PRIMARY STANDARD AND TRACEABILITY CHAIN FOR MICROFLOW OF LIQUIDS

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Abstract

To enlarge calibration capacity of existing liquid flow test bench in CMI - Czech Republic to lower flows a new primary test bench using gravimetric method at atmospheric conditions is being established. The range of flow is (0,001 – 100) mL/min.
With accuracy increasing of offered flow meters, of course, requirements to reduce the uncertainty of the primary or secondary standard has been rising.
Detailed design of the CMI primary standard equipment and experience with used measuring methods are subject of the article. The main focus of the paper are gap analysis for different dynamic testing methods (weighing, volumetric with piston) as well as detailed uncertainty analysis. Also a traceability chain to the end users´ flow meters is presented.

Analysis of measurement conditions

Manufacturers usually indicate accuracy of meters ±1% to ± 2% of the measured value and this accuracy also depends on the flow. OIML and WELMEC documents requires that a calibration standard equipment should reproduce a unit of flow or delivered volume with expanded uncertainty less than 1/3 to 1/5 of the accuracy of the measuring instrument to be tested. So we can determine the expanded uncertainty of the standard equipment (0,2 to 0,6)%. A lot of sensors are integrated directly in manufacturing process or facilities and therefore it is appropriate to develop a transportable standard equipment, too.
Following effects were considered by generation of mathematical models for microflow of liquids:

- Uncertainty due to balance weighing and reading, weighing results are influenced by several factors, such as resolution and sensitivity, calibration of balance (eccentricity, linearity, repeatability), accuracy class and density of weights used for calibration,
- Uncertainty due to the determination of the density of the liquid being measured - this value may be obtained from the literature [1] or direct measurements,
- Uncertainty due to liquid temperature and its change - as it is relevant, if temperature affects the density of liquid it should be measured at each measurement,
- The uncertainty due to evaporation of liquid, what is a phase, in which the liquid changes to gas [2]. Evaporation comes at any temperature, the effect of evaporation of liquid from the weighing container during the test is significant. The intensity of evaporation can be determined by experiment or by theoretical calculation. In the case of a closed system with volumetric method as well as using standard flowmeters this effect does not occur,
- Uncertainty due to capillary effect - liquids are trying to have a shape with a minimal surface. It is geometrically sphere that has the lowest ratio of area and volume. The reason for this is the surface tension in liquids, which has its base in intermolecular forces. This phenomenon is very significant just in measuring of very low flows of liquids and causes attaching the last drop of liquid on the end of the measuring tube. Theoretical calculation of the weight of a drop using the formula for the surface tension is not accurate, because separating droplets from the pipe throat a neck is creating the first and after it has been fallen a part of its mass remains in the pipe. Therefore, it is necessary to calculate the mass of droplets using a correction factor, which, however, is variable depending on neck diameter of the measuring tube and the relevant flow [3]. At this stage of measurements and the need to repeat tests, we can estimate only volume and hence drops weight related to the measured quantity and so as much as possible to reduce and to estimate the uncertainty caused by this phenomenon,
- Uncertainty due to change of expansion pipe – volume in pipe subjects volume expansion of its own pipeline, but also due to changes of the liquid temperature,
- The uncertainty caused by the expansion of liquid - this effect actually means the volume change of the liquid due to a changing temperature between the temperature measured near the meter under test and temperature of the liquid measured in a weighing container,
- Uncertainty due to the dynamic characteristic of the switching valve, it should be determined experimentally, as far as it is relevant for a given method,
- Uncertainty due to the characteristics of the measuring instruments and measurement standards - repeatability, reproducibility, indication, calibration,
- Uncertainty due to flow oscillating and presence of air bubbles.

Proposal of standard equipment

Model of standard equipment has been designed according to Figure 1 with the following elements:
- replaceable storage tank (1) for different types of liquids,
- filter (2) for flow less than 500 g/h,
- the source of flow (3), with a pump range (10 to 600) g / h and (500-6000) g / h
- regulatory branches with the possibility of flow adjustment (4),
- mass flow meters working on the Coriolis principle in overlapping flow range (10-600) g/h and (500-6000) g/h (5) and in temperature range (0-50) ° C as reference standards,
- measuring branches for fixing of the meter under test, (6),
- balances with weighing range of 220 g (7) with an accuracy of 0,1 mg,
- digital control unit and a software evaluation device (8),
- thermometers Pt100, class 1/10B and pressure gauges (9).

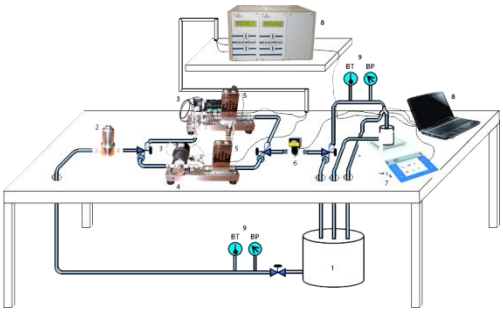


Fig. 1 Standard equipment for calibration of measuring instruments in microflow range

Implementation of standard equipment

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

Fig. 2 Standard equipment

Experiment

During the experiment, measurements were made on two reference standards. The first measurement results were very unstable. Gradually it was managed to reduce some of the uncertainty effects. Measurements were carried out separately in the range of (10 to 600) g/h, referred to the reference standard P2, and in the range (500-6000) g/h for standard P1. Analysis of the effects observed in experiment are listed below.

The impact of construction of spoiler lip

Respect to the forecast of the capillary phenomenon special tubes have been manufactured for this standard equipment – called cannula, (Fig. 3) with internal diameter 0,3 mm, 0,6 mm, 1,2 mm and 2,5 mm according to Figure 5. The number of drops (Fig. 4) was determined from drops weighing and their recording by use each cannula diameter.

Figure 3 Cannulas and their involvement

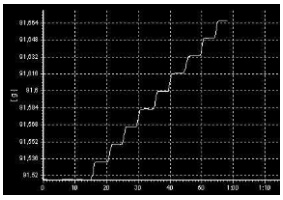


Figure 4 Drops recording

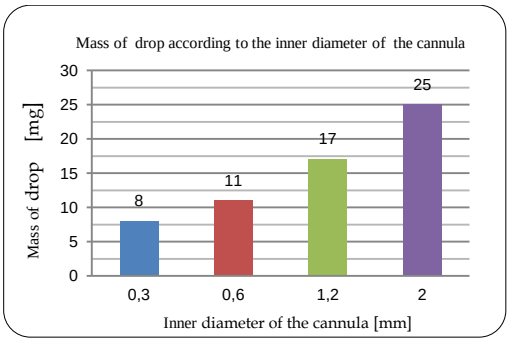


Figure 5 Mass of drops

Constriction of a hole at the end of the measuring tube increases pressure and thus it was established experimentally which cannula should be used (Table 2).

Evaporation

The experiment detected a large impact of measured quantity and time of the test due to evaporation. Because of pumps and mass flow meters warming during the tests the whole standard equipment could not be placed in a protective case, what was considered

the first. The balance space could not be covered, too and therefore on the basis of the measurements with different weighing containers (Figure 6) was achieved the smallest evaporation effect using the closed container with a small hole only (Figure 7).

Figure 6 Weighing containers

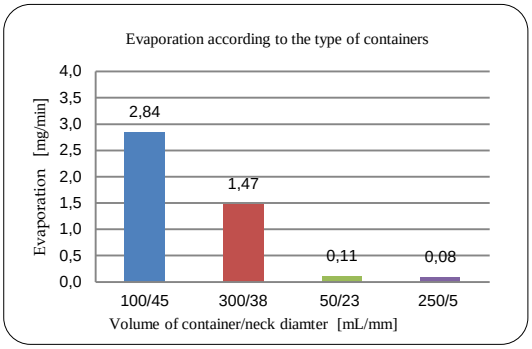


Figure 7 Evaporation impact due to varied containers

Another source of evaporation is by using a test liquid. Measurements were made with three kinds of liquids (Table 1). Evaporation was tested for all liquids under the same conditions and results are shown in Figure 8. Significant effect of evaporation was observed at the lowest flow measurements (Table 2), that was observed experimentaly. The oil Shell is the most appropriate liquid for reduction of evaporation. The design of standard equipment makes it possible. Up to now we have used water only and experimentally observed effect of evaporation was accounted as a correction to the measurement result.

Table 1 Identification of liquids

	Distilled water	**Nonan**	**Oil Shell**
Identification	H_2O	C9H20	Shell Morlina
View	colourless liquid	colourless liquid	colourless mineral oil
Density by 20 °C	0,998 g/cm^3	0,718 g/cm^3	0,850 g/cm^3
Kinematic viscosity	0,01004 mm^2 s^{-1}	-	5,0 mm^2 s^{-1}
Solubility in water	-	insoluble	insoluble
Combustibility	nonflammable	flammable	flammable

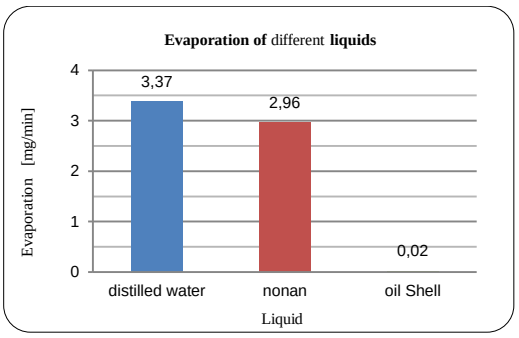


Figure 8 Evaporation impact due to varied liquids

Table 2 Impact of evaporation of distilled water and the uncertainty influence of drops due to flow and used cannula

Flow	**g/h**	**10**	**150**	**400**	**1500**	**3000**
The inner diameter of the cannula	mm	0,3	0,3	0,6	1,2	2
Delivered mass	g	5	20	40	80	160
Evaporation	%	0,097	0,004	0,003	0,0008	0,0004
Drop uncertainty	%	0,159	0,040	0,027	0,021	0,005

Stability of flow

Flow were recorded at one-second intervals for all experimental measurements. It was not possible to avoid rapid increase of flow at start and end of the test due to standing start method and oscillation during tests. Figure 4 and 5 represents the flow at 10 g/h and 600 g/h. This oscillation does not have significant influence by standing start method, when delivered volume is compared. It is not possible to consider the mean flow value as a standard value in this case.

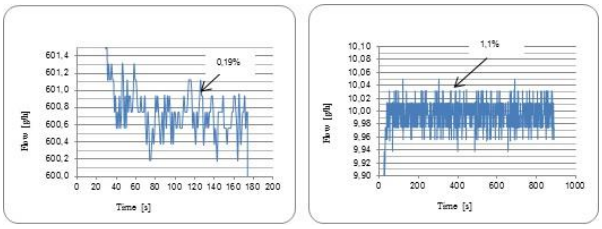


Fig. 9 Flow stability

Stability of the reference standard

The analysis of the tests showed that the flowmeter is suitable for use as a reference standard. It is necessary to properly carry out its internal calibration, called "Zero calibration". This phenomenon is known from measurements of larger flows, too. It was experimentally found a big influence, if zero calibration has not been done.

The mass flowmeter also allows the determination of a liquid density being measured. Manufacturers specified accuracy of the density of 5 kg/m^3. This accuracy is insufficient for reference standard and it is necessary to determine the density according to the density calculation based on temperature measurements or directly by other appropriate method.

Temperature was measured during the experiment at three places. At the entrance to the measuring lines, close to the balance and in reference standards. At inlet and outlet of the measuring lines were used 3-wire Pt100, class 1/10B, sensor 2 mm diameter and 11 mm length. During the measurement, there has been recorded an increase in temperature in the reference standard due to its measuring principle. Since mass units were compared, this does not affect the measurement result.
Since the pressure of water was found from 0,2 bar to 0,5 bar, pressure corrections are not considered in the calculation.

Calibration

Relative errors were calculated for repeating measurements at selected points of flow according to the equation (1):

$$e = \frac{m_m - \left[m_{e2} - m_{e1} + \left(m_{e2} - m_{e1}\right)\frac{q_o}{q_s} \cdot 3600 \right].k_v}{\left[m_{e2} - m_{e1} + \left(m_{e2} - m_{e1}\right)\frac{q_o}{q_s} \cdot 3600 \right].k_v} \cdot 100 \qquad (1)$$

where m_m is delivered mass determined by reference standard in [g],

m_{e1}, m_{e2} is mass on balance at the start or at the end of tests in [g] ,

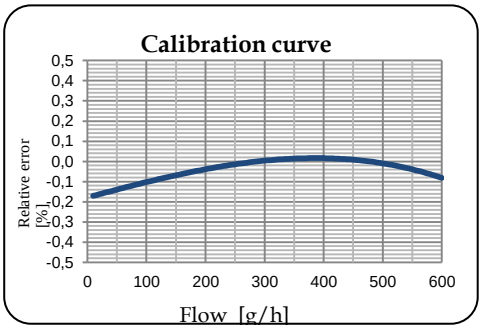
q_o is intensity of evaporation in [g/s],

q_s is the mean flow during the test in [g/h]

k_v is the correction for buoyancy by weighing.

Results of the calibration are calibration curves of the relative error e depended on the flow q_m are shown in the equation (2) and Figure 10 (standard P2):

$q_m(P2)$ = -0,0000000014*e^3 - 0,0000002274*e^2 +



0,0008028470*e - 0,1790231650 (2)

Fig 10 Calibration curve of reference standard P2

For the calibrated range expanded uncertainty for each flow were calculated (Table 3).

Table 3 Relative error and expanded uncertainty of the standard P2

Flow	g/h	10	30	50	100	150	300	400	600
Relative error	%	-0,33	0,01	-0,14	-0,09	-0,14	-0,07	-0,01	-0,04
Standard uncertainty type A	%	0,13	0,09	0,04	0,05	0,09	0,04	0,02	0,02
Standard uncertainty type B	%	0,25	0,06	0,06	0,13	0,07	0,05	0,08	0,05
Expanded uncertainty	%	0,56	0,22	0,15	0,28	0,23	0,13	0,16	0,11

Linear regression analysis

The measured results were evaluated by the method of linear regression analysis, too. This method brings uncertainties, which are negligible in relation to other uncertainties of the uncertainty budget. Determination of this uncertainty was done according to [4]. Linear regression analysis is a tool for estimation of conventional true values x_i and the average values of the calibrated meter data y_i. Application to the experimental measurement - calibration of reference standards by the weighing method with balance, we received equation for uncertainty evaluation (3):

$$u^2(m_e) = \frac{U^2}{n} + \frac{s^2}{b_1^2}\left[\frac{1}{j} + \frac{1}{n} + \frac{\left(m_m - \overline{m}_m\right)^2}{b_1^2 \sum_{i=1}^{n} \left(m_e - \overline{m}_e\right)^2} \right] \qquad (3)$$

where U is expanded uncertainty of measurement

n is number of measurements repeating

s^2 is variance of the residuals given by regression analysis,

b_1 is a coefficient obtained by the method of least squares,

j is number of repetitions in one point of flow,

m_e is delivered mass during the test on balance in [g],

m_m is delivered mass during the test through the meter under test in [g],

Fig. 11 shows the results of determining the uncertainty from the residual variance of the linear regression analysis $u(m_e)$ according to the mass m_e. The minimum value of this uncertainty is by the mass of 30 grams, the maximum at 80 grams. Uncertainty is dependent on the number of repeated measurements j. To compare the maximum uncertainty from the residual variance to uncertainty budget by weighing method we receive the ratio 0,8. The ratio of the minimum uncertainty and standard uncertainty is 0,6. Therefore uncertainties arising from the residual variance should be considered and included into the appropriate uncertainty budget.

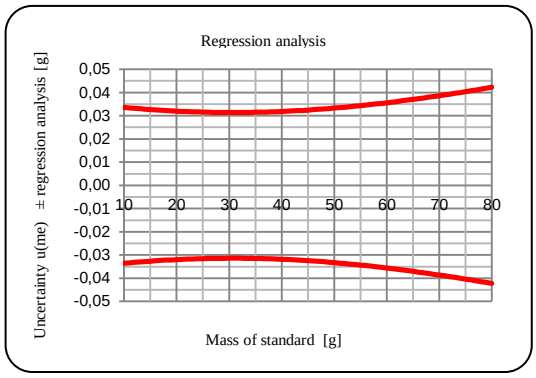


Fig. 11 Regression analyse applied to the reference standard P2

Traceability chain

In connection with the proposed scheme it is necessary to take into account flow range of reference standards and their types. Furthermore, it is necessary to select the minimum number of transmissions between primary standards and measuring instruments. The following scheme is considered with primary standards based on balance and volume methods based on piston prover at the same level, that could be compared. Reference (transport) standards could be used in relation to the meters.

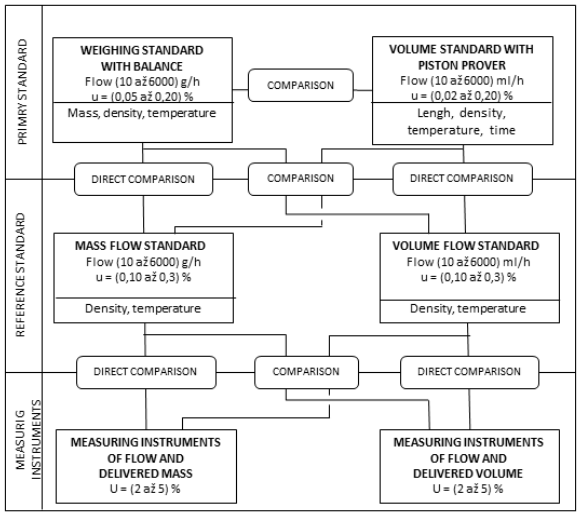


Fig. 12 Traceability chain

Uncertainty evaluation

Uncertainty analysis for varies measuring methods showed differences in the used methods. Uncertainty budgets analysed numerically were compared to model at the same or very similar conditions. Table 4 lists the standard uncertainties of type B. On the basis of these values there can be assumed that the expanded uncertainty for the area miniflowrates and microflowrates will meet the required uncertainty (0,2 to 0,6)%.

Table 4 Measuring methods and estimated uncertainty

Method	Standard uncertainty type B [%]
Weighing method – balance – flowmeter with the output in volume units	0,162
Weighing method – balance – flowmeter with the output in mass units	0,156
Weighing method – mass flowmeter - flowmeter with the output in volume units	0,603
Weighing method – mass flowmeter - flowmeter with the output in volume units	0,270*)
Volume method – piston – flowmeter with the output in volume units	0,050

*) An external thermometer was used for density determination

Summary

Based on the presented results, it can be concluded that the chosen weighing method for calibration of measuring instruments for small flow rates (10-6000) g/h could be considered with the expanded uncertainty less than (0,2 to 0,6)%.

Theoretical analysis of the uncertainties and the results of the experiment showed, that the advantage for microflowrates measurements would be to exclude the impact of evaporation of liquid, capillary effect and the impact of environmental conditions, if we compare these effects to the higher flows. Effect of evaporation can be solved with low-gradient liquid evaporation, for example Shell oil. Since it is assumed the calibration of medical instruments, we prefer distilled water. Development of piston prover with a servomotor and therefore closed circuit appears as good solution. In addition instead of the impact of evaporation, capillary effect and environmental conditions the impact of uncertainty caused by the of liquid density conversion would be eliminated. We assume also better flow stability.

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