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NEW PRIMARY STANDARD WITH PISTON PROVER FOR MICROFLOW OF LIQUIDS

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Abstract

The Czech Metrology Institute continues in development of a method in the area of very small flows of liquid aimed at reducing uncertainties and expanding flow to lower values. The primary standard developed based on mass method has successfully been internationally compared and the results have shown data stability.
With increasing accuracy of flow meters on the market, requirements for reduction of uncertainty of primary or secondary standards are rising.
In case of very small flows, i.e. below 10 ml/h, some calibrations and subsequent result evaluations by means of the weighing method become a little complicated. With the aim to automate the calibration process as well as to reduce uncertainty of measurement and also enlarge the range to lower flows, an innovative primary standard with 200 mL micro piston prover has been developed. Long-term experience with development of a volumetric method using a similar type of piston prover with a special fluid displacement system in the area of the larger flows was applied.
The paper is focused on the presentation of the comparison of measurement results between the volumetric standard with the piston prover and gravimetric standard in one place. A detailed design of the new test equipment with integrated micro piston prover into the testing line is presented as well. Further, experimental measurements in very low flow rates (1 to 6,000) ml/h are described and evaluated. Achieved measurement uncertainties are also presented. The results show the benefits of the applied method.

1. Introduction

The Czech Metrology Institute uses for measurements in the area of microflow of liquids a gravimetric primary standard in the range of (10 to 6000) g/h. This standard was fully adapted for required conditions, proclaimed as the Czech national standard and 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.
For calibration of flow meters as infusion pump calibrators, peristaltic pumps, mass and volumetric flow meters or micro-injection pumps 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.
According to previous research, the negative effect of evaporation could be excluded by using liquids other than distilled water with lower evaporation ability. Unfortunately, a large number of calibrated devices are from the health sector and experience has shown the necessity of using clean, i.e. distilled water.
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, also the effect on the final value in form of density in the calibration of volumetric flow meters and especially the experience with calibration in zero position (zero calibration) are very important. The range of low flows is also limited.
For these reasons the Czech Metrology Institute has chosen the direction of development of a piston

standard, with the aim of reducing the measurement uncertainty while maintaining comfort during calibration. In this area , long term experience in the field of gas flow measurement using the volumetric method with a bell prover, in the field of development of piston standards used in testing equipment for verification of water meters has been used together with experience in the field of very small flow measurements.
Also in this work, the experience of a flow standard equipment manufacturer and the national metrology institute was commonly used.
The aim of the project was to design and produce a standard equipment based on a piston standard for calibration of flow meters in the field of small flows from 1 ml to 6000 ml.

2. Mass primary standard

With respect to the required accuracy and measuring range a balance with weighing range of 220 g with resolution of 0.1 mg was specified. The balance is placed on a separate base due to possible vibration from pumps. The equipment is controlled electronically and all necessary data are recorded directly into the PC. All data can be recorded in selected intervals to have an analysis of the entire measuring process.

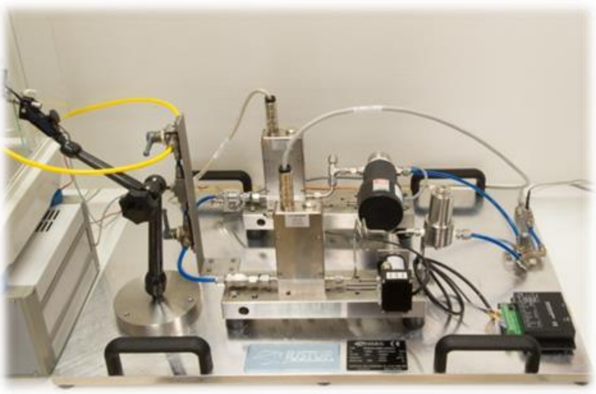

Figure 1: Mass primary standard.

The following measuring parameters and uncertainties were confirmed:

Table 1: Parameters of the standard for a very small liquid flows

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

3. Piston standard

3.1 Solution proposition

Several design issues have been addressed during the project:

- Designing the optimal piston diameter in relation to the required test flow range as well as volume required from 0,1 to 200 ml. An important question in this area was also the manufacturability in relation to the length of the piston, avoiding possible shape deformations as well as the possibility of admeasurement (gauging) in sufficient accuracy. For this reason, stainless steel 1.4404 (AISI316L) was chosen as the material for production of the very piston – made of a raw precision-drawn bar with cured surface without residual internal stress.
- 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.
- Another question was the motion screw, gearbox and servo drive. By optimizing particular parameters it was necessary to achieve a ratio of 1:2000. We chose a slack-free ball motion screw with a diameter of 12mm and a 5mm pitch of highest accuracy class IT1 with direct connection to the gearbox with servo drive via a preloaded pair of flanged nuts. A stage itself was the optimization of the servo drive motion control, setting and debugging of control parameters.
- 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. The piston is sealed with a special set of gaskets consisting of a scraper, sealing („X“ type) and a guide ring, ensuring high level of tightness.
- The piston prover is positioned in an inclined position to enquire easier removal of eventual air bubbles.
- The equipment is equipped 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 sensing with a diameter of 3mm and pressure sensing 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 at night without presence of the operator, while they can be changed or controlled remotely over remote access. All measured parameters are logged and it is possible to back evaluate particular variables, e.g. temperature stability in the piston prover, temperature stability in the room, pressure, various pulses etc.

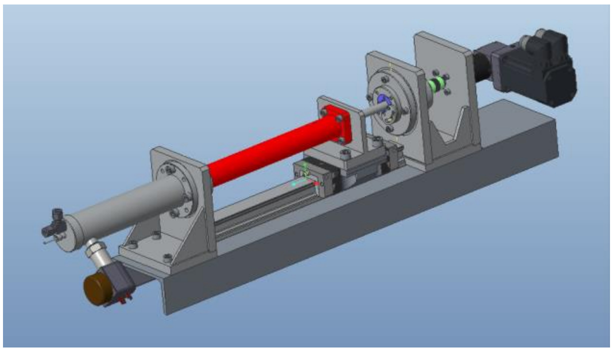

Figure 2: Designing of piston prover PP002.

3.2 Piston 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 initial concerns about the impact of roughness on the measurement have proved to be unfounded, since after polishing of the surface, roughness can’t be determined by common means. The piston is clamped to the measuring machine where the centre of clamping will be the centre of the piston. Subsequently, radiuses (distance of the clamped centre and surface of the piston) are counted using the measuring machine’s probe. 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°.

Due to the unevenness of the piston, as well as the possible deviation of the actual and selected piston centre, the individual radiuses in one plane of the admeasurement are not the same. Therefore, the cross-sectional area of the piston at the selected point is calculated as a sum of partial areas (8 areas, each bordered by two adjacent radiuses).

We calculate one part of the surface ℗ using the surface of the Archimedean screw:

$$p=\frac{\alpha_1-\alpha_2}{6}*(r_1^2+r_1*r_2+r_2^2)$$

after simplification (if delta alpha = 45°)

$$p=\frac{\pi/4}{6}*(r_1^2+r_1*r_2+r_2^2)$$

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

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

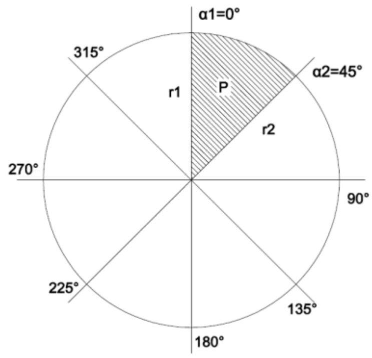

Figure 3: Piston admeasurement.

3.3 Measurement principles

The main purpose of the described standard equipment is to calibrate the largest possible range of meters. Therefore, the equipment has been deigned to allow direct measurement of flown volume, volumetric as well as gravimetric, but also to calibrate the quantity of volumetric and gravimetric flow. Some meters have the ability to connect to automatic read out, some meters need to read manually. The control software and particular applied calculations have been adapted respectively.

When applying the mass 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).

When applying the Massflow 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 distilled water.

In case the meter is connected with a volume output of flown amount or flow, a similar principle is applied - described as Volume and Volumeflow, whereby the flow volume from the piston is directly applied as the correct value.

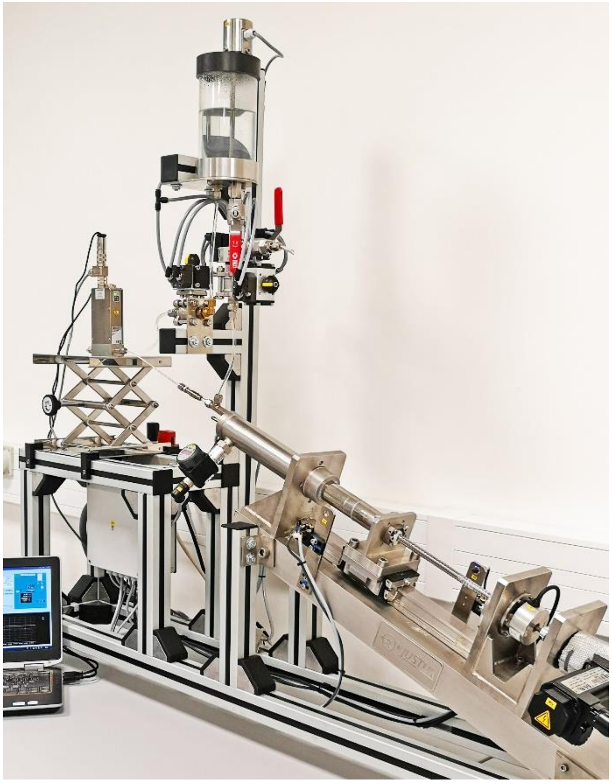

Figure 4: Piston prover standard PP002.

3.4 Measurement uncertainty

Following effects were considered by generation of mathematical models:

- Effect of water temperature change in the body of the piston. The component takes into account the effect of water temperature change ΔtP to the constant (not displaced during the course of the test) volume of water in the body of the piston prover. The change of this volume is not compensated by the application.
- Effect of pressure change to the volume of water in the body of the piston
- Effect of temperature change to the dimensions of the piston chamber shell
- Effect of temperature change to the dimensions of the piston body. The component takes into account the change in temperature of the piston body in between the start and the end of the test (i.e. storage effect). A correction of thermal volumetric expansion due to different temperature during the piston calibration and measurements is not included in this component.
- Effect of the final step size of the piston position sensor. The component takes into account the final step size of the incremental sensor for piston position at the start and end of the measurement.
- Effect of play of the mechanical connection of the helix with the piston. The component takes into account the play in between the helix and the piston body to the measured volume.
- Effect of thermal expansion of the helix. The component takes into account the change in dimension of the helix in longitudinal direction due to its thermal expansion, which affects the real longitudinal position of the piston body when measuring with the incremental sensor
- Effect of mechanical accuracy of the helix. The component takes into account the effect of the screw‘s production inaccuracy to the actual longitudinal position of the piston body when measuring with the incremental sensor.
- Effect of the piston‘s body calibration uncertainty
- Effect of water temperature difference in between the meter and standard. The component takes into account the effect of the change in density and hence of the volume flow in between the piston and the meter. The effect is compensated by the control program, so the source of uncertainty is the water temperature measurement by temperature sensors.
- Operating accuracy of the temperature sensor
- This component includes the measuring chain stability, including the temperature sensor and the analog-digital encoder.
- Effect of water pressure change in between the meter and the standard
- The component takes into account the effect of volume flow change in between the meter and the standard due to different water pressure. The control program compensates this value based on the mean value of pressures during the measurement, thus the source of uncertainty the determination of pressure by the sensors in the standard and in the meter.

- Pressure sensor operating accuracy. This component includes the stability of the measuring chain, including the pressure sensor and the analog-digital encoder.
- Pressure sensor calibration
- Effect of tubing expansion effect in between the meter and the standard

The following graph represents the overall expanded uncertainty of the equipment (k=2) depending on the testing flow and volume:

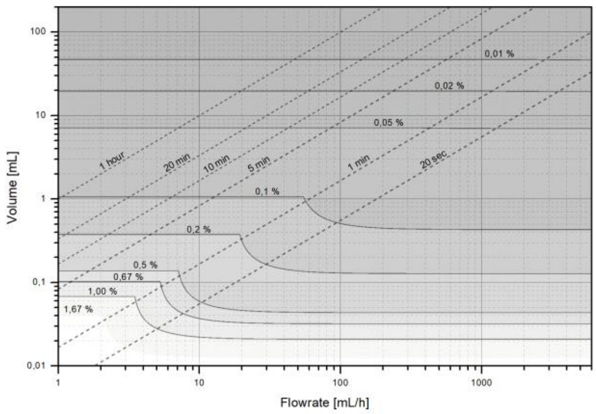

Figure 5: Expanded uncertainties values.

4. 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.
Measurements of temperature stability in the laboratory where the equipment was to be placed were conducted. Long-term measurements (10h) showed stability better than 0,07°C (Max-Min). Real measurements with the PP002 have shown a bigger temperature change during the measurement and were measured directly in the piston against the measurement in the laboratory, at the level of approx. 0,1°C. This fact is probably caused by heating up the water by mass flow meters, that where used for evaluation of results. However, such 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.

4.1 Comparative measurements with the reference standard meters

The functionality of the entire test equipment with a piston prover was inspected with reference mass flow meters Bronkhorst – Coriflow (P1, P2, P3) linked to the primary standard with weighing scales described in the text bellow and also with production calibration. These measurements were performed in the range of (1 až 6000) g/h. The resulting values are mean values from 5 to 10 repeat measurements, standard deviations are shown at each point of flow.

The figure 6 shows the relative error values of P1 (mass meter CoriFlow) in the range 550 to 6000 g/h when measured by gravimetric method at Mass primary standard with weighing scale (red line) and by Piston prover using mass method described in 3.3. (blue and green lines). The blue line shows the results where zero setting was applied only at the beginning of whole run of tests comparing the green line with zero setting at each testing point.

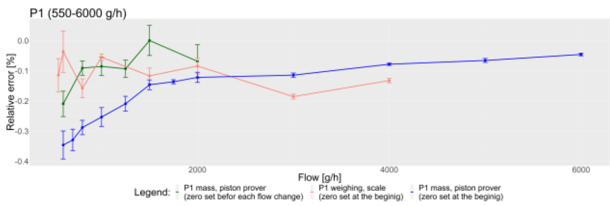

Figure 6: P1 (550-6000) g/h.

The figure 7 shows relative error values of P2 meter in the range 10 to 600 g/h. Here, all three methods - Piston prover Mass (green line), Piston prover Mass flow (blue line) and gravimetric method at Mass primary standard with weighing scale (red line) - were compared. This experiment showed a good continuity of individual measurements, especially in the range where the reference scale is very stable. Worse stability results have been shown in the Mass flow method, due shorter test times. This effect can be eliminated by optimization of test times.

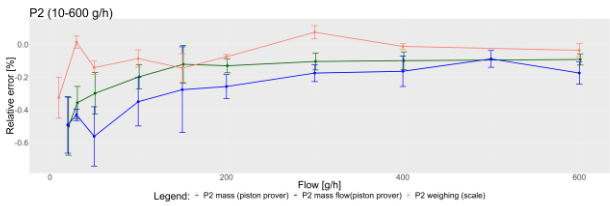

Figure 7: P2 (10-600) g/h.

The figure 8 shows the relative error values of both meters P2 and P3 in the common range (20 to 200) g/h measured by the piston standard. Here we can see P3 Mass (dark green line), P3 Mass flow (blue line) and P2 Mass method (light green line) on one graph. This experiment showed a good continuity of the individual measurements, using different measurement methods.

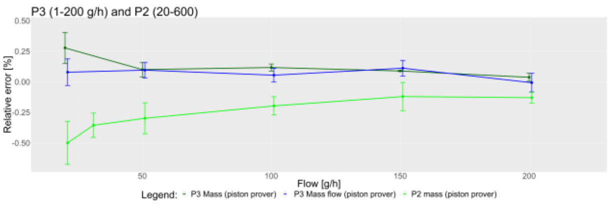

Figure 8: P3 (1-200) g/h and P2 (20-600) g/h.

The figure 9 shows the relative error values in the lower range of P3 meter (1 to 20) g/h, whereby the upper range (20-200g / h) is shown in the Figure 8. Here, two (Mass - green line and Mass flow - blue line) measurement methods were compared. In this case the worst repeatibility of Mass method mesurements has been demonstrated.

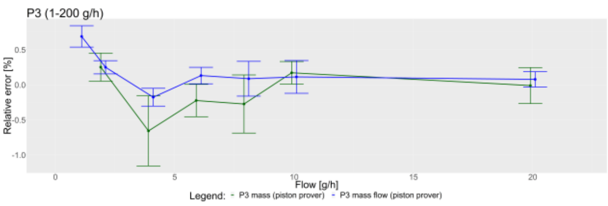

Figure 9: P3 (1-200) g/h.

6. Conclusion

Based on the presented results, it can be concluded that the chosen piston prover method for calibration of measuring instruments for a very small flows (1-6000) g/h could be considered with the expanded uncertainty less than (0,05 to 0,6)%.
The traceability chain was presented in [3].
Experimental measurements as well as practical experience to date have confirmed the original intention to implement a piston standard equipment for the use of flow meter calibrations in the area of very small flow rates while achieving high accuracy and stability of results, simple calibration, and minimizing equipment maintenance costs.
In the next period, a direct comparison of the piston to the scales is planned, thus avoiding the impact of the reference meters and further reduction of the up to now designated measurement uncertainty is expected.
The design of the equipment ensures long-term tightness stability with simple visual leak control and very high performance. Excluding some effects such as temperature stability, flow stability, evaporation, etc. has a major effect on reducing partial uncertainties, which ultimately contributes to high accuracy and stability of measurement. The piston equipment allows calibration by a more methods, so the field of tested instruments is being extended.

References

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