdot m^{-1} \cdot K^{-1}$) | 0.600 | 0.200 | 0.596 | 0.591 |
| Density $\rho$ ($kg \cdot m^{-3}$) | 998.21 | 789.34 | 996.18 | 994.17 |

For the anemometric regime with flow rates larger than 2 g/h (33 µl/min), the relation between the mass flow rate $q_m$ and the heater power $P_{heater}$ is $P_{heater} \sim q_m^{0.33}$ [8]. Assuming that the heater power is linearly dependent on the heat capacity of the liquid, the conversion factor should be as described in Equation (3):

$$q_{m,Sol} \cong q_{m,H2O} \cdot \left(c_{p,H2O}/c_{p,Sol}\right)^3 \quad (3)$$

And for volume flow rate ($q_V$) we get Equation (4):

$$q_{V,Sol} \cong q_{V,H2O} \cdot \left(\rho_{H2O}/\rho_{Sol}\right) \cdot \left(c_{p,H2O}/c_{p,Sol}\right)^3 \quad (4)$$

Applying Equation (4) to the measured data, we obtain the theoretical flow rates for the solution 1 and solution 2, reported in Table 6 and the deviations as a function of the Reynolds number shown in Figure 10.

**Table 6:** Theoretical flow rates for the solution 1 and solution 2.

| Reference flow rate (µl/min) | SLI-1000 (µl/min) | | | | |
|---|---|---|---|---|---|
| | Water | Sol. 1 | Sol. 1 scaled | Sol. 2 | Sol. 2 scaled |
| 559.9 | 542.0 | 551.2 | 550.7 | 557.2 | 559.3 |
| 509.9 | 493.6 | 501.9 | 501.5 | 507.4 | 509.4 |
| 459.9 | 445.3 | 452.8 | 452.4 | 457.5 | 459.6 |
| 359.9 | 351.5 | 356.8 | 357.1 | 360.6 | 362.7 |
| 260.0 | 256.4 | 260.3 | 260.5 | 263.1 | 264.6 |

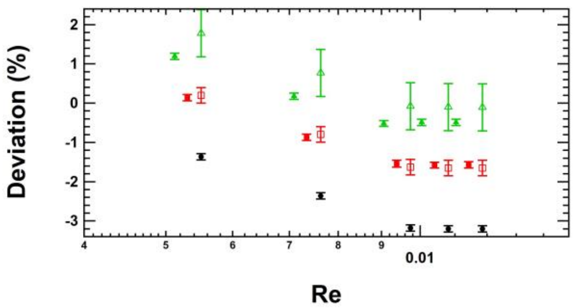


**Figure 10:** SLI-1000. Deviations as a function of Reynolds number for the different liquids: water (black circles), solution 1 (red squares), solution 2 (green triangles). Full symbols are measured data and open symbols are theoretical values.

The deviations of the measured flow rates and the theoretical expected flow rates for the SLI-1000 and the corresponding uncertainties are reported in Table 7 resp.
Table 8.

**Table 7:** Deviations of the measured flow rates and the theoretical expected flow rates for the SLI-1000 in Table 6.

| Deviation of the SLI-1000 (%) | | | | |
|---|---|---|---|---|
| Water | Sol. 1 | Sol. 1 scaled | Sol. 2 | Sol. 2 scaled |
| -3.20 | -1.57 | -1.65 | -0.49 | -0.11 |
| -3.20 | -1.58 | -1.65 | -0.49 | -0.10 |
| -3.18 | -1.54 | -1.63 | -0.52 | -0.08 |
| -2.36 | -0.87 | -0.80 | 0.18 | 0.76 |
| -1.37 | 0.14 | 0.20 | 1.19 | 1.78 |

**Table 8:** Uncertainties of the measured deviations and the scaled deviations for the SLI-1000 in Table 7.

| Uncertainties (k=2) (%) | | | | |
|---|---|---|---|---|
| Water | Sol. 1 | Sol. 1 scaled | Sol. 2 | Sol. 2 scaled |
| 0.08 | 0.08 | 0.20 | 0.08 | 0.60 |
| 0.08 | 0.07 | 0.20 | 0.08 | 0.60 |
| 0.08 | 0.09 | 0.20 | 0.07 | 0.60 |
| 0.08 | 0.08 | 0.20 | 0.08 | 0.60 |

| | | | | |
|---|---|---|---|---|
| 0.08 | 0.08 | 0.20 | 0.08 | 0.60 |

The uncertainties for the theoretical expected flow rates (scaled) were chosen in a way that the measured and the theoretical data are consistent within the uncertainties. This means that by applying a scaling factor to data measured with one liquid and to estimate the flow rate for another liquid it has to be taken into account that the theoretical value is more uncertain than the measured value. This example also show the importance to calibrate the flow sensor with the process-oriented liquid that will be used for the measurements. This will increase the quality of the measurements results and considerably decrease the uncertainty, if needed.

## 5. Conclusion

Calibration results of Coriolis and thermal mass flow meters performed with the METAS piston provers with the liquids water and the reference oils 2BW, 5BW and 10AW were discussed in this paper.
No obvious dependency of the various liquids with different viscosities could be observed for the Coriolis mass flow meters.
The thermal mass flow meters showed obviously strong dependencies on the thermal properties of the liquids. Estimating a scaling behavior for the reference oils with the sensor parameters set to the water calibration turned out to be a very difficult task and is beyond of the scope of this paper. The calibrations of a thermal mass flow meter with water and aqueous solutions offered a scaling behaviour and an estimation of the uncertainty of these theoretical predictable flow rates. As these scaling effects are already important, no effect of the viscosity on the flow meter performance could be investigated.
These measurements show the importance of calibrating the flow sensor with the process-oriented liquid that will be used for the measurements. This will increase the quality of the measurements results and considerably decrease the uncertainty, if needed.

## References

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