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Comparison of the primary low gas flow standards between MIKES and METAS

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Abstract

At Centre for Metrology and Accreditation (MIKES), a primary calibration system for gas mass flows between 0.42 mg/s and 625 mg/s is based on dynamic weighing providing traceability directly to the national mass and time standards. To evaluate the agreement of the system with a volumetric primary calibrator, the international comparison between MIKES and the Swiss Federal Office of Metrology and Accreditation (METAS, Switzerland) was carried out. At METAS, the primary low gas flow standard provides traceability to the Swiss national measurement standards for length and time. The results of the comparison showed that the two systems do not deviate more than $\pm 0.15\%$ in the mass flow range 0.42–625 mg/s.

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1. Introduction

In Finland, the need for accurate low gas flow measurements has been grown in many areas, for example in the pharmaceutical and semiconductor industry or for air pollution measurements. To establish the traceability links for these measurements, a calibration system for gas flow meters has been developed at the Centre for Metrology

and Accreditation (MIKES) since 2002 [1,2]. The system provides traceability of gas flow measurements to the national mass and time standards.

At METAS the primary calibration system for low gas flows is volumetric, thus traceability has been established to the length and time standards. The METAS primary system has been formerly compared with a low gas flow standard at Laboratoire National d'Essais (LNE, France) [3].

In this paper, a comparison between the calibration system at MIKES and METAS is reported. A commercial calibrator based on laminar flow elements (LFE) was used as a transfer standard in the comparison. The LFE-system was calibrated at MIKES and then transported to METAS in the

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end of April 2003. After measurements, the transfer standard was returned back to MIKES and calibrated again at the beginning of July.

2. The MIKES dynamic weighing system

2.1. Description

The dynamic weighing system (DWS) is located on a table in an air-conditioned laboratory. There are no windows in the laboratory and vibrations are kept at minimum. The balance is a commercial mass comparator with capacity of 10,100 g and a resolution of 1 mg, located on a stone plate in a draft shield.

For air density calculations, ambient air temperature, pressure and humidity are measured with thermistors connected to a digital multimeter (DMM), digital hygrometer and barometer (calibrated at MIKES). The volume of the gas cylinder is 7 dm³. When it is filled to the maximum working pressure, 5 MPa, the mass of the cylinder is 10 kg. The line pressure regulator is located outside the draft shield after the PTFE connecting tube (i.d. 1 mm, o.d. 2 mm).

A PC collects measuring data from the digital multimeter and the device under test (DUT). The measurement time is typically from 250 s to 10,000 s. Software for data acquisition has been developed at MIKES.

Three digital mass flow controllers (DMFC) with different nominal flow rates are used for controlling

the gas flow rates (see Fig. 1). At flow rates higher than 30 mg/s, the gas flows through a vessel with a volume of 1 dm³ to prevent fluctuations in the readings of DMFCs. The flow-controlling unit can be placed up- or downstream of the DUT. The calibration set-up for this comparison has been shown in Fig. 1.

The mass flow is determined by linear fitting of the air buoyancy corrected balance readings over time. This allows the evaluation of gas flow and balance stability afterwards using the calculated mass residuals. The proper operation of the DWS was checked with reference weights and reference mass flows.

Between two calibrations at MIKES, the primary standard was modified and transferred to the air-conditioned laboratory. Manual needle valves in a flow-controlling unit were replaced with DMFCs. After modifications, the expanded uncertainty ($k = 2$) of the MIKES DWS was dropped off from 0.8% to 0.3% in the flow range 0.42 mg/s to 2 mg/s and from 0.4% to 0.2% in the flow range 2–625 mg/s.

2.2. Uncertainty analysis

During measurements, the net force F acting on the weighing pan is

$$F = m_{cg} - \rho_a g V_0 + \delta F_U + \delta F_{\text{conv}}, \quad (1)$$

where m_c , ρ_a , V_0 , δF_U , δF_{conv} are the mass of the gas cylinder, density of air, volume of the gas cylinder, parasitic force caused by the PTFE connecting tube

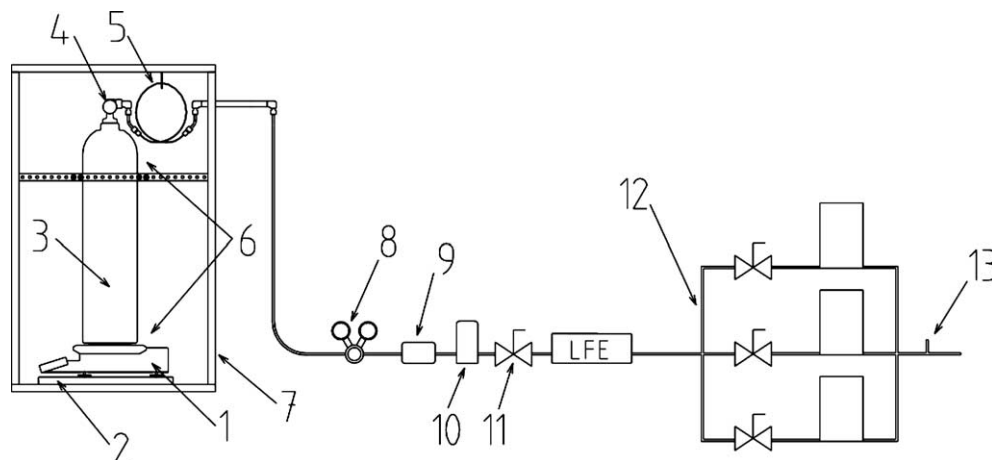


Fig. 1. MIKES dynamic weighing system and the calibration set-up with laminar flow elements used for the comparison. 1. Balance, 2. stone plate, 3. aluminium gas cylinder, 4. shut off valve, 5. PTFE connecting tube, 6. temperature sensor, 7. draft shield, 8. two stage pressure regulator, 9. filter, 10. single stage pressure regulator, 11. shut off valve, 12. gas flow controlling unit, 13. temperature sensor.

and force caused by natural convection, respectively. From Eq. (1) we can derive an equation for m_c

$$m_c = L + \delta L + \rho_a V_0 - \delta m_U - \delta m_{\text{conv}}, \quad (2)$$

where L is the balance indication and δL the correction due to the non-ideal behaviour of the balance. The acceleration of gravity is supposed to be constant and its uncertainty insignificant. Thus an equation for \dot{m} is

$$\dot{m} = \dot{L} + \delta \dot{L} + V_0 \dot{\rho}_a + \rho_a \dot{V}_0 - \delta \dot{m}_U - \delta \dot{m}_{\text{conv}} \quad (3)$$

and

$$\begin{aligned} \dot{m} &\approx \frac{1}{\Delta t} (\Delta L + \Delta \delta L + V_0 \Delta \rho_a + \rho_a \Delta V_0 - \Delta \delta m_U \\ &\quad - \Delta \delta m_{\text{conv}}) \\ &\equiv G(\Delta L, \Delta \delta L, V_0, \Delta V_0, \rho_a, \Delta \rho_a, \Delta \delta m_U, \Delta \delta m_{\text{conv}}, \Delta t), \end{aligned} \quad (4)$$

where ΔL , ΔV_0 , $\Delta \rho_a$ are the measured gas mass loss during the measurement process, calculated change in the volume of the gas cylinder and the change in air density, respectively. Assuming that variables are independent to each other, the combined standard uncertainty can be calculated in the following way [4]

$$\begin{aligned} u_c^2(\dot{m}) &= \sum_{i=1}^9 c_i u^2(y_i) = \sum_{i=1}^9 \left(\frac{\partial G}{\partial y_i} \right)^2 u^2(y_i) \\ y_i &\in \{\Delta L, \Delta \delta L, V_0, \Delta V_0, \rho_a, \Delta \rho_a, \Delta \delta m_U, \Delta \delta m_{\text{conv}}, \Delta t\}. \end{aligned} \quad (5)$$

Table 1 shows the uncertainty budgets at gas mass flows of 0.42 mg/s, 300 mg/s and 625 mg/s, respectively. $u(\Delta L)$ is the uncertainty due to flow stability estimated as a deviation of mass residuals. The uncertainty of the mass loss from the weighed gas cylinder $u(\Delta \delta L)$ has been estimated to be 5 mg at maximum. The uncertainty of the cylinder volume $u(V_0)$ was 0.58 dm³. The change in the volume of the gas cylinder $u(\Delta V_0)$ due to pressure drop was determined taking into account the magnitude of gas flow and the measuring time. Because the measuring computer program causes drift to time measurements, the uncertainty of the measuring event time $u(\Delta t)$ was 0.2 s. The reference weights were used for investigating the effect of the connecting tube δm_U . The magnitude of the force resulting from natural convection had been studied theoretically and experimentally [1]. The resulting standard uncertainty was 0.2 mg/s at worst.

Table 1

Uncertainty budgets for the MIKES primary gas flow standard at gas flow rates of 0.42 mg/s, 300 mg/s and 625 mg/s, respectively

y_i	Unit	0.42 mg/s			300 mg/s			625 mg/s		
		c_i	$u(y_i)$	$c_i u(y_i)$	c_i	$u(y_i)$	$c_i u(y_i)$	c_i	$u(y_i)$	$c_i u(y_i)$
$u(\Delta L)$	kg	1.00×10^{-4}	5.00×10^{-6}	5.01×10^{-10}	4.10×10^{-3}	5.00×10^{-6}	2.05×10^{-8}	4.01×10^{-3}	5.00×10^{-6}	2.01×10^{-8}
$u(\Delta \delta L)$	kg	1.00×10^{-4}	1.94×10^{-6}	1.94×10^{-10}	4.10×10^{-3}	3.04×10^{-5}	1.25×10^{-7}	4.01×10^{-3}	4.06×10^{-5}	1.63×10^{-7}
$u(V_0)$	m ³	4.47×10^{-8}	5.77×10^{-4}	2.58×10^{-11}	5.05×10^{-6}	5.77×10^{-4}	2.91×10^{-9}	9.45×10^{-6}	5.77×10^{-4}	5.45×10^{-9}
$u(\Delta V_0)$	m ³ /s	1.18×10^{-4}	3.06×10^{-7}	3.63×10^{-11}	4.84×10^{-3}	2.30×10^{-6}	1.11×10^{-8}	4.76×10^{-3}	4.78×10^{-6}	2.28×10^{-8}
$u(\rho)$	kg/m ³	5.31×10^{-11}	1.46×10^{-3}	7.76×10^{-14}	1.63×10^{-8}	1.46×10^{-3}	2.38×10^{-11}	3.33×10^{-8}	1.46×10^{-3}	4.86×10^{-11}
$u(\Delta \rho)$	kg/m ³	1.06×10^{-6}	3.92×10^{-6}	4.17×10^{-12}	4.34×10^{-5}	4.81×10^{-5}	2.09×10^{-9}	4.26×10^{-5}	1.02×10^{-4}	4.36×10^{-9}
$u(\Delta t)$	s	4.20×10^{-11}	2.00×10^{-1}	8.39×10^{-12}	1.28×10^{-6}	2.00×10^{-1}	2.57×10^{-7}	2.48×10^{-6}	2.00×10^{-1}	4.97×10^{-7}
$u(\Delta \delta m_U)$	kg	1.00×10^{-4}	1.00×10^{-6}	1.00×10^{-10}	4.10×10^{-3}	1.00×10^{-6}	4.10×10^{-9}	4.01×10^{-3}	6.00×10^{-6}	2.41×10^{-8}
$u(\Delta \delta m_{\text{conv}})$	kg	1.00×10^{-4}	2.04×10^{-13}	2.04×10^{-17}	4.10×10^{-3}	3.06×10^{-12}	1.25×10^{-14}	4.01×10^{-3}	6.37×10^{-12}	2.56×10^{-14}
u_c	kg/s		5.48×10^{-10}				2.87×10^{-7}			5.24×10^{-7}
$U (k=2)$	%		0.26				0.20			0.16

3. The METAS volumetric standard for low gas flows

METAS is operating with a volumetric standard for gas flows in the range of 3 cm³/min to 30 dm³/min based on three glass tubes and mercury sealed pistons since 1994. Various improvements were necessary to achieve and even exceed the minimum requirements of 0.2% for the expanded uncertainty ($k = 2$) [5]. The most important of them were the use of diode laser interferometers for the dynamic measurement of the piston position, the use of stable thermistors for the gas temperature measurement at different sites and the replacement of the entire electronics and software. Furthermore a strict separation of any heat dissipating parts and the use of pneumatically driven valves were necessary to minimise temperature gradients within the measurement chamber that is stabilised to ± 0.1 °C.

The experimental setup and the uncertainty evaluation have already been described in detail in [3]. For the big and the medium tube that were used for the calibrations, the best measurement capabilities are 0.13% relative expanded uncertainty under the conditions that the minimum measuring time is 15 s and the minimum measuring distance is 300 mm. Other characteristic values are the relative repeatability standard deviation that is normally smaller than 5×10^{-5} the flow generation included, and the relative inter-tube reproducibility that is smaller than 1.5×10^{-4} .

For the sake of direct comparability, the volume flows at standard conditions expressed in cm³/min have been transformed to mass flows expressed in mg/s using a gas density (N₂) at standard conditions (0 °C, 101.325 kPa) of 1.25053 mg/cm³.

4. The transfer standard

In the comparison, a flow terminal with two LFEs was used as a transfer standard. The full-scale rates of the elements were 20.8 mg/s and 625 mg/s. The laminar flow elements and flow terminal (mol-box1, version 5.00f) were manufactured by DH Instruments. During the calibrations at MIKES and METAS, the upstream pressure before the LFE was 270 ± 1 kPa and 270 ± 0.5 kPa, respectively. Each LFE was calibrated at 12 points.

To monitor the stability of the transfer standard, the pressure transducers of the mass flow terminal were calibrated at MIKES Pressure Laboratory four times between October 2002 and June 2003. The calibration results of the upstream pressure transducer are shown in Fig. 2. Fig. 3 shows the difference between the up- and downstream pressure transducers.

As shown in Fig. 2, the error of the upstream pressure sensor has been increased during the exercise. For example at the point 283 kPa, the error increased by 9 Pa between the last two calibrations.

Calibration results of the pressure transducers show that there was a significant drift in both

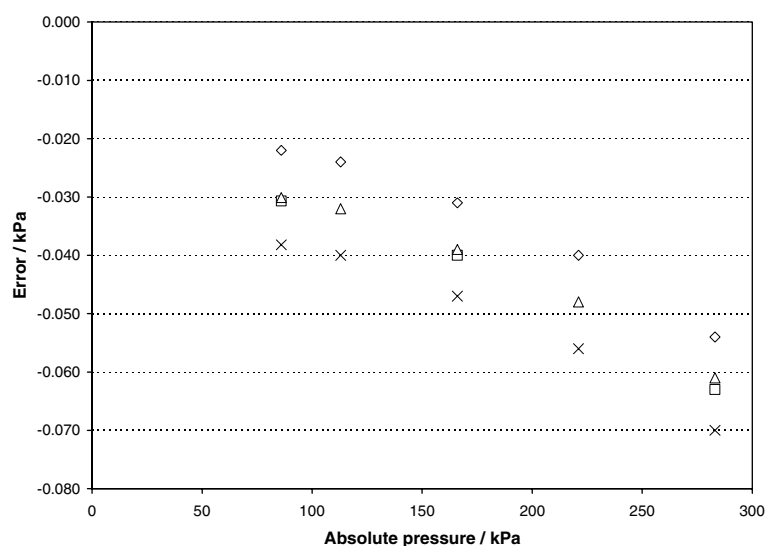


Fig. 2. The error of the upstream pressure transducer of the transfer standard. □ = October 2002, ◇ = January 2003, Δ = February 2003, × = June 2003.

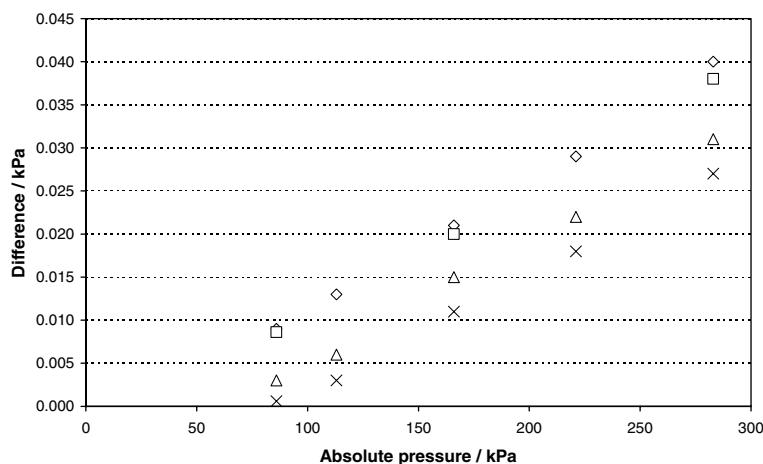


Fig. 3. The difference of the pressure readings between the up- and downstream pressure transducers of the transfer standard. \square = October 2002, \diamond = January 2003, Δ = February 2003, \times = June 2003.

sensors. Therefore, a proper compensation of the pressure transducer readings at the operating pressure is necessary before starting the measurements. That was executed with the tare function of the flow terminal. The estimated maximum drift during a single measurement set was 1 Pa and was negligible for the measurement result.

5. Measurement results

5.1. Calibration of the low flow range

Fig. 4 shows the measurement results obtained with the low range laminar flow element. The first MIKES calibration was performed using the older

version of DWS, which caused a significant difference between MIKES and METAS at the lowest measurement points. The difference was mainly due to the unstable gas flow at MIKES in the range from 0.42 mg/s to 5 mg/s. Also changes in ambient conditions and vibrations at MIKES had affected to the results.

When comparing results obtained with the improved version of the DWS to those obtained with the METAS system, the relative difference between the reference and the transfer standard (Δq_r) at the flow range from 0.42 mg/s to 20.8 mg/s were between -0.09% and 0.06% . The relative corrections to the indications of the mass flow terminal were between -0.07% and 0.27% .

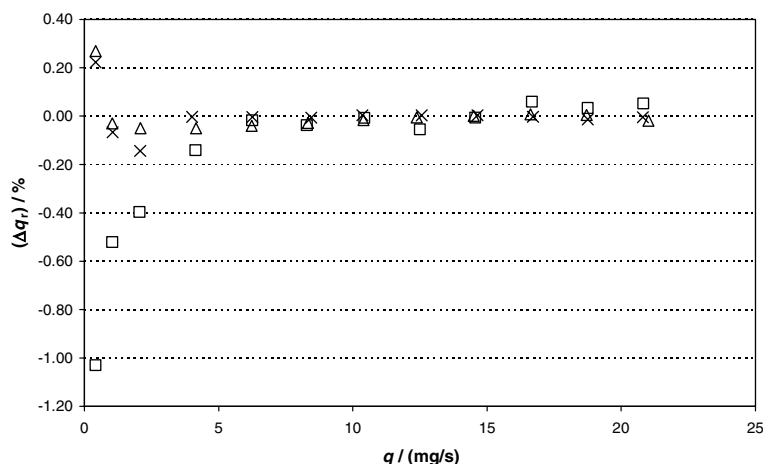


Fig. 4. Relative difference between the reference and the transfer standard (Δq_r) measured by MIKES and METAS at the measurement points (q) 0.42 mg/s to 20.8 mg/s. \square = MIKES April 2003, Δ = METAS May 2003, \times = MIKES June 2003.

5.2. Calibration of the higher flow range

Fig. 5 shows the calibration results obtained with the laminar flow element with nominal flow rate of 625 mg/s. The relative difference between the reference and the transfer standard at the flow range from 20.8 mg/s to 625 mg/s were between -0.15% and 0.15% . The relative corrections to the indications of the mass flow terminal were between -0.28% and 0.03% .

5.3. Degrees of equivalence

Between the two calibration sets at MIKES improvements were made to the DWS which fixed the problems caused by unstable gas flow and ambi-

ent conditions and lowered the measurement uncertainty, too. So, only the results of the second MIKES calibration set were compared with METAS results. The first calibration set at MIKES was used for checking the measurement uncertainty evaluations.

The degree of equivalence between two laboratories at each measurement point ($i, j = 1, \dots, 24$) was calculated as described in [6]

$$d_{ij} = \Delta q_{ri} - \Delta q_{rj}, \quad (6)$$

where subscripts i refer to measurements carried out at MIKES and subscript j at METAS. The associated expanded uncertainty is $U_{ij} = 2u(d_{ij})$

$$u^2(d_{ij}) = u^2(\Delta q_{ri}) + u^2(\Delta q_{rj}) \quad (7)$$

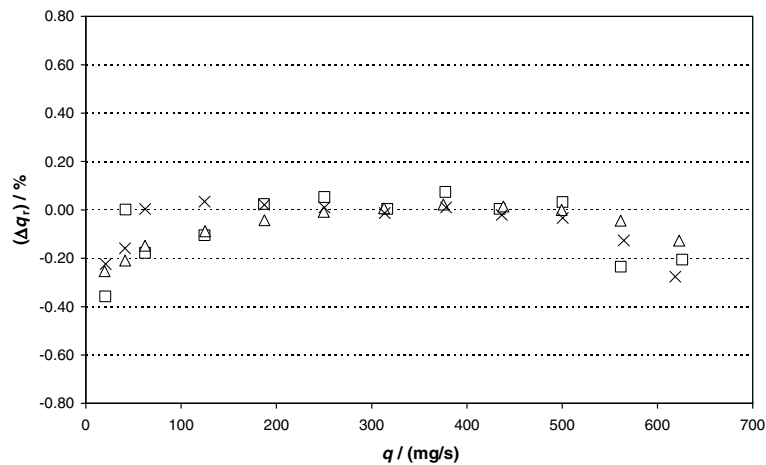


Fig. 5. Relative difference between the reference and the transfer standard (Δq_r) measured by MIKES and METAS at the measurement points (q) 20.8 mg/s to 625 mg/s. \square = MIKES April 2003, Δ = METAS May 2003, \times = MIKES June 2003.

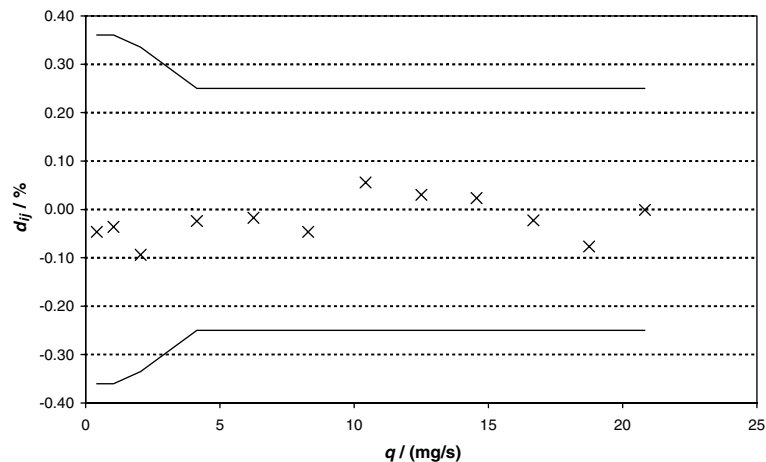


Fig. 6. Degrees of equivalence (\times) and its uncertainties (—) in the flow range from 0.42 mg/s to 20.8 mg/s.

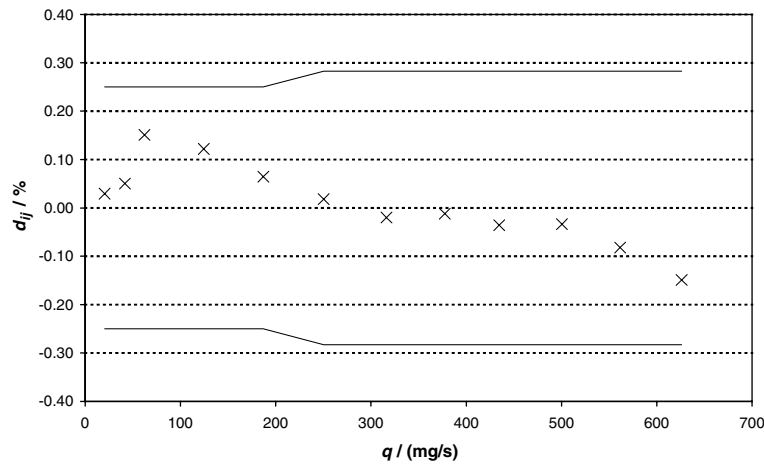


Fig. 7. Degrees of equivalence (×) and its uncertainties (—) in the flow range from 20.8 mg/s to 625 mg/s.

Table 2

The results of the comparison in the flow range from 0.42 mg/s to 20.8 mg/s

Flow/ (mg/s)	Δq_r (MIKES)/%	Δq_r (METAS)/%	$d_{ij}/\%$	$U(d_{ij})/\%$	D_n
0.42	0.22	0.27	−0.05	0.36	−0.13
1.0	−0.07	−0.03	−0.04	0.36	−0.10
2.1	−0.14	−0.05	−0.09	0.34	−0.28
4.1	−0.07	−0.05	−0.02	0.34	−0.07
6.3	−0.06	−0.04	−0.02	0.25	−0.07
8.3	−0.07	−0.03	−0.05	0.25	−0.19
10.4	0.04	−0.02	0.06	0.25	0.22
12.5	0.03	−0.01	0.03	0.25	0.12
14.6	0.03	0.00	0.02	0.25	0.09
16.7	−0.02	0.01	−0.02	0.25	−0.09
18.8	−0.07	0.00	−0.08	0.25	−0.31
20.8	−0.02	−0.02	0.00	0.25	0.0

Table 3

The results of the comparison in the flow range from 20.4 mg/s to 625 mg/s

Flow/ (mg/s)	Δq_r (MIKES)/%	Δq_r (METAS)/%	$d_{ij}/\%$	$U(d_{ij})/\%$	D_n
20.4	−0.22	−0.25	0.03	0.25	0.12
41.7	−0.16	−0.21	0.05	0.25	0.20
62.2	0.00	−0.15	0.15	0.25	0.60
125	0.03	−0.09	0.12	0.25	0.49
187	0.02	−0.04	0.06	0.25	0.26
250	0.01	−0.01	0.02	0.28	0.06
316	−0.01	0.01	−0.02	0.28	−0.07
377	0.01	0.02	−0.01	0.28	−0.04
435	−0.02	0.01	−0.04	0.28	−0.13
500	−0.03	0.00	−0.03	0.28	−0.12
562	−0.13	−0.04	−0.08	0.28	−0.29
626	−0.28	−0.13	−0.15	0.28	−0.53

and for the normalised difference

$$D_n = d_{ij}/U_{ij}. \quad (8)$$

The degrees of equivalence and uncertainties $U(d_{ij})$ at the comparison range are shown in Figs. 6 and 7. Tables 2 and 3 show the results of the comparison in the numerical format.

6. Conclusions

The comparison between MIKES and METAS was carried out to compare two primary gas flow standards based on two different principles. At MIKES, the traceability of gas flow measurements is realised with the dynamic weighing method whereas at METAS the traceability is based on the volumetric method. The comparison was carried out using a commercial LFE-system as the transfer standard. The obtained results showed a good agreement between these two gas flow standards.

When comparing the results obtained with the former version of the MIKES DWS, there was a difference of 1.3% at maximum between MIKES and METAS in the flow range between 0.42 mg/s and 5 mg/s. It was mainly due to unstable gas flow, varying ambient air conditions and vibrations of the ground transmitted through the balance base. During calibrations at METAS, MIKES standard was improved and installed in an air-conditioned laboratory. After modifications, the relative differences between MIKES and METAS calibrations were between −0.09% and 0.06% in the flow range 0.42–20.8 mg/s and between −0.15% and 0.15% in the gas flow range 20.8–625 mg/s.

The smallest flows for both laminar flow elements were below 10% of their nominal flows. At these operating conditions, the reproducibilities of both elements are lower than in the range 10–100%. Despite of this, the achieved results were good even for the lower calibration points.

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