NEW PRIMARY STANDARD WITH PISTON PROVER FOR MICROFLOW OF LIQUIDS

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Introduction

- gravimetric primary standard in the range of (10 to 6 000) g/h
- development of a piston standard with the aim
  - to automate the calibration process
  - to reduce uncertainty of measurement
  - enlarge the range to lower flows from 1 g/h

- To present detailed design of the new test equipment with integrated micro piston prover into the testing line
- comparison of measurement results between the volumetric standard with the piston prover and gravimetric standard in one place
Mass primary standard

- gravimetric primary standard in the range of (10 to 6 000) g/h
- this standard was fully adapted for required conditions, proclaimed as the Czech primary standard
- successfully internationally compared within the EURAMET No. 1379 project with degree of equivalence (En value) in the range (0,09 to 0,38) for the range of flow (500 to 6000) g/h

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Prime Parameter</th>
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</thead>
<tbody>
<tr>
<td>Measuring range:</td>
<td>(10 to 6 000) g/h</td>
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<tr>
<td></td>
<td>(10 to 6 000) ml/h</td>
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<tr>
<td>Working pressure:</td>
<td>(140 – 340) Pa</td>
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<tr>
<td>Testing liquid:</td>
<td>distilled water</td>
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<tr>
<td>Water temperature:</td>
<td>(25 ± 5) °C</td>
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<tr>
<td>Expanded uncertainty (CMC) – mass flow</td>
<td>0,50 % for flow (10 to 30) g/h</td>
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<tr>
<td></td>
<td>0,15 % for flow (30 to 2000) g/h</td>
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<td></td>
<td>0,20 % for flow (2000 to 6000) g/h</td>
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<tr>
<td>Expanded uncertainty (CMC) – volumetric flow</td>
<td>0,60 % for flow (10 to 30) ml/h</td>
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<tr>
<td></td>
<td>0,20 % for flow (30 to 2000) ml/h</td>
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<td>0,25 % for flow (2000 to 6000) ml/h</td>
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</table>
Mass primary standard

- For calibration usually secondary mass standards are used in the same range.
- The reason is a more practical measurement even at the cost of a slight increase in measurement uncertainty.
- “More practical measurements” means elimination of certain effects that occur in the mass method, especially the impact of liquid evaporation, influence of changing temperature with the effect on density in the calculation of the correct value and also the duration of particular measurements.
- The negative effect of evaporation could be excluded by using liquids other than distilled water or with lower evaporation ability or corrections.
- The use of secondary standards has its practical advantages at the installation of the meter or the measurement time, but also disadvantages in the form of heating up the meters and thus the increase of the temperature gradient.
Development of the piston prover facility

- For these reasons the direction of development of a piston standard, with the **aim of reducing the measurement** uncertainty while **maintaining comfort during calibration** has been chosen.

- Experience from bigger flows were used.

- The equipment is with a collecting water tank for displacement of the liquid, distilled water is used as standard, but the equipment also **allows calibration with other liquids**.

- The system is equipped with **Pt100 temperature** sensors with a diameter of 3mm and **pressure sensor** directly in the piston chamber.

- The equipment is **controlled by software** that allows to pre-define an amount of sequences in measurement points and various test types and methods, so that **tests can run automatically**.
Piston standard – solution proposals

- Designing the optimal piston diameter in relation to the required test flow range as well as volume required from 0.1 mL to 200 mL.
- By analysing various factors a nominal piston diameter of **35mm** was defined, movement speed of the piston corresponding to a flow range from 2.89x10^-4 mm/s to 1.73 mm/s.
- Motion screw, gearbox and servo drive were used based on optimizing particular parameters it was necessary to achieve a **ratio of 1:2000**.
- An encoder for reading pulses with a resolution of **65536 pulses per revolution** is solid attached to the screw, corresponding to a volume of 1.002x10^-6 ml per 1 pulse.
- The piston itself moves along the ball runner block with high accuracy, special sealing is used.
- The piston prover is positioned in an **inclined position** to ensure easier removal of eventual air bubbles.
Piston standard – admeasurement

- The admeasurement of the piston was done using a probe coordinate measuring machine type SIP CMM 5, with a measurement uncertainty of 0.001 mm.
- The piston is clamped to the measuring machine where the centre of clamping will be the centre of the piston.
- Radiuses are counted on an imaginary line lying on the surface of the piston parallel to the axis of the piston, always at distances of 5 mm from each other.
- After reaching the end of the piston the piston is rotated by 45° and the measurement is repeated. The entire process is done in eight planes to measure radiuses at angles: 0°, 45°, 90°, 135°, 180°, 225°, 270° and 315°.

We calculate one part of the surface $\oplus$ using the surface of the Archimedean screw:

$$ p = \alpha_1 - \alpha_2 \times (r_1^2 + r_1 \times r_2 + r_2^2) $$

after simplification (if delta alpha = 45° )

$$ p = \pi 46 \times (r_1^2 + r_1 \times r_2 + r_2^2) $$

Subsequently, the cutting area of the piston is calculated at one point of the measurement:

$$ P = \sum_{i=1}^{8} p_i $$
Measurement principles

- **Mass collection method**
  the value of mass before and after the test from the meter is read by the control PC. The difference in mass values from the meter is compared to the mass value of water displaced from the piston (based on the volume, temperature and mass of distilled water).

- **Mass flow method**
  the equipment is set to the desired flow rate. The system waits until the meter starts to indicate flow and settles within the selected tolerance. After reaching it, the system starts to average the instantaneous mass flow (automatically, approximately every second). The test stops after the requested amount has flown. Subsequently, based on the average flow from the meter and the time of test it calculates mass and compares it repeatedly to the mass of water displaced from the piston based on volume, temperature and mass of water.

- **Volume collection and volume flow method**
  In case the meter with a volume output of delivered volume or flow is connected, a similar principle is applied, the volume or flow volume from the piston is directly applied as the correct value.
Following main effects were considered by generation of mathematical models:

- Water temperature change
- Pressure change
- Final step size of piston position
- Mechanical connection of the helix of the piston
- Thermal expansion of the helix
- Mechanical accuracy of the helix
- Piston calibration uncertainty
- Water temperature difference between piston and MUT
- Pressure sensor calibration
- Tubing expansion effect
Confirmation of metrology parameters

Parameters having a significant impact on the correct measured value have been monitored during the development of the piston. These are especially: **accuracy of delivered quantity, temperature and flowrate**.

As the piston standard is a volumetric meter, the **stability and accuracy of temperature** measurement are significant factors.

**Long-term measurements** (10h) showed stability **better than 0,07°C** (Max-Min).

Real measurements with the piston have shown a bigger temperature change during the measurement **approx. 0,1°C**.

This fact is probably caused by heating up the water by mass flow meters, that were used for evaluation of results. Temperature fluctuations have very little effect on the resulting measurement uncertainty. **The uncertainty contribution is at a level of 0,002% at a testing volume of 5ml.**
The functionality was inspected with reference mass flow meters Bronkhorst – Coriflow (P1, P2, P3) linked to the primary standard with weighing scales.

These measurements were performed in the range of (1 až 6 000) g/h.

The resulting values are mean values from 5 to 10 repeat measurements, standard deviations are shown at each point of flow.

Comparative measurements by reference standard meters

![Graph showing comparative measurements for P2 (10-600 g/h)]
Confirmation of metrology parameters

For P1 (550-6000 g/h)

Legend:
- P1 mass, piston prover (zero set before each flow change)
- P1 weighing, scale (zero set at the beginning)
- P1 mass, piston prover (zero set at the beginning)

For P3 (1-20 g/h)

Legend:
- P3 mass (piston prover)
- P3 mass flow (piston prover)
Conclusion

- the expanded uncertainty (0.05 to 0.6)\%.
- Experimental measurements have confirmed stability of results, simple calibration
- The design of the equipment ensures long-term tightness stability with simple visual leak control
- Excluding some effects such as temperature stability, flow stability, evaporation, etc. has a major effect on reducing partial uncertainties
- calibration by a more methods, so the field of tested instruments could be extended

In the next period,
- a direct comparison of the piston to the scales is planned, thus avoiding the impact of the reference meters