CIPM Low-Pressure Gas Flow Key Comparison

(CCM.FF-K6.2017)

Comparison of Primary Gas Flow Standard

Spanning the Range from 2 mL/min to 10 L/min

Final Report

Pilot
CMS (Chinese Taipei)

Participants
NIST (United States of America)
KRISS (Republic of Korea)
NMIA (Australia)
NMIJ/AIST (Japan)
CMI (Czech Republic)
INRIM (Italy)
LNE (France)
METAS (Switzerland)
PTB (Germany)

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September 2021

Abstract

The CCM.FF-K6.2017 comparison was organised for the purpose of determination of the degree of equivalence of the national standards for low-pressure gas flow measurement over the range 2 mL/min to 10 L/min. Four molbloc-L flow elements and a molbox1+ were used as the transfer standards.

Ten laboratories from three RMOs participated between August 2017 and January 2020 – EURAMET: INRIM (Italy); LNE (France); PTB (Germany); METAS (Switzerland); CMI (Czech Republic); SIM: NIST (USA); APMP: NMIJ/AIST (Japan); KRISS (Korea); NMIA (Australia); CMS (Chinese Taipei). The measurements were provided at prescribed reference pressure and temperature conditions. All results were used in the determination of the key comparison reference value (KCRV) and the uncertainty of the KCRV. The reference value was determined at each flow separately following procedure presented by M. G. Cox [1]. The degree of equivalence with the KCRV was calculated for each flow and laboratory. This KCRV can now be used in the further regional comparisons.

Revision History

Date	Revision Ver.	Changes
2020/06/18	Draft A ver.2	1. Add the section of revision history 2. Update the METAS' reported error of the transfer standard and the lab uncertainty (U_{base}) at nominal flow rates 2 mL/min and 5 mL/min due to pilot lab's mistake. 3. Remove NMIJ's test result at nominal flow rates 2 mL/min to 25 mL/min due to NMIJ's request. 4. Revised of METAS and NMIJ in Figure 6 - Measurement methods of participants 5. Add Annex B: The detailed procedure of consistency check 6. Add Annex C: The re-test data from NMIJ 7. Add Annex D: Comparison pass/fail/inconclusive criterion
2020/12/07	Draft A ver.3	1. Update the test facilities information of LNE, PTB and CMI in Annex A 2. Add Annex E: 2 mL/min data analysis by Median of Absolute Deviations (MAD)
2021/02/17	Draft B ver.1	1. Revised the Table 2, Table 8 of the original NMIJ results at 25 mL/min, 10 mL/min and 5 mL/min showing to meet the CIPM comparison guidelines. Add the note to describe the symbol in Table 2, 7, 8, 9, 10. 2. Revised 5.4 chapter, add "The reproducibility was calculated by taking the standard deviation of the mean, also called the standard uncertainty of the three measurements made at each flow set point" to illustrate how $U_{R,i}$ evaluate by pilot lab. 3. To simplify the contents of the report, Draft B deleted Annex B: The detailed procedure of consistency check, and revised Annex C, D, and E to B, C, and D. If participating laboratories need detailed chi-square verification process, they can directly obtain it from pilot lab.

		4. In conclusion, add item 7 to explain the activities that can be performed in the future based on the results of this comparison.
2021/07/12	Draft B ver.2	1. Revise the item 6 and item 7 description in summary.
2021/09/07	Final	

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1 Introduction

A comparison was organized to determine the degree of equivalence of the participating laboratories spanning the gas flow range from 2 mL/min to 10 L/min of nitrogen, under the reference conditions 101.325 kPa and 0 °C.

The comparison will be carried out by using a high precision transfer standard based on four molbloc-L flow elements and a molbox1+. The transfer standards were provided by Fluke USA and Laboratoire National de Métrologie et d'Essais (LNE) France. A comparison was initialized as a CCM Key Comparison for low-pressure gas flow. The results of this comparison can be used for a review of CMC tables.

2 Participants

Ten laboratories participated in the comparison and CMS is the coordinator and pilot of the comparison and supplied the transfer standard package. The K6 comparison started in August 2017 and finished in January 2020. The detailed schedule is listed in Table 1.

Each laboratory had several weeks for providing the measurements and for sending the transfer standards to the next laboratory. The transfer standards were calibrated by the pilot laboratory before and during the comparison to assess its calibration stability. The expanded uncertainties are shown in Table 2. The cells marked in yellow indicate the data points being removed after discussion with NMIJ. The cells marked in red indicate the data points being excluded after the Chi-test.

Table 1. Schedule and facilities used during the KC

No.	Participants	Date	Contact
1	CMS (Chinese Taipei)	Aug, 2017 – Oct, 2017	Chun-Lin Chiang
2	INRIM (Italy)	Oct, 2017 – Nov, 2017	Pier Giorgio Spazzini
3	LNE (France)	Dec, 2017 – Jan, 2018	Jean Barbe/Marc Lefebvre
4	PTB (Germany)	Feb, 2018 – March, 2018	Rainer Kramer
5	CMI (Czech Republic)	April, 2018 – May, 2018	Zdenek Krajicek
6	METAS (Switzerland)	June, 2018 – July, 2018	Bernhard Niederhauser
7-a	KRISS (Korea)	Oct, 2018 – Nov, 2018	Woong Kang
CMS 2nd Check: Aug, 2018 – Sep, 2018			
8-a	NMIJ/AIST (Japan)	Dec, 2018 – Jan, 2019	Toshihiro Morioka
9	NMIA (Australia)	Feb, 2019 – March, 2019	Khaled Chahine

No.	Participants	Date	Contact
10	NIST (USA)	April, 2019 – May, 2019	John Wright
CMS 3rd Check: May, 2019 – July, 2019			
8-b	NMIJ/AIST (Japan)	July, 2019 – Aug, 2019	Toshihiro Morioka
7-b	KRISS (Korea)	Dec, 2019 – Jan, 2020	Woong Kang

Table 2. Expanded uncertainties (%) of the reference flow measurement reported by participants (base uncertainty, $U_{base,i}$, k = 2)

Labs / Nominal Flow (mL/min)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
10000	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
7500	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
5000	0.12	0.06	0.15	0.10	0.10	0.13	0.12	0.03	0.025	0.10
2500	0.12	0.06	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.10
1000	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.11
1000	0.05	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
750	0.05	0.06	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.10
500	0.05	0.06	0.15	0.10	0.10	0.13	0.12	0.03	0.025	0.10
250	0.05	0.07	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.11
100	0.05	0.08	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.11
100	0.05	0.08	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.11
75	0.05	0.09	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.11
50	0.05	0.10	0.15	0.10	0.10	0.13	0.11	0.03	0.025	0.11
25	0.05	0.12	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.11
10	0.05	0.21	0.15	0.10	0.16	0.13	0.12	0.03	0.050	0.11
10	0.05	0.23	0.10	0.10	0.16	[-]	0.14	0.03	0.050	0.11
5	0.05	0.44	0.10	0.12	0.16	[-]	0.19	0.03	0.050	0.11
2	0.05	0.48	0.10	0.25	0.25	[-]	[-]	0.03	0.080	0.11

Note: The symbol "[-]" is mean that this result not been submitted.

3 Transfer Standards

3.1 Specifications

The comparison was carried out by using 4 molbloc-L flow elements, with full scale flows in nitrogen and a molbox1+ for flow calculation. The technical specifications of the transfer standards are listed in Table 3, and the transfer standard package and all the accessories are shown in Figure 1 to Figure 3. All the accessories, including pressure regulators, shut-off valves, mass flow controllers, and filters, were necessary for installing and operating the transfer standard, and were all assembled completely and provided by the pilot laboratory.

Table 3. Technical specifications of transfer standard (Fluke/molbox1+(Serial number:2352))

	molbloc-L_A	molbloc-L_B	molbloc-L_C	molbloc-L_D
Model	1E4-VCR -V-Q	1E3-VCR -V-Q	1E2-VCR -V-Q	1E1-VCR -V-Q
Serial number	7074	7073	7072	2128
Q_{min} (mL/min)	1000	100	10	2
Q_{max} (mL/min)	10000	1000	100	10

Figure 1. Illustration of the transfer standard package

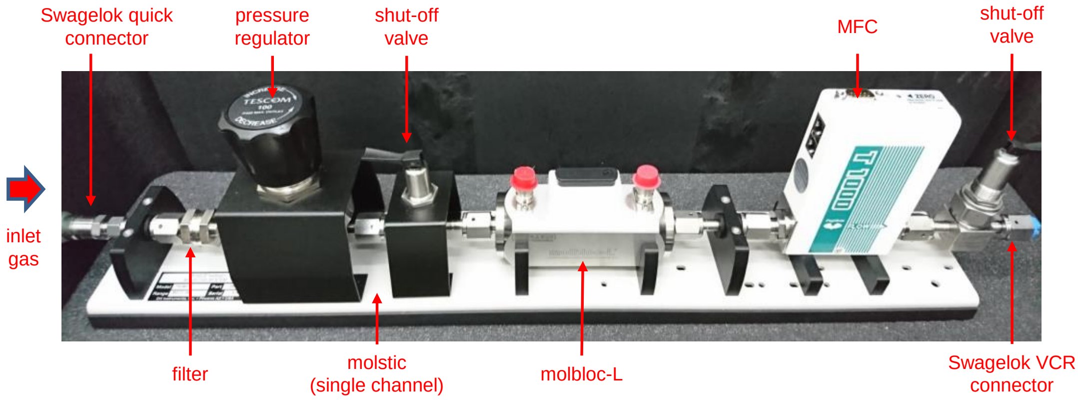


Figure 2.Detailed illustration of the molbloc-L assembly

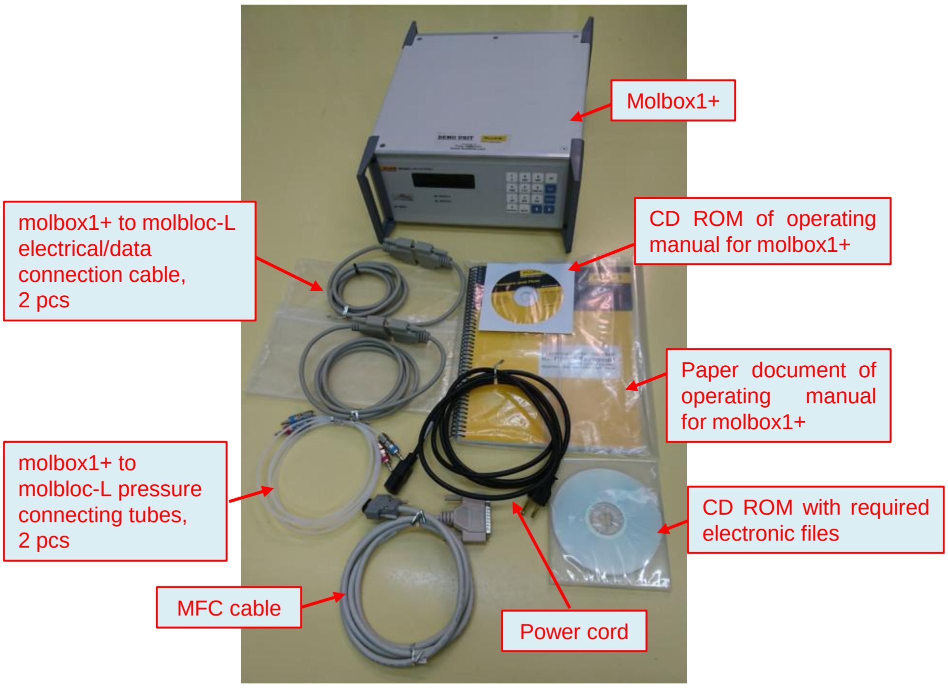


Figure 3. Illustration of molbox1+ and the accessories

3.2 Working Principle

The measurement principle follows laminar flow theory. Under the well-known laws of gas behavior, the flow of a known gas in the laminar flow regime can be calculated from the flow path geometry and the gas pressure and temperature. The schematics of the molbloc-L flow element and the transfer standard package are shown in Figure 4 and Figure 5, respectively.

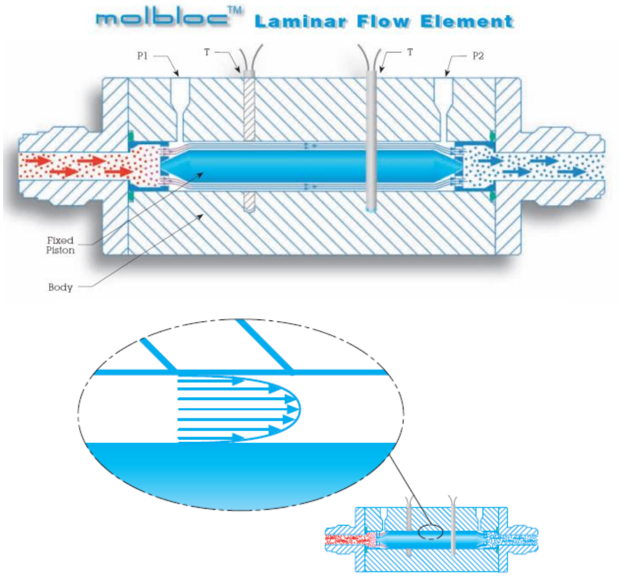


Figure 4. Schematic of a molbloc-L flow element

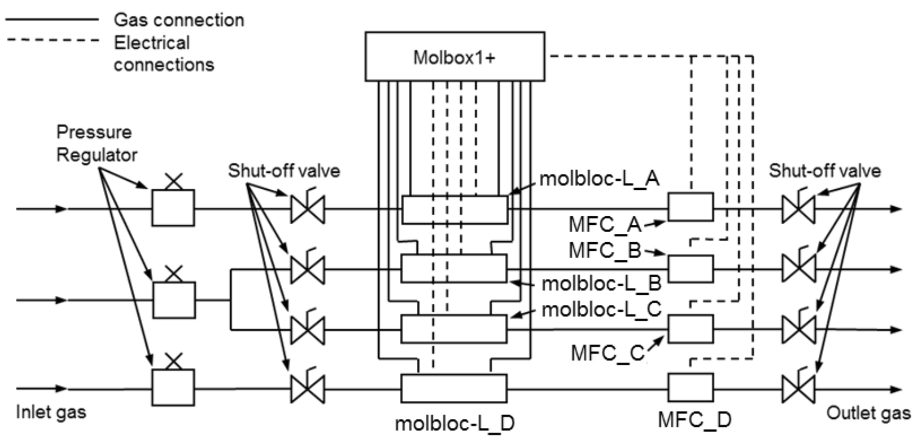


Figure 5. Schematic of the transfer standard package

4 Measurement Procedure

All participating laboratories used their standard calibration procedure to perform measurements and compared them to the measurement values of the transfer standards. The measurement methods of participants are shown in Figure 6.

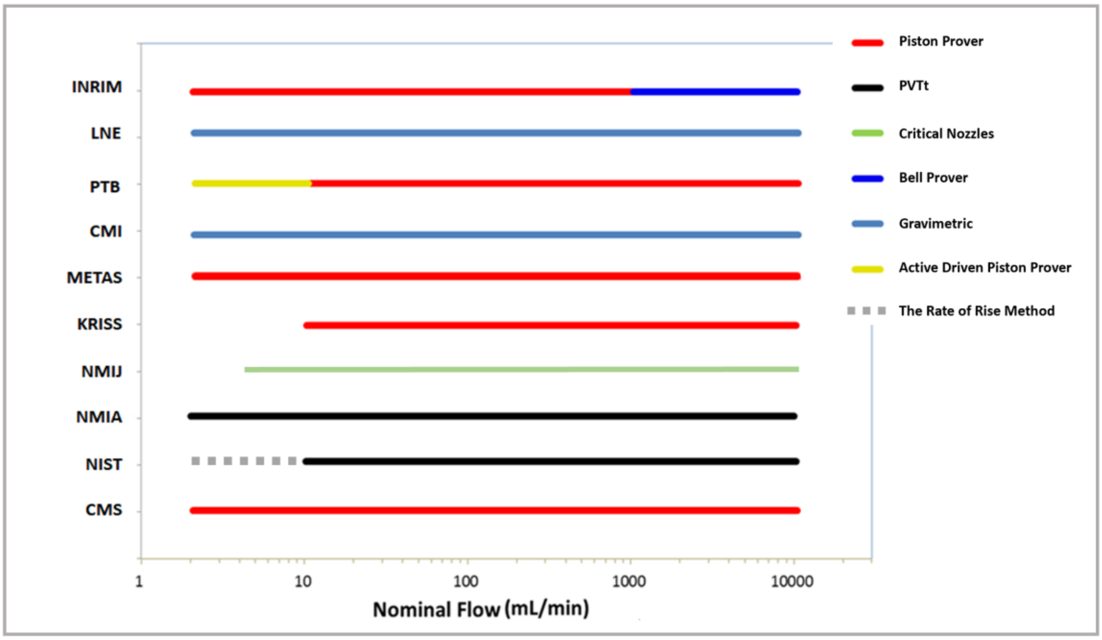


Figure 6. Measurement methods of participants

4.1 Working Gas and Ambient Conditions

4.1.1. Working Gas

- The comparison activity used nitrogen, at least grade 4.5 with purity 99.995 %, as the calibration gas.
- Dynamically produced nitrogen was strictly not recommended since it could contain argon.

4.1.2. Ambient Temperature

- The range of the ambient temperature range was within (20 to 25) °C.
- The entire transfer standard package immersed in the room temperature conditions overnight before collecting data.

4.1.3. Pressure Conditions

- Adjusted upstream pressure within the range of (270 to 275) kPa(a) by transfer standard pressure regulator.
- Never connected a pressure source to the molstic assembly greater than 650 kPa absolute.

4.2 Leakage Test

Each molbloc-L flow element was assembled completely before transportation, and the pilot lab checked every molstic for leaks. The effect of leak flow on the measurement result was more critical for molbloc-L_D. The leak measurements for molbloc-L_D performed by both LNE and the pilot lab were all below 0.001 mL/min.

Participants monitored the change of the absolute pressure in the section between the two shut off valves and recorded the leakage values in the spreadsheet. The details of the procedures of the leak test for the molbloc-L_D flow element as described in section 6 - the measurement procedure in the Technical Protocol of CCM.FF-K6.2017.

4.3 Test Flow Rates

The test flow rates are shown in Table 4. Each calibration point should be conducted with 3 repeated measurements, and the mean was calculated to determine the measurement result for each test flow rate. After changing the flow rate to the next calibration point, at least wait for 20 min before the measurement.

Table 4.Test flow rates for each molbloc-L (unit: mL/min)

molbloc-L_A	molbloc-L_B	molbloc-L_C	molbloc-L_D
10000	1000	100	10
7500	750	75	5
5000	500	50	2
2500	250	25	[-]
1000	100	10	[-]

4.4 Determination of Relative Error of the Transfer Standard

The relative error of the transfer standard (x) was the value used to compare the participants' results. It is defined as the difference between the volumetric flow rate indicated by the transfer standard and the volumetric flow rate measured by the (national) reference flow standard. The standard volumetric flow reference conditions are 101.325 kPa and 0 °C:

$$x = \frac{Q_{\mathrm{TS}} - Q_S}{Q_S} \tag{1}$$

Where

x is the relative error of the transfer standard;

Q_{TS} is the flow rate of the transfer standard at the reference conditions (mL/min);

Q_S is the flow rate of the (national) reference flow standard at the reference conditions (mL/min).

5 Measurement Results

5.1 Relative Errors of the Transfer Standards Obtained at the Pilot Laboratory

The stability of the transfer standards was evaluated by the pilot laboratory - CMS, Chinese Taipei, 3 times in 2017/09, 2018/08 and 2019/08, before and during the comparison. The results of the relative errors of the transfer standards measured by the pilot laboratory, CMS (Chinese Taipei) are shown in Table 5 and Figure 7.

Table 5. The relative errors of the transfer standards measured by the pilot laboratory

Transfer standard	Nominal Flow	Sep-17	Aug-18	Aug-19
		x		
	(mL/min)	(%)		
molbloc-L_A	10000	-0.05	-0.06	-0.09
molbloc-L_A	7500	-0.01	-0.02	-0.06
molbloc-L_A	5000	-0.01	-0.01	-0.04
molbloc-L_A	2500	0.00	-0.02	-0.04
molbloc-L_A	1000	0.01	0.01	0.01
molbloc-L_B	1000	-0.05	-0.06	-0.05
molbloc-L_B	750	-0.04	-0.05	-0.04
molbloc-L_B	500	-0.05	-0.06	-0.06
molbloc-L_B	250	-0.05	-0.06	-0.07
molbloc-L_B	100	-0.04	-0.08	-0.07
molbloc-L_C	100	0.07	0.10	0.09
molbloc-L_C	75	0.08	0.12	0.12
molbloc-L_C	50	0.08	0.11	0.11
molbloc-L_C	25	0.03	0.08	0.03
molbloc-L_C	10	0.02	0.06	0.04
molbloc-L_D	10	0.08	0.09	0.02
molbloc-L_D	5	0.12	0.16	0.10
molbloc-L_D	2	0.21	0.24	0.17

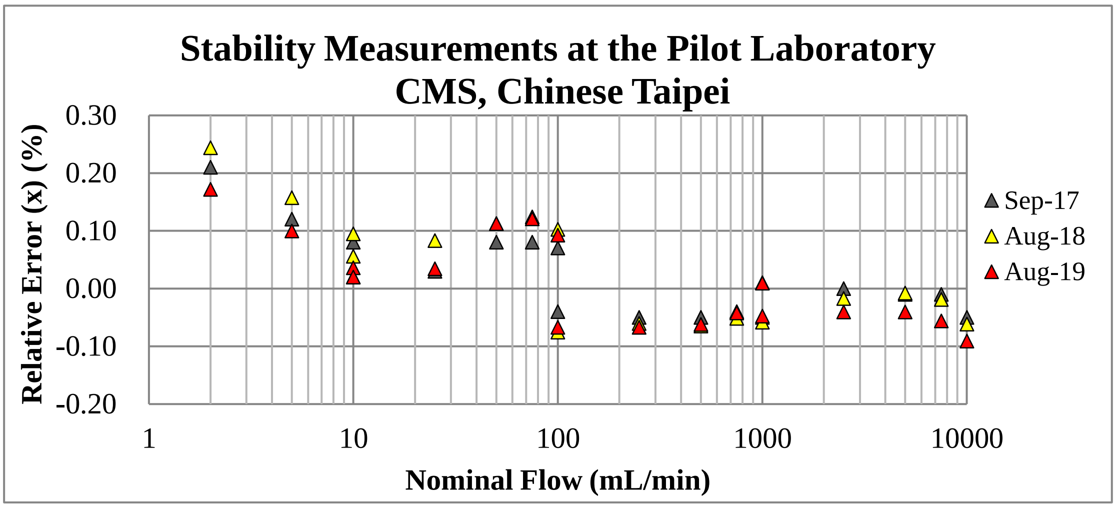


Figure 7. The relative errors of the transfer standards measured by the pilot laboratory

5.2 Determination of the Uncertainties of the Transfer Standards

The uncertainty of the transfer standard is calculated using equation (2) and (3), with symbols $x_{\mathrm{TS,i1}}$, $x_{\mathrm{TS,i2}}$ and $x_{\mathrm{TS,i3}}$ to represent the relative error of the transfer standard for the 3 calibration checks respectively. The uncertainties of the transfer standard are treated as rectangular probability distributions. CMS slightly adjusted and amplified the uncertainties of each transfer standard properly according to the feedback reported from the participants, as shown in Table 6 and Figure 8.

$$u_{\mathrm{TS,i}} = \frac{max(x_{\mathrm{TS,i1}}, x_{\mathrm{TS,i2}}, x_{\mathrm{TS,i3}}) - min(x_{\mathrm{TS,i1}}, x_{\mathrm{TS,i2}}, x_{\mathrm{TS,i3}})}{2\sqrt{3}} \quad (2)$$

$$U_{\mathrm{TS,i}} = k \cdot u_{\mathrm{TS,i}} \quad (3)$$

Where

$x_{\mathrm{TS,i}}$ is the relative error of the transfer standard;

the subscript 1,2,3 represents the order of the sequence of the measurement by the pilot lab;

the subscript i represents the calibration point corresponding to each flow rate;

$u_{\mathrm{TS,i}}$ is the standard uncertainty of each test point;

$U_{\mathrm{TS,i}}$ is the expanded uncertainty (k = 2) of each test point.

Table 6. The expanded uncertainties of the transfer standards evaluated by the pilot laboratory

Transfer Standard	Set Point Nominal Flow (mL/min)	Prior to Adjusting U_{TS} (%)	After Adjusting U_{TS} (%)
molbloc-L_A	10000	0.024	0.03
molbloc-L_A	7500	0.028	0.03
molbloc-L_A	5000	0.021	0.03
molbloc-L_A	2500	0.021	0.03
molbloc-L_A	1000	0.019	0.03
molbloc-L_B	1000	0.007	0.04
molbloc-L_B	750	0.007	0.04
molbloc-L_B	500	0.009	0.04
molbloc-L_B	250	0.010	0.04
molbloc-L_B	100	0.021	0.04
molbloc-L_C	100	0.019	0.06
molbloc-L_C	75	0.028	0.06
molbloc-L_C	50	0.022	0.06
molbloc-L_C	25	0.034	0.06
molbloc-L_C	10	0.021	0.06
molbloc-L_D	10	0.046	0.06
molbloc-L_D	5	0.034	0.06
molbloc-L_D	2	0.042	0.06

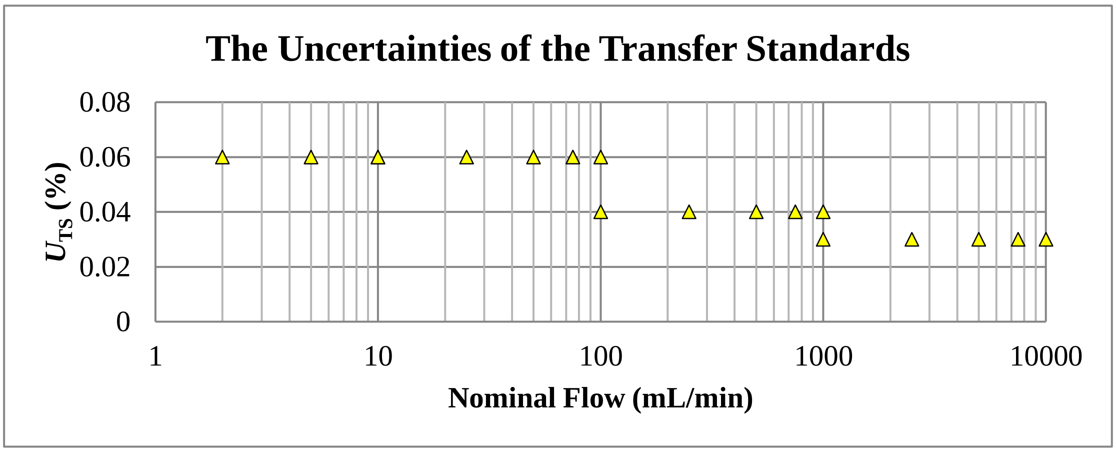


Figure 8. The expanded uncertainties of the transfer standards

5.3 Relative Errors of the Transfer Standards Obtained at the Participating Laboratories

The relative errors of the transfer standards from the participating laboratories are summarized in the Table 7 and Figure 9.

Table 7. Relative errors (%) of the transfer standard obtained by the participating laboratories

Nominal Flow (mL/min)	Relative Error (%)									
	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
10000	-0.211	-0.151	-0.120	-0.128	-0.18	-0.111	-0.251	-0.131	-0.163	-0.062
7500	-0.154	-0.116	-0.070	-0.103	-0.14	-0.093	-0.212	-0.096	-0.121	-0.019
5000	-0.119	-0.096	-0.053	-0.095	-0.13	-0.061	-0.176	-0.076	-0.106	-0.008
2500	-0.162	-0.107	-0.053	-0.107	-0.14	-0.050	-0.128	-0.085	-0.119	-0.017
1000	-0.132	-0.056	-0.013	-0.071	-0.10	-0.039	-0.047	-0.055	-0.070	0.010
1000	-0.163	-0.121	-0.086	-0.120	-0.16	-0.037	-0.106	-0.099	-0.169	-0.059
750	-0.152	-0.124	-0.062	-0.105	-0.15	-0.032	-0.089	-0.081	-0.159	-0.052
500	-0.161	-0.142	-0.088	-0.122	-0.15	-0.045	-0.117	-0.096	-0.171	-0.065
250	-0.160	-0.157	-0.052	-0.128	-0.17	-0.058	-0.140	-0.100	-0.168	-0.060
100	-0.166	-0.170	-0.023	-0.136	-0.19	-0.059	-0.092	-0.107	-0.162	-0.076
100	0.065	0.094	0.069	0.192	0.01	0.040	0.181	0.099	0.038	0.102
75	0.077	0.096	0.089	0.200	0.03	0.084	0.210	0.119	0.060	0.124
50	0.066	0.083	0.112	0.175	0.03	0.086	0.216	0.158	0.065	0.112
25	0.035	0.061	0.108	0.120	-0.02	0.093	0.234	0.148	0.036	0.083
10	0.000	0.100	0.092	0.030	-0.02	0.083	0.256	0.047	0.046	0.056
10	0.090	0.170	-0.025	0.145	-0.10	[-]	0.352	-0.035	0.148	0.095
5	0.201	0.314	0.003	0.209	-0.18	[-]	0.347	0.127	0.255	0.157
2	0.221	0.545	0.037	0.151	-0.33	[-]	[-]	0.072	0.393	0.244

Note: The symbol "[-]" is mean that this result not been submitted.

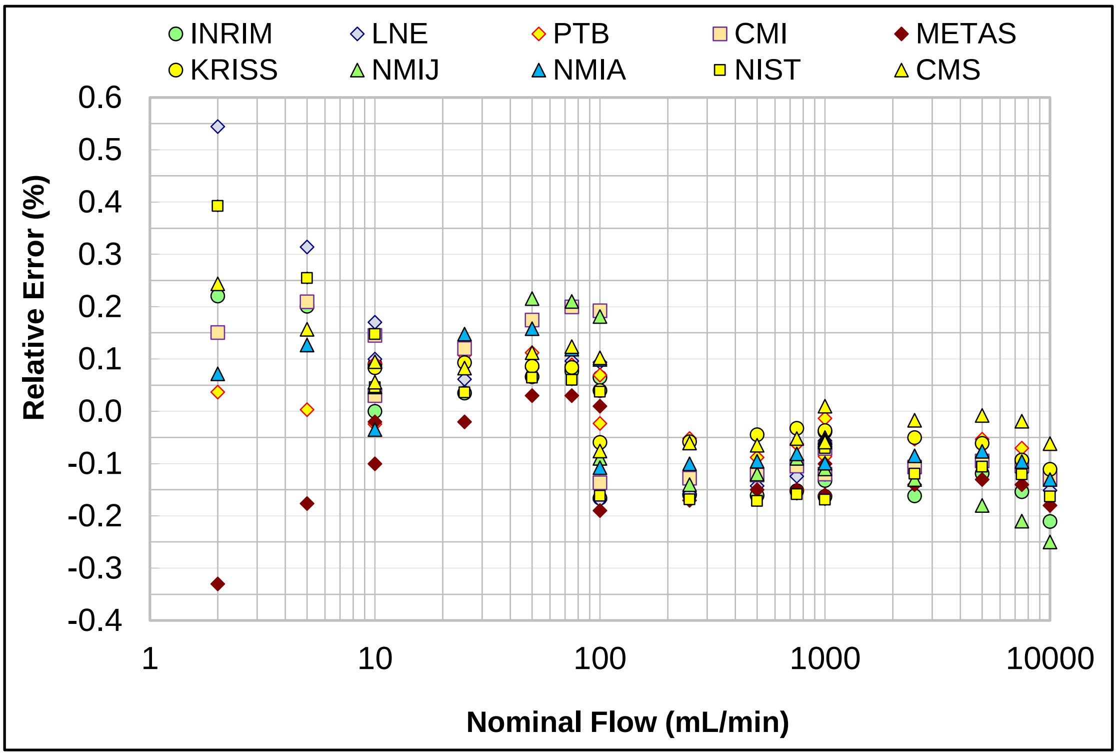


Figure 9. Relative errors (%) of the transfer standard obtained by the participating laboratories

5.4 Determination of the Uncertainties of the Reported Values by Participants

The determination of the uncertainties of the reported values by participants is evaluated by the following equations.

$$U_{x,i} = \sqrt{(U_{base,i})^2 + (U_{R,i})^2 + (U_{TS,i})^2} \tag{4}$$

$$U_{x,i} = k \cdot u_{x,i} \tag{5}$$

Where

$U_{x,i}$ is the expanded uncertainty of the reported value from the participating laboratory; the subscript *x* stands for the participant, and the subscript *i* indicates the flow rate set point number ($k = 2$);

$U_{base,i}$ is the participating lab's reference standard without uncertainty due to the best existing device or transfer standard reproducibility;

$U_{R,i}$ is the reproducibility for the three repeated measurements of the transfer standard in the participant's lab;

The reproducibility was calculated by taking the standard deviation of the mean, also called the standard uncertainty of the three measurements made at each flow set point.

$U_{TS,i}$ is the expanded uncertainties of the transfer standards evaluated by the pilot laboratory.

The reported participating lab expanded uncertainty ($U_{\text{base,i}}$), the reproducibility ($U_{\text{R,i}}$), and expanded uncertainty of the reported value from the participating laboratory ($U_{\text{x,i}}$) are listed in Table 8, Table 9 and Table 10, respectively. The values shown in the tables are the calculated results after rounding, but we used the original data to calculate during the calculations. The red cells in Table 8 indicate that the data points are treated as discrepant results, i.e. the data points are removed from the calculation of the KCRVs and its uncertainties, which is described in section 6.1.3 and 6.1.4.

Table 8. Reported participating lab expanded uncertainty, $U_{\text{base,i}}$, $k = 2$ (%)

Nominal Flow (mL/min)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
10000	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
7500	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
5000	0.12	0.06	0.15	0.10	0.10	0.13	0.12	0.03	0.025	0.10
2500	0.12	0.06	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.10
1000	0.12	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.11
1000	0.05	0.06	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.10
750	0.05	0.06	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.10
500	0.05	0.06	0.15	0.10	0.10	0.13	0.12	0.03	0.025	0.10
250	0.05	0.07	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.11
100	0.05	0.08	0.15	0.10	0.09	0.13	0.12	0.03	0.025	0.11
100	0.05	0.08	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.11
75	0.05	0.09	0.15	0.10	0.09	0.13	0.11	0.03	0.025	0.11
50	0.05	0.10	0.15	0.10	0.10	0.13	0.11	0.03	0.025	0.11
25	0.05	0.12	0.15	0.10	0.08	0.13	0.12	0.03	0.025	0.11
10	0.05	0.21	0.15	0.10	0.16	0.13	0.12	0.03	0.05	0.11
10	0.05	0.23	0.10	0.10	0.16	[-]	0.14	0.03	0.050	0.11
5	0.05	0.44	0.10	0.12	0.16	[-]	0.19	0.03	0.050	0.11
2	0.05	0.48	0.10	0.25	0.25	[-]	[-]	0.03	0.080	0.11

Note: The symbol "[-]" is mean that this result not been submitted.

Table 9. Reproducibility, $U_{R,i}$, k = 2 (%)

Nominal Flow (mL/min)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
10000	0.06	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7500	0.03	0.01	0.01	0.00	0.00	0.00	0.00	0.01	0.00	0.00
5000	0.03	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.00	0.00
2500	0.03	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01
1000	0.03	0.01	0.01	0.02	0.01	0.00	0.00	0.02	0.00	0.00
1000	0.02	0.01	0.01	0.00	0.00	0.01	0.02	0.00	0.00	0.01
750	0.01	0.00	0.01	0.00	0.00	0.00	0.00	0.01	0.00	0.00
500	0.02	0.01	0.01	0.00	0.01	0.00	0.00	0.01	0.00	0.01
250	0.02	0.01	0.00	0.01	0.00	0.01	0.00	0.00	0.00	0.00
100	0.03	0.01	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.01
100	0.01	0.01	0.00	0.00	0.01	0.00	0.00	0.02	0.00	0.01
75	0.02	0.01	0.01	0.00	0.01	0.01	0.00	0.02	0.00	0.00
50	0.02	0.02	0.01	0.00	0.01	0.01	0.00	0.02	0.00	0.01
25	0.01	0.04	0.01	0.00	0.03	0.01	0.00	0.04	0.00	0.01
10	0.02	0.07	0.03	0.01	0.03	0.00	0.01	0.02	0.00	0.02
10	0.04	0.02	0.01	0.05	0.02	[-]	0.00	0.04	0.006	0.01
5	0.04	0.05	0.02	0.01	0.03	[-]	0.00	0.10	0.008	0.02
2	0.05	0.15	0.02	0.03	0.12	[-]	[-]	0.03	0.016	0.02

Note: The symbol “[-]” is mean that this result not been submitted.

Table 10. Uncertainty of the reported value $U_{x,i}$ = RSS [$U_{base,i}$, $U_{TS,i}$, $U_{R,i}$], k = 2 (%).

Nominal Flow (mL/min)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
10000	0.14	0.07	0.15	0.10	0.09	0.13	0.12	0.04	0.04	0.10
7500	0.13	0.07	0.15	0.10	0.09	0.13	0.12	0.04	0.04	0.10
5000	0.13	0.07	0.15	0.10	0.10	0.13	0.12	0.04	0.04	0.10
2500	0.13	0.07	0.15	0.10	0.09	0.13	0.12	0.04	0.04	0.10
1000	0.13	0.07	0.15	0.11	0.10	0.13	0.12	0.05	0.04	0.11
1000	0.07	0.08	0.16	0.11	0.10	0.14	0.13	0.05	0.05	0.11
750	0.06	0.08	0.16	0.11	0.10	0.14	0.12	0.05	0.05	0.11
500	0.07	0.08	0.16	0.11	0.11	0.14	0.13	0.05	0.05	0.11
250	0.07	0.08	0.16	0.11	0.09	0.14	0.13	0.05	0.05	0.12
100	0.07	0.09	0.16	0.11	0.10	0.14	0.13	0.05	0.05	0.12
100	0.08	0.10	0.16	0.12	0.11	0.14	0.12	0.07	0.07	0.13
75	0.08	0.11	0.16	0.12	0.11	0.14	0.12	0.07	0.07	0.13
50	0.08	0.12	0.16	0.12	0.12	0.14	0.13	0.07	0.07	0.13
25	0.08	0.14	0.16	0.12	0.10	0.14	0.13	0.08	0.07	0.13
10	0.08	0.23	0.16	0.12	0.17	0.14	0.13	0.07	0.08	0.13
10	0.09	0.24	0.12	0.13	0.17	[-]	0.13	0.08	0.08	0.13
5	0.09	0.45	0.12	0.13	0.17	[-]	0.15	0.12	0.08	0.13
2	0.09	0.51	0.12	0.26	0.28	[-]	[-]	0.07	0.10	0.13

Note: The symbol "[-]" is mean that this result not been submitted.

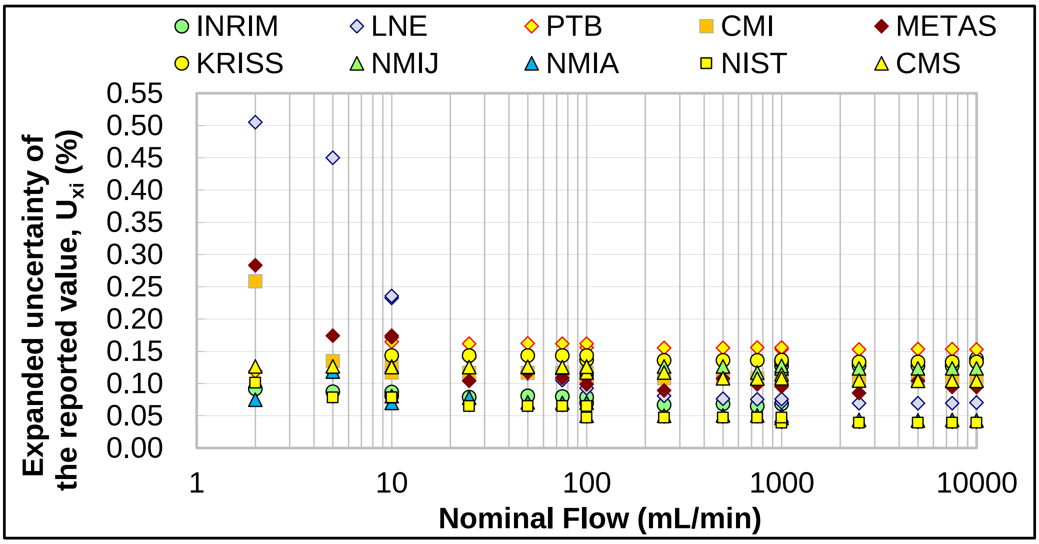


Figure 10. Uncertainty of the reported value $U_{x,i}$ = RSS [$U_{base,i}$, $U_{TS,i}$, $U_{R,i}$], k = 2 (%)

6 Evaluation

6.1 Determination of the Key Comparison Reference Values (x_{ref}) and Its Uncertainties

The reference value was determined at each flow rate individually. The method of determination of the reference value at each flow rate was based on the guidelines by Cox[1] and current recommendations of CCM Working Group on Fluid Flow (CCM-WGFF).

6.1.1 KCRV

The KCRV was calculated as a weighted mean error (WME):

$$\text{KCRV} = x_{\text{ref}} = \frac{\frac{x_1}{u^2(x_1)}+\frac{x_2}{u^2(x_2)}+\dots\dots\dots+\frac{x_{\text{n}}}{u^2(x_{\text{n}})}}{\frac{1}{u^2(x_1)}+\frac{1}{u^2(x_2)}+\dots\dots\dots+\frac{1}{u^2(x_{\text{n}})}} \tag{6}$$

Where,

- x_1, x_2, ... x_{n} are relative errors of the transfer standard at one flow rate in different independent laboratories 1, 2, …... n;
- $u(x_1)$, $u(x_2)$, ... $u(x_{\text{n}})$ are standard uncertainty of the relative error in different independent laboratories 1, 2, …..., n , which is described in section 5.4, including;
  - The uncertainty of participating lab's reference standard ($u_{\text{base,i}}$).
  - The uncertainty caused by the reproducibility for the n repeated measurements of the transfer standard in the participant's lab ($u_{\text{R,i}}$).
  - The expanded uncertainties of the transfer standards evaluated by the pilot laboratory ($u_{\text{TS,i}}$).

6.1.2 The Uncertainty of KCRV

The standard uncertainty of the reference value $u(x_{\text{ref}})$ is given by

$$\frac{1}{u^2(x_{\text{ref}})} = \frac{1}{u^2(x_1)} + \frac{1}{u^2(x_2)} + \dots\dots\dots + \frac{1}{u^2(x_{\text{n}})} \tag{7}$$

The expanded uncertainty of the reference value $U(x_{\text{ref}})$ is

$$U(x_{\text{ref}}) = k \cdot u(x_{\text{ref}}) \tag{8}$$

6.1.3 The Consistency Check

The chi-squared test for the consistency check was performed using values of relative errors of the transfer standard at each flow rate.

At first the chi-squared value χ^2_{obs} was calculated by

$$\chi^2_{\text{obs}} = \frac{(x_1 - x_{\text{ref}})^2}{u^2(x_1)} + \frac{(x_2 - x_{\text{ref}})^2}{u^2(x_2)} + \ldots\ldots\ldots\ldots + \frac{(x_n - x_{\text{ref}})^2}{u^2(x_{\text{n}})} \tag{9}$$

The degrees of freedom ν was assigned

$$\nu = \text{n} - 1 \tag{10}$$

where n is the number of evaluated laboratories.

The consistency check failed if

$$P_r\,\{\chi^2_\nu > \chi^2_{\text{obs}}\} < 0.05 \tag{11}$$

The function CHIINV(0.05; ν) in Excel was used.
The consistency check failed if CHIINV(0.05; ν) $<\chi^2_{\text{obs}}$

If the consistency check passed, then x_{ref} and $U(x_{\text{ref}})$ were accepted as the key comparison reference value x_{ref} and the expanded uncertainty of the key comparison reference value $U(x_{\text{ref}})$, respectively.

If the consistency check failed, then the laboratory with the highest value of $(x_{\text{i}} - x_{\text{ref}})^2/u^2(x_{\text{i}})$ was excluded for the next round of evaluation and the new reference value y (WME), the new standard uncertainty of the reference value $u(x_{\text{ref}})$ and the chi-squared value χ^2_{obs} was calculated again without the values of the excluded laboratory. The consistency check was calculated again, too. This procedure was repeated until the consistency check passed.

6.1.4 The results of the consistency check

To pass the consistency check, we excluded some laboratory data after four rounds of the consistency check. After the data points are excluded, we get the new reference value, the new standard uncertainty of the reference value $u(x_{\mathrm{ref}})$ and the chi-squared value χ^2_{obs} without the values of excluded laboratory. The new KCRVs and their uncertainties are listed in Table 11.

Table 11. The calculated values of the new KCRVs and their uncertainties after four rounds of consistency check

Nominal Flow	x_{ref}	$u(x_{\mathrm{ref}})$	$U(x_{\mathrm{ref}})$	Consistency Check	
(mL/min)	(%)	(k = 1, %)	(k = 2, %)	CHIINV(0.05,n-1)	Okay! / OOOPs
10000	-0.149	0.011	0.023	16.919	Okay!
7500	-0.111	0.011	0.023	16.919	Okay!
5000	-0.093	0.011	0.023	16.919	Okay!
2500	-0.103	0.011	0.023	16.919	Okay!
1000	-0.062	0.012	0.023	16.919	Okay!
1000	-0.129	0.012	0.025	16.919	Okay!
750	-0.118	0.012	0.024	16.919	Okay!
500	-0.131	0.012	0.025	16.919	Okay!
250	-0.136	0.012	0.025	16.919	Okay!
100	-0.136	0.013	0.025	16.919	Okay!
100	0.081	0.015	0.031	16.919	Okay!
75	0.099	0.015	0.031	16.919	Okay!
50	0.105	0.016	0.031	16.919	Okay!
25	0.067	0.016	0.033	15.507	Okay!
10	0.040	0.018	0.036	15.507	Okay!
10	0.090	0.021	0.043	12.592	Okay!
5	0.205	0.023	0.045	11.070	Okay!
2	0.074	0.030	0.061	7.815	Okay!

6.2 Determination of the Differences to Key Comparison Reference Values

6.2.1 d_i (Differences between the Participating Labs to KCRV)

When the KCRV was determined, the differences between the participating laboratories and the KCRV corresponding to different nominal flow rates can be calculated according to the following equation.

$$d_\mathrm{i} = x_\mathrm{i} - x_\mathrm{ref} \tag{12}$$

- For the independent laboratories contributing to the KCRV:
  The covariance between the result of a laboratory (with the contribution to the KCRV) and the KCRV is the variance of the KCRV itself. The uncertainty of d_i can be calculated using the following equation.

$$u(d_\mathrm{i}) = \sqrt{u^2(x_\mathrm{i}) + u^2(x_\mathrm{ref}) - 2 \times u^2(x_\mathrm{ref})} = \sqrt{u^2(x_\mathrm{i}) - u^2(x_\mathrm{ref})} \tag{13}$$

- For the independent laboratories without contribution to the KCRV:
  Since there is no covariance between the result of a laboratory without the contribution and the KCRV, the uncertainty of d_i can be calculated using the following equation.

$$u(d_\mathrm{i}) = \sqrt{u^2(x_\mathrm{i}) + u^2(x_\mathrm{ref})} \tag{14}$$

The expanded uncertainties $U(d_\mathrm{i})$ are determined by the following equation.

$$U(d_\mathrm{i}) = 2 \cdot u(d_\mathrm{i}) \tag{15}$$

The d_i values using different transfer standards are shown in Figure 11 to Figure 14.

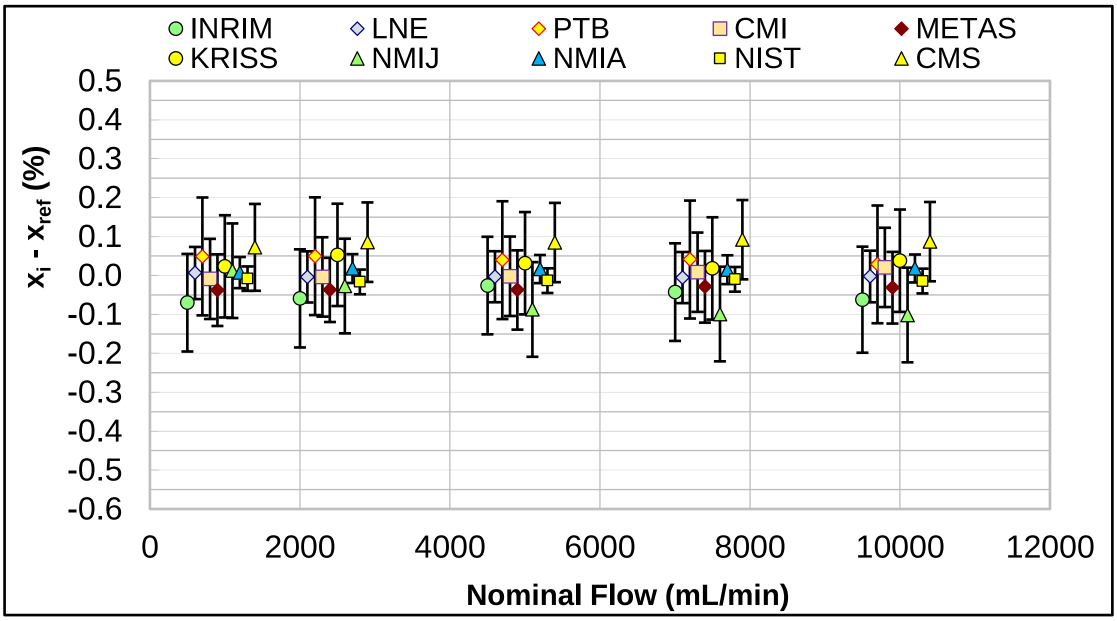


Figure 11. The d_i values using transfer standard molbloc-L_A

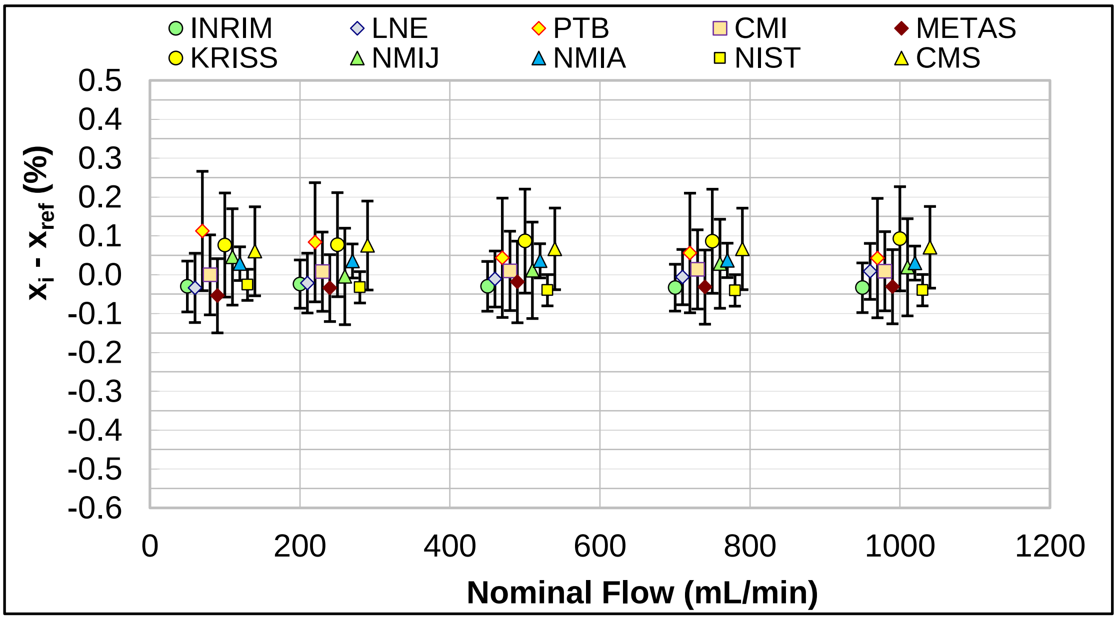


Figure 12. The d_i values using transfer standard molbloc-L_B

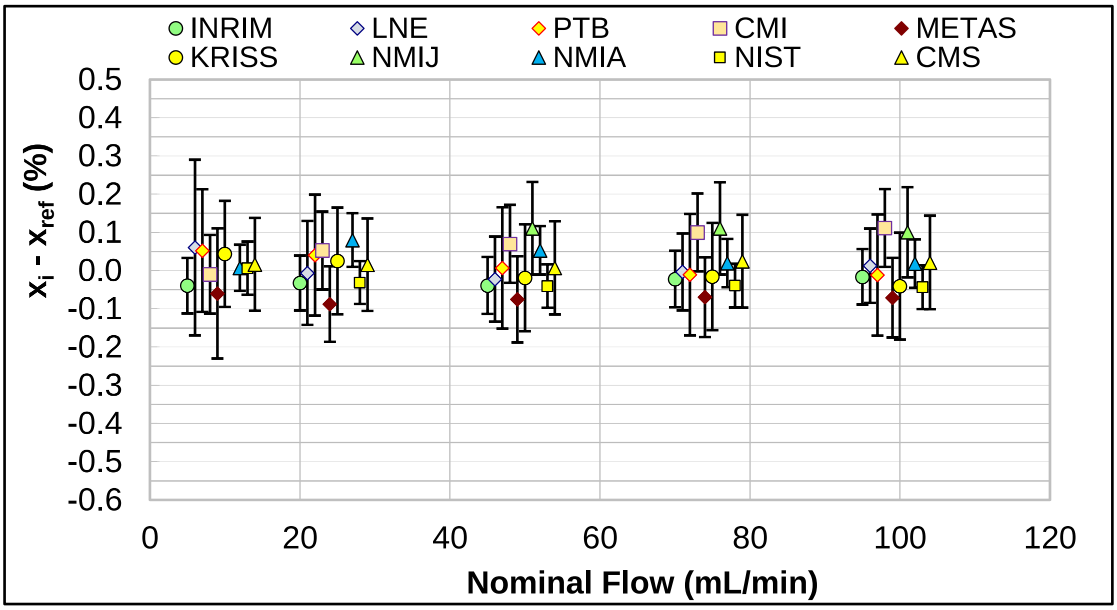


Figure 13. The d_i values using transfer standard molbloc-L_C

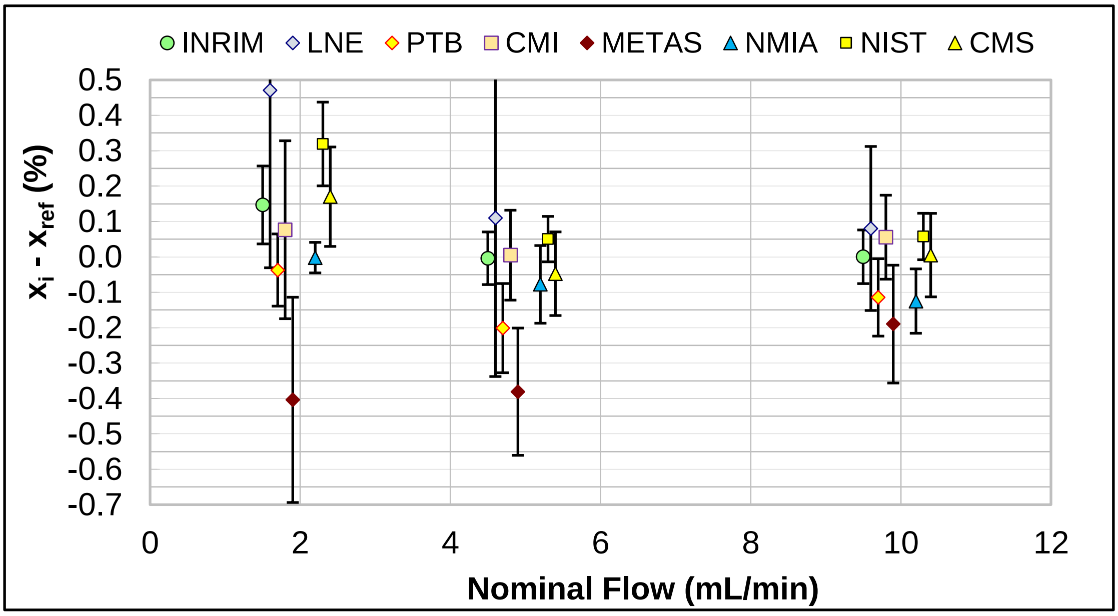


Figure 14.The d_i values using transfer standard molbloc-L_D

6.2.2 E_n (Degree of Equivalence, DoE)

Based on these differences, the Degree of Equivalence (DoE) is calculated according to the following equation

$$E_\mathrm{n} = \frac{d_\mathrm{i}}{U(d_\mathrm{i})} \tag{15}$$

The DoE is a measure for the equivalence of the results of any laboratory with the KCRV.

- The results of a laboratory are equivalent if $|E_\mathrm{n}| \leq 1$.
- The laboratory was determined as not equivalent if $|E_\mathrm{n}|$ >1.2, and was marked in red in Table 12.
- For values of DoE in the range $1 < |E_\mathrm{n}| \leq 1.2$ might indicate a possible warning in the measurement process, and was marked in blue in Table 12.

The E_n values using different transfer standards are shown in Figure 15 to Figure 18.

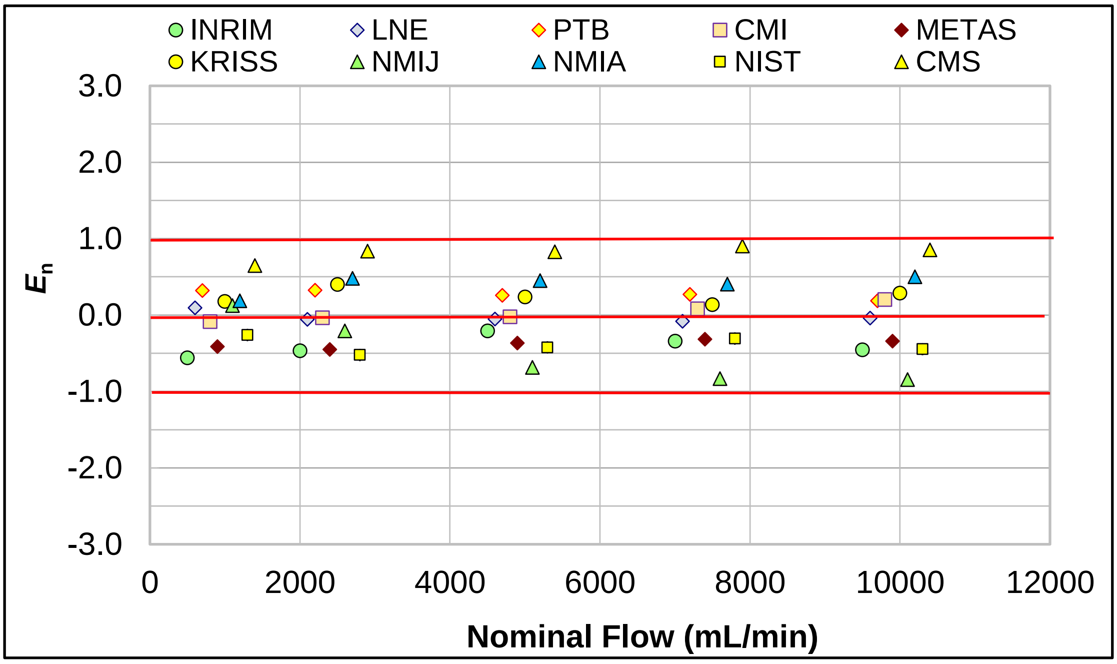


Figure 15. The E_n values using transfer standard molbloc-L_A

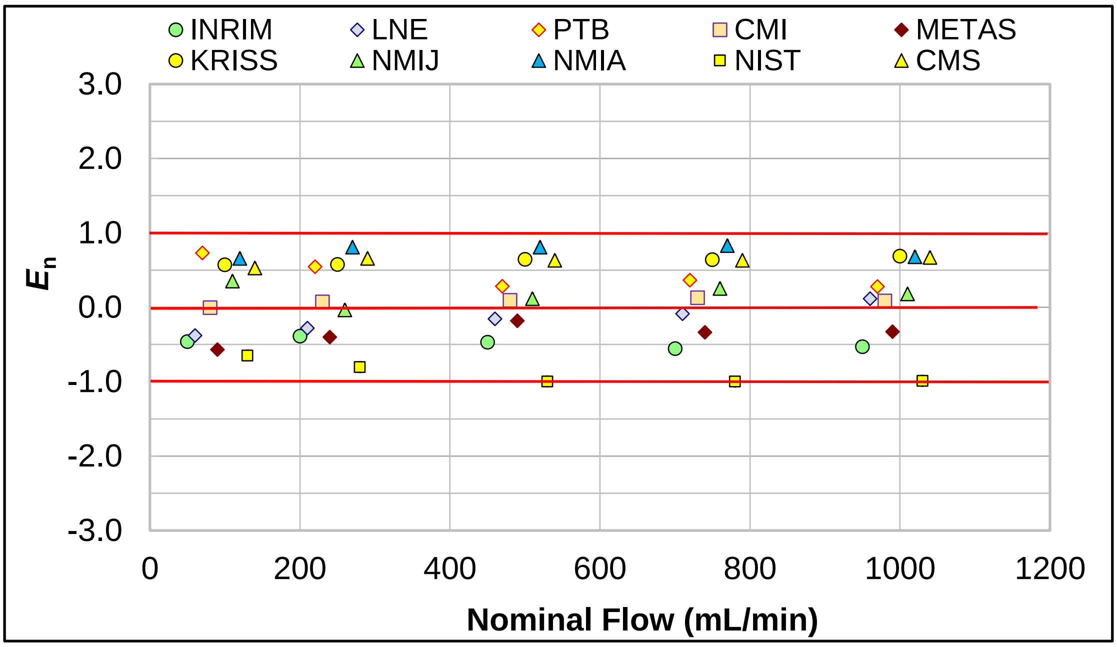


Figure 16. The E_n values using transfer standard molbloc-L_B

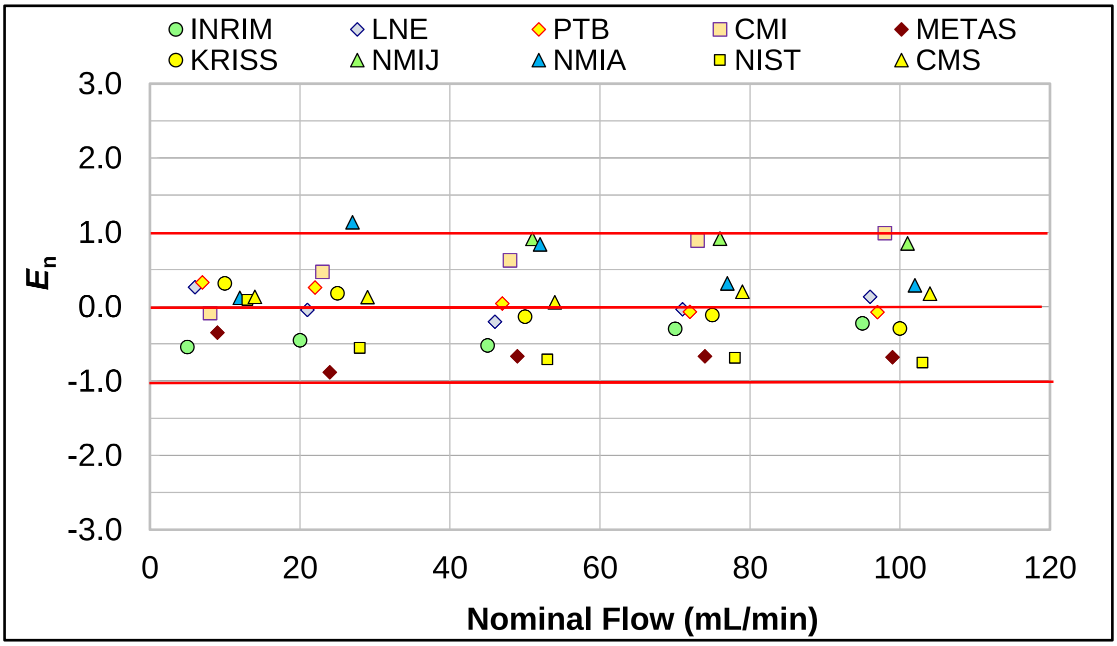


Figure 17. The E_n values using transfer standard molbloc-L_C

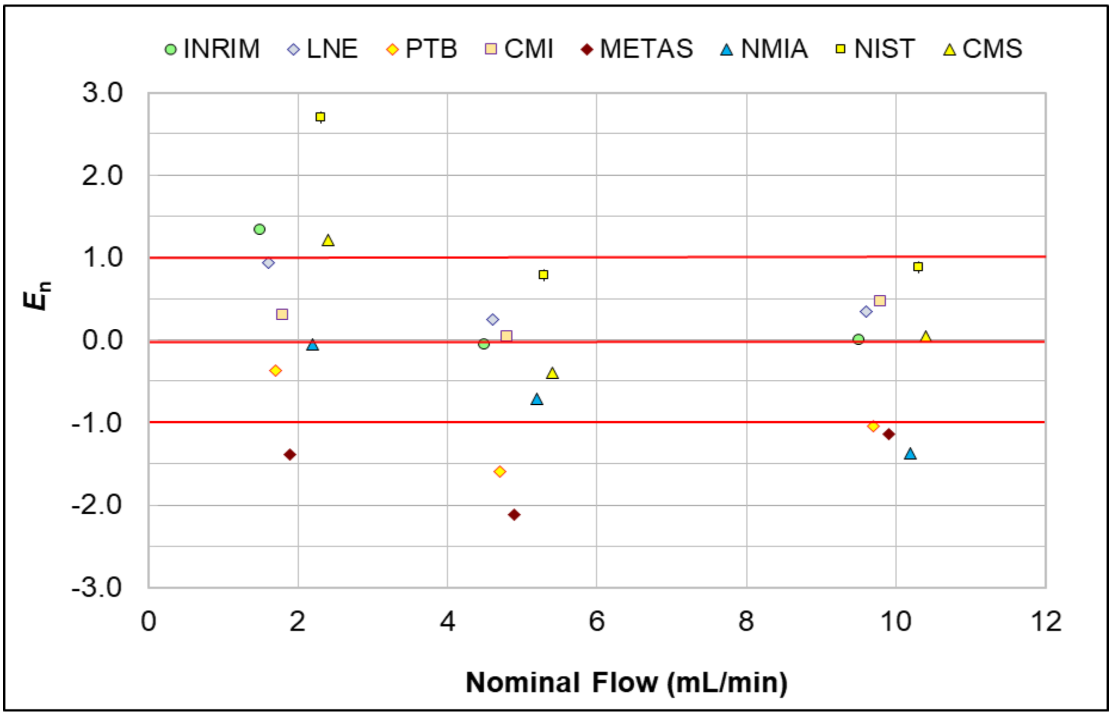


Figure 18. The E_n values using transfer standard molbloc-L_D

Table 12. List of d_i and E_n of each participating lab

Nominal flow	INRIM			LNE			PTB			CMI			METAS		
	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n
[mL/min]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]
10000	-0.06	0.14	-0.46	0.00	0.07	-0.04	0.03	0.15	0.19	0.02	0.10	0.20	-0.03	0.09	-0.34
7500	-0.04	0.13	-0.34	-0.01	0.07	-0.08	0.04	0.15	0.27	0.01	0.10	0.08	-0.03	0.09	-0.32
5000	-0.03	0.13	-0.21	0.00	0.07	-0.05	0.04	0.15	0.26	0.00	0.10	-0.02	-0.04	0.10	-0.37
2500	-0.06	0.13	-0.47	0.00	0.07	-0.05	0.05	0.15	0.33	0.00	0.10	-0.04	-0.04	0.08	-0.45
1000	-0.07	0.13	-0.56	0.01	0.07	0.09	0.05	0.15	0.32	-0.01	0.10	-0.09	-0.04	0.09	-0.41
1000	-0.03	0.06	-0.53	0.01	0.07	0.12	0.04	0.15	0.28	0.01	0.10	0.09	-0.03	0.10	-0.32
750	-0.03	0.06	-0.55	-0.01	0.07	-0.09	0.06	0.15	0.36	0.01	0.10	0.13	-0.03	0.10	-0.33
500	-0.03	0.06	-0.47	-0.01	0.07	-0.15	0.04	0.15	0.28	0.01	0.10	0.09	-0.02	0.10	-0.18
250	-0.02	0.06	-0.39	-0.02	0.08	-0.28	0.08	0.15	0.54	0.01	0.11	0.07	-0.03	0.09	-0.40
100	-0.03	0.07	-0.46	-0.03	0.09	-0.38	0.11	0.15	0.73	0.00	0.10	0.00	-0.05	0.10	-0.57
100	-0.02	0.07	-0.22	0.01	0.10	0.13	-0.01	0.16	-0.07	0.11	0.11	0.99	-0.07	0.10	-0.68
75	-0.02	0.07	-0.30	0.00	0.10	-0.03	-0.01	0.16	-0.07	0.10	0.11	0.89	-0.07	0.10	-0.66
50	-0.04	0.07	-0.52	-0.02	0.11	-0.20	0.01	0.16	0.04	0.07	0.11	0.62	-0.08	0.11	-0.67
25	-0.03	0.07	-0.45	-0.01	0.14	-0.04	0.04	0.16	0.26	0.05	0.11	0.47	-0.09	0.10	-0.88
10	-0.04	0.07	-0.54	0.06	0.23	0.26	0.05	0.16	0.33	-0.01	0.11	-0.09	-0.06	0.17	-0.35
10	0.00	0.08	0.00	0.08	0.23	0.35	-0.11	0.11	-1.05	0.06	0.12	0.47	-0.19	0.17	-1.14
5	0.00	0.07	-0.05	0.11	0.45	0.25	-0.20	0.13	-1.60	0.00	0.13	0.04	-0.38	0.18	-2.12
2	0.15	0.11	1.33	0.47	0.50	0.94	-0.04	0.10	-0.36	0.08	0.25	0.31	-0.40	0.29	-1.39

Nominal flow	KRISS			NMIJ			NMIA			NIST			CMS		
	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n	d_i	$U(d_i)$	E_n
[mL/min]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]	[%]	[%]	[-]
10000	0.04	0.13	0.29	-0.10	0.12	-0.83	0.02	0.04	0.50	-0.01	0.03	-0.45	0.09	0.10	0.85
7500	0.02	0.13	0.14	-0.10	0.12	-0.81	0.01	0.04	0.40	-0.01	0.03	-0.31	0.09	0.10	0.90
5000	0.03	0.13	0.24	-0.09	0.12	-0.72	0.02	0.04	0.46	-0.01	0.03	-0.42	0.08	0.10	0.83
2500	0.05	0.13	0.40	-0.03	0.12	-0.22	0.02	0.04	0.48	-0.02	0.03	-0.52	0.09	0.10	0.84
1000	0.02	0.13	0.18	0.01	0.12	0.10	0.01	0.04	0.19	-0.01	0.03	-0.26	0.07	0.11	0.65
1000	0.09	0.13	0.69	0.02	0.13	0.15	0.03	0.04	0.68	-0.04	0.04	-0.98	0.07	0.11	0.67
750	0.09	0.13	0.64	0.03	0.11	0.25	0.04	0.04	0.83	-0.04	0.04	-1.00	0.07	0.10	0.63
500	0.09	0.13	0.65	0.01	0.12	0.09	0.04	0.04	0.81	-0.04	0.04	-0.99	0.07	0.11	0.63
250	0.08	0.13	0.58	0.00	0.12	-0.04	0.04	0.04	0.81	-0.03	0.04	-0.80	0.08	0.11	0.66
100	0.08	0.13	0.57	0.05	0.12	0.37	0.03	0.04	0.66	-0.03	0.04	-0.65	0.06	0.11	0.53
100	-0.04	0.14	-0.29	0.10	0.12	0.85	0.02	0.06	0.29	-0.04	0.06	-0.75	0.02	0.12	0.18
75	-0.02	0.14	-0.11	0.11	0.12	0.92	0.02	0.06	0.31	-0.04	0.06	-0.69	0.02	0.12	0.20
50	-0.02	0.14	-0.13	0.11	0.12	0.91	0.05	0.06	0.84	-0.04	0.06	-0.71	0.01	0.12	0.06
25	0.03	0.14	0.18	[-]	[-]	[-]	0.08	0.07	1.14	-0.03	0.06	-0.55	0.02	0.12	0.13
10	0.04	0.14	0.31	[-]	[-]	[-]	0.01	0.06	0.12	0.01	0.07	0.09	0.02	0.12	0.13
10	[-]	[-]	[-]	[-]	[-]	[-]	-0.12	0.09	-1.37	0.06	0.07	0.88	0.01	0.12	0.04
5	[-]	[-]	[-]	[-]	[-]	[-]	-0.08	0.11	-0.71	0.05	0.06	0.78	-0.05	0.12	-0.40
2	[-]	[-]	[-]	[-]	[-]	[-]	0.00	0.04	-0.05	0.32	0.12	2.70	0.17	0.14	1.21

Note: The symbol "[-]" is mean that this result not been submitted.

7 Summary

1. CCM.FF-K6.2017 was piloted by CMS from 2017 to 2020. Eight NMIs tested four transfer standards ranging from 2 mL/min to 10 L/min, whereas NMIJ tested the four transfer standards ranging from 5 mL/min to 10 L/min, and KRISS tested the three transfer standards ranging from 10 mL/min to 10 L/min. The calculated values of the KCRVs and their uncertainties are listed in Table 11.
2. The reference value was determined at each flow rate individually. The method of determination of the reference value at each flow rate was based on the guidelines by Cox[1] and the current recommendations of CCM Working Group on Fluid Flow (CCM-WGFF)[2].
3. Removed NMIJ's test result at nominal flow rates 5 mL/min to 25 mL/min since NMIJ had concerns about their initial test data. Their re-test data are listed in Annex B for reference.
4. The chi-squared test for consistency check was performed using values of relative errors of the transfer standard at each flow rate. To fulfill the consistency check, after four rounds of the consistency checks, we excluded some laboratories' data points with molbloc-L_D as the transfer standard to pass in the consistency check. The excluded lab data include NMIA at nominal flow rate 10 mL/min, PTB and METAS at nominal flow rate 5 mL/min, and METAS, INRIM, NIST and CMS at nominal flow rate 2 mL/min.
5. NMIA's reproducibility (U_R) results at nominal flow rates 25 mL/min with molbloc-L_C, 10 mL/min with molbloc-L_D, and 5 mL/min with molbloc-L_D are 0.04 % , 0.04 % and 0.10 %, respectively, which are all larger than the reported lab uncertainty (U_{base}) 0.03 % in this comparison. Therefore, we suggest NMIA to clarify and re-evaluate the lab uncertainty in the flow rate ranging from 2 mL/min to 25 mL/min.
6. The KCRV of 10 mL/min to 2 mL/min may not be representative. For example, the reasons are four NMIs (total eight NMIs submitted 2 mL/min measurement results) could not pass the consistency check at 2 mL/min, and NMIA 's reproducibility (U_R) result at 2 mL/min is 0.033 %, which is larger than the reported lab uncertainty (U_{base}) 0.03 %.
7. In this comparison, we found some issues may need more experiments to understand in the future. For example, the results of chi-squared test for consistency check of flow rates of 5 mL/min and 2 mL/min cannot provide enough correlation to identify that the results of all participants are from the same population (only a few participating laboratories passed the critical value). Therefore, we may need another comparison for very low flow rates at lower than 10 mL/min to investigate the detail measurement issues such as TS performance at very low flows, and the influence evaluation of

measurement uncertainties of individual laboratories in very low flow rates as 2 mL/min on the comparison results. Based on the conclusion of WGFF meeting discussion at June 24, 2021. The participates will form a group to further study of the TS performance at the low flow rate and will have new comparison in the future.

8. Test facilities of each NMI that used in this comparison are shown in section Annex A- Test facilities.

8 References

[1] Cox M.G., Evaluation of key comparison data. Metrologia, 2002, 39, 589-595.
[2] WGFF Guidelines for CMC Uncertainty and Calibration Report Uncertainty, Working Group for Fluid Flow, October 21, 2013.
[3] Transfer standard uncertainty can cause inconclusive inter-laboratory comparisons, Metrologia, 2016, 53(6), 1243–1258.
[4] J. Randa, “Proposal for KCRV and Degree of Equivalence for GTRF Key Comparisons,” GT-RF/2000-12, August 2000.
[5] J. Randa, “Update to Proposal for KCRV & Degree of Equivalence for GTRF Key Comparisons,” GT-RF/2005-04, February 2005.

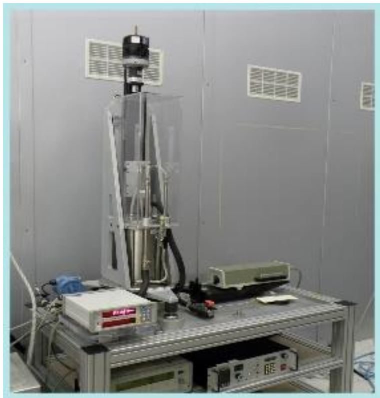
9 Terms and abbreviations

BIPM = Bureau International des Poids et Mesures (the International Bureau of Weights and Measures)
CCM = Consultative Committee for Mass and Related Quantities
CIPM = Comité International des Poids et Mesures (International Committee for Weights and Measures)
CMC = Calibration and Measurement Capabilities
DoE = Degree of Equivalence
FF = Fluid Flow
GUM = Guide to the Expression of Uncertainty in Measurement
KC = Key Comparison
KCRV = Key Comparison Reference Value
NMI = National Metrology Institute
TS = Transfer Standard
VIM = Vocabulaire International de Metrologie
WGFF = Working Group for Fluid Flow

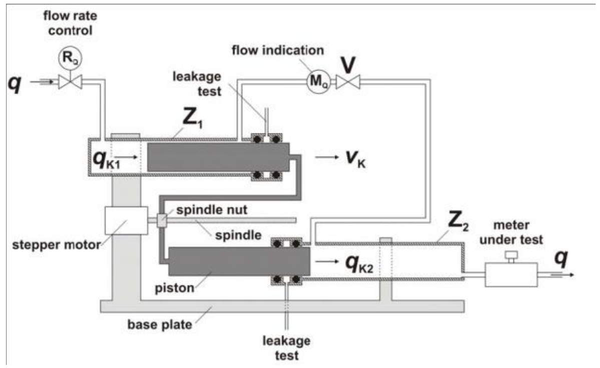
10 Annex

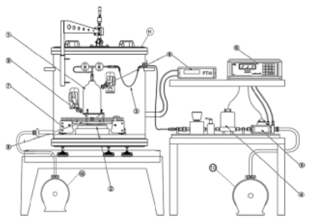
10.1 Annex A: Test facilities of each participant

CMS - Center for Measurement Standards	
	Facility
Range of flow rate	(2 to 24000) mL/min
Working temperature	(22 to 24) °C
Working pressure	Ambient pressure
Expanded uncertainty U_{base} (k = 2)	(0.10 to 0.11) %
Picture of the facility	
System description	Piston prover is a standard device for gas meter calibration, ranging from 2 mL/min to 24000 mL/min at standard conditions 296.15 K and 101.325 kPa. The facility contains five precisely manufactured glass tubes, integrated with start-stop photocells and an electronic digital timer to achieve automatic calibration. The gas flow is collected in a precision-bore glass cylinder under mercury sealed piston.

INRIM		
	Facility 1 (MicroGAS)	**Facility 2 (BellGAS)**
Range of flow rate	(0.1 to 1600) SCCM (CMC from (2 to 1600) SCCM)	(1 to 120) L/min
Working temperature	Ambient, Selectable from (15 to 25) °C	Ambient, Selectable from (15 to 25) °C
Working pressure	Ambient	Ambient
Expanded uncertainty U_{base} (k = 2)	0.05 %	0.12 %
Picture of the facility		
System description	Piston prover with fine thermal regulation (better than 0.05 °C during calibration), active piston regulated by the pressure within the cylinder; flow rate computed through balance of mass method; DUT installed upstream of the test rig.	Bell prover; passive bell with pressure compensation; flow rate computed through balance of mass method; DUT installed upstream of the test rig.

LNE	
	Facility
Range of flow rate	0.021 mg/s to 210 mg/s ((1 mL/min to 10 L/min) N_2)
Working temperature	21 °C ± 2 °C
Working pressure	100 kPa to 500 kPa
Expanded uncertainty U_{base} (k = 2)	0.54 % to 0.060 %
Picture of the facility	
System description	The dynamic gravimetric flow standard periodically measures the mass of a gas-filled, high pressure cylinder while gas is withdrawn from the cylinder over a period of time (typically minutes to hours). The system allows to make accurate real-time mass flow measurements with continuous compensation for buoyancy corrections to the apparent weight of the cylinder.

PTB		
	Facility 1	**Facility 2**
Range of flow rate	Q_{act}= 10 mL/min to 10 L/min	Q_{act}= 0.1 mL/min to 0.13 L/min
Working temperature	Ambient (18 °C to 22 °C)	Ambient (18 °C to 22 °C)
Working pressure	Ambient (p_e< 100 mbar)	p = 0.1 MPa to 0.6 MPa
Expanded uncertainty U_{base} (k = 2)	0.15 %	0.08 %
Picture of the facility		
System description	Three mercury sealed piston / cylinder systems are calibrated with a coordination measurement machine in diameter, the displacement of the pistons caused by the flow rate of the MUT is determined by a heterodyne interferometer. Cylinders with 19 mm, 44 mm and 144 mm diameter are usable.	Two pistons in series realize positive and negative displacement which depend from piston diameter, spindle pitch and rotation frequency of stepper motor. By fitting the rotation frequency, the flow rate will be matched to the flow rate of MUT. The matching is checked by flow indication in connection line between pistons or by pressure stability if valve V is closed.

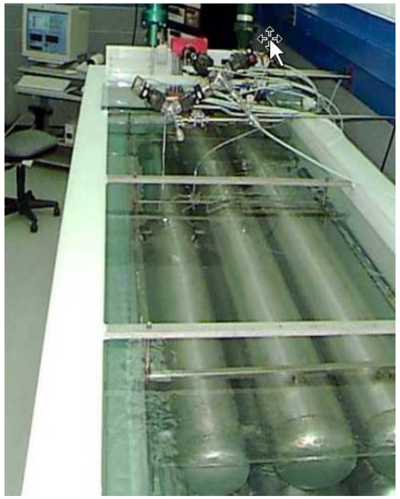
CMI - Czech metrology institute	
	Facility
Range of flow rate	1 mL/min to 20000 mL/min
Working temperature	20 °C ± 5 °C at meter under test
Working pressure	20 kPa abs to 600 kPa abs at meter under test
Expanded uncertainty U_{base} (k = 2)	(1 - 3) mL/min: $-0.22248 \cdot Q + 0.8895$, where Q is in mL/min, (3 to 20000) mL/min: 0.1 %
Picture of the facility	 Schematic of primary low mass flow standard in new vacuum vessel: 1: Gas bottle and pressure regulators. 2: Electronic balance. 3: Catenary capillary. 4: Mass flow controller. 5: Molbloc. 6: Molbox. 7: Automated mass handler. 8: Reference mass. 9: Ambient conditions measurement. 10: Vacuum pump. 11: Vacuum vessel. 
System description	The dynamic primary gravimetric system in vacuum or hermetic mode measures and calculates the mass flow from the decrease of mass of a pressurized cylinder filled with a pressurized gas. This cylinder is exhausted through the stabilization flow meter and rests on an electronic balance with an automated mass handler for zeroing the balance, calibrating the balance and placing the pressurized cylinder on the balance during the measurement. The calculated flow is corrected for the effects upon the system during measurement. The accuracy of the gravimetric standard depends on (in) accuracy of buoyancy corrections which are the main contribution to the uncertainty. Placing the pressurized cylinder s and the weighing system into a vacuum recipient decreases these influences to practically zero. VIČAR, M., KRAJÍČEK, Z., PRAŽÁK, D., SEDLÁK, V., GRONYCH, T., HAJDUK, T., TESAŘ, J.: Gravimetric flow standard in the vacuum and hermetic modes. *Measurement Science and Technology*, Vol. 29 (2018), 095011, 7pp. doi: 10.1088/1361-6501/aad1e2

METAS	
	Facility
Range of flow rate	3 mL/min to 30000 mL/min
Working temperature	(20.0 ± 0.1) °C
Working pressure	Ambient pressure (95 ± 2) kPa
Expanded uncertainty U_{base} (k = 2)	0.1 % to 0.2 %
Picture of the facility	
System description	The primary volumetric standard consists of a set of three precision-machined glass cylinders with mercury-sealed pistons. The velocity of the piston is calculated from the continuously measured difference in height of the raising piston by stabilised HeNe-laser interferometry and the elapsed time signal directly taken from the Swiss time standard. By using pressure sensors below the pistons and temperature sensors on the glass surface, the volume flow can be referenced to standard conditions.

KRISS	
	Facility
Range of flow rate	(10 to 20000) mL/min
Working temperature	(20 to 24) ℃
Working pressure	(100 to 700) kPa
Expanded uncertainty U_{base} (k = 2)	0.13 %
Picture of the facility	
System description	Piston prover of KRISS consists of six cylinders made of glass tubes with different diameters, mercury sealed pistons, a laser interferometer and a timer. The flow rate is from 10 mL/min to 20 L/min which can be generated by selecting one of the six cylinders. The working flow is passed through the flow meter and finally flow feeds into a mercury sealed piston. The timer and interferometer are synchronized to measure the travelling time and length of piston.

NMIJ		
	Facility 1	**Facility 2**
Range of flow rate	(4.2 to 337,620) mL/min	(4.2 to 337,620) mL/min
Working temperature	(22 to 24) ℃	(22 to 24) ℃
Working pressure	(50 to 700) kPa	(50 to 700) kPa
Expanded uncertainty U_{base} (k = 2)	(0.05 to 0.46) %	(0.07 to 0.75) %
Picture of the facility		
System description	Balance system is the primary standard for critical nozzle calibration, ranging from 4.2 mL/min to 337,620 mL/min at standard conditions 296.15 K and 101.325 kPa. The facility is based on the static gravimetric method, and the mass flow rate is calculated from the mass of gas which is accumulated in the measuring cylinder.	Critical nozzles are the secondary standard for a gas meter calibration whose range is the same as those of the balance system. These nozzles are calibrated with the balance system. The facility, in which a nozzle was installed, was used at this KC.

NMIA	
	Facility
Range of flow rate	2×10^{-6} m³ h^{-1} to 100 m³ h^{-1}
Working temperature	20 °C ± 0.5 °C
Working pressure	(0 to 700) kPa
Expanded uncertainty U_{base} (k = 2)	0.012 % to 0.03 %
Picture of the facility	
System description	The NMIA's 670 L PVTt standard consists of 8 cylinders connected in parallel. The standard can operate with either an 80 L or a 670 L volume. During measurement, the device under test (DUT) is placed at inlet of the PVTt with the gas flowing from DUT to the PVTt. The mass flow rate passing through the meter is determined from measurements of pressure, temperature and knowledge of the chemical composition of the gas used (e.g.: equation of state) at start and end of gas deposition. A three-way valve with limit switches in conjunction with a high-precision timer are used to measure the time interval of gas deposition. These measurements are then used to calculate the mass flow rate.
NIST	

	Facility 1
Range of flow rate	0.1 mL/min to 2000 L/min
Working temperature	23 °C ± 2 °C at meter under test
Working pressure	100 kPa to 2.5 MPa at meter under test
Expanded uncertainty U_{base} (k = 2)	1 % to 0.025 %
Picture of the facility	
System description	The 34 L and 677 L *PVTt* standard measures the change in mass of gas in a collection tank over a measured time interval. The mass change is calculated from pressure and temperature measurements of the gas and the volume of the collection tank. For flows ≤ 10 mL/min, the Rate of Rise (RoR) method was used to calculate flow. The RoR method uses the slope of a mass versus time plot to determine the mass flow.

10.2 Annex B: The re-test data from NMIJ

molbloc-L_C and molbloc-L _D revise results of NMIJ at 5 mL/min to 25 mL/min

1. Pilot Lab description:

In the Draft A version 1 report, the results of 10 mL/min and 25 mL/min of NNIJ are unusual. After pilot lab and NMIJ discussion, we found that the reason might cause from TS calibrated by the sonic nozzle used.

NMIJ based on Draft A version 1 report to check carefully, so that the mistakes were caught from the A7-T5 nozzle calibration. NMIJ re-calibrated the A7-T5 nozzle, as well as the balance system. The calibration traceability is showing in Figure B1. As a result, NMIJ obtained new calibration curves of discharge coefficients of A7-T5 sonic nozzle. Then, they re-calculated the results of unusual flow rates from the old data at the KC.

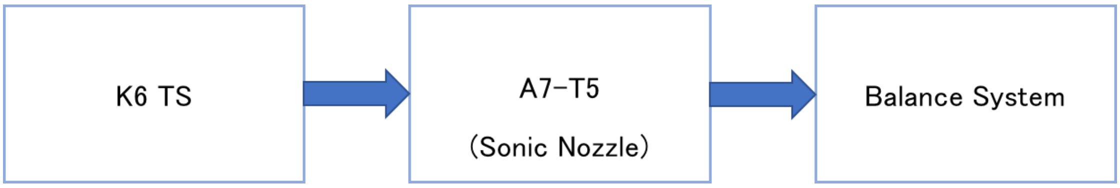


Figure B1: Low flow rate (5 mL/min to 25 mL/min) calibration traceability of the molbloc-L_C, molbloc-L_D in NMIJ

Therefore, NMIJ revised the results of 5 mL/min, 10 mL/min, and 25 mL/min, and then re-submitted it to the pilot lab on 22, April. Pilot lab based on newly submitted data from NMIJ to evaluate the E_n values of the 5 mL/min to 25 mL/min. The E_n values of all data are less than 1.

The renew results of molbloc-L_C, molbloc-L _D of NMIJ marked by "boldface" shown in Table B1.

Table B1: molbloc-L_C and molbloc-L_D revise result

	Q_{TS}	x_{ref}	$u(x_{ref})$	$U(x_{ref})$	x_i	U_{base}	d_i	E_n
	(mL/min)	(%)	(k=1, %)	(k=2, %)		(%)	(%)	
molbloc-L_C	25	0.067	0.016	0.033	**0.035**	**0.12**	**-0.032**	**-0.24**
molbloc-L_C	10	0.040	0.018	0.036	**0.016**	**0.14**	**-0.024**	**-0.15**
molbloc-L_D	10	0.090	0.021	0.043	**0.111**	**0.14**	**0.021**	**0.13**
molbloc-L_D	5	0.205	0.023	0.045	**0.072**	**0.19**	**-0.133**	**-0.66**

2. NMIJ description:

As mentioned at the pilot lab description, NMIJ re-calibrated not only the balance system (the primary standard) but also the A7-T5 sonic nozzle. Then, we re-calculated the flow rate results from the previous data by using those calibration curves of discharge coefficients obtained. We submitted the renewal data to pilot lab and asked for revision on 22, April. The new calibration curve of discharge coefficients (D.C.) of A7-T5 shown in Figure B2.

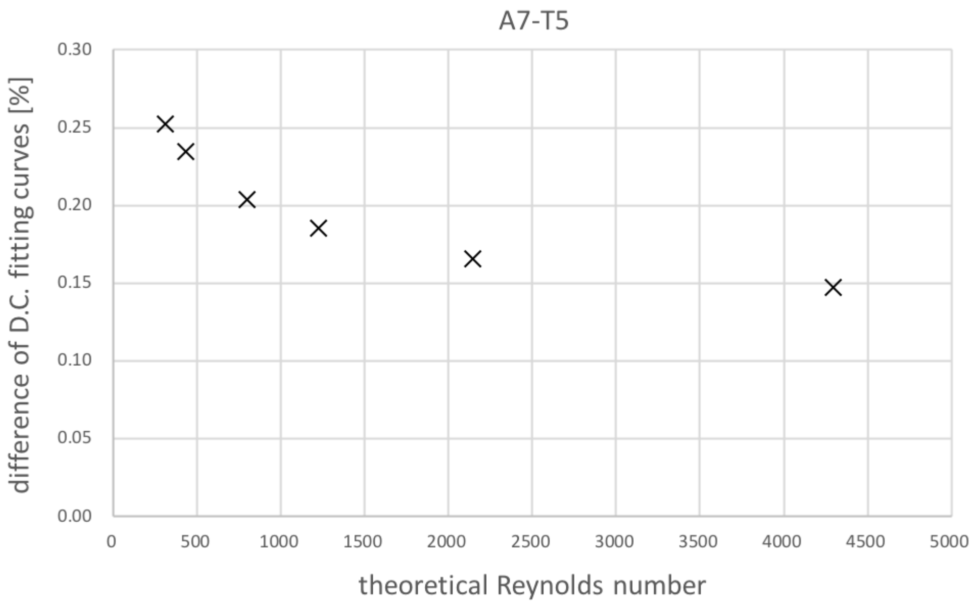


Figure B2: The re-calibration curves of discharge coefficients of A7-T5 nozzle.

3. Pilot lab conclusion:

A. The data of 5 mL/min, 10 mL/min and 25 mL/min of NMIJ in CCM.FF-K6.2017 comparison not used to calculate the KCRV.

B. NMIJ has confirmed that their sonic nozzle calibration system is as expected and needed.

10.3 Annex C: Comparison pass/fail criterion

The definitions and the results of the presently used comparison pass/fail/inconclusive criterion [3] are listed as follows:

- Criterion A: Participant *i* passes if $|E_\mathrm{n}| \le 1$ and fails if $|E_\mathrm{n}| > 1$.
  - Pass: $|E_\mathrm{n}| \le 1$
  - Fail(X) : $|E_\mathrm{n}| > 1$

- Criterion B:
  - Pass : $|E_\mathrm{n}| \le 1$ and $\left|\frac{U_\mathrm{TS}}{U_\mathrm{base,i}}\right| \le 2$
  - Fail(X) : $|E_\mathrm{n}| > 1$ and $\left|\frac{U_\mathrm{TS}}{U_\mathrm{base,i}}\right| \le 2$
  - ? : Otherwise

- Criterion D:
  - Pass: $P_i \ge 0.35$ and $|E_\mathrm{n}| \le 1$, where P_i is the probability content of the intervals (a_i, b_i) under the KCRV distribution; a_i and b_i are the 2.5th and 97.5th percentile confidence limits for lab i based on $u_{base,i}$, respectively.
  - Fail (X): $|E_\mathrm{n}| > 1$
  - ? : Otherwise

Q_{TS} =10000 mL/min(molbloc-L_A)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 7500 mL/min (molbloc-L_A)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 5000 mL/min (molbloc-L_A)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 2500 mL/min (molbloc-L_A)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 1000 mL/min (molbloc-L_A)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 1000 mL/min (molbloc-L_B)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	Pass

Q_{TS} = 750 mL/min (molbloc-L_B)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	?	Pass

Q_{TS} = 500 mL/min (molbloc-L_B)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	?	Pass

Q_{TS} = 250 mL/min (molbloc-L_B)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	?	Pass

Q_{TS} = 100 mL/min (molbloc-L_B)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Q_{TS} = 100 mL/min (molbloc-L_C)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	Pass
Criterion D	Pass	Pass	Pass	?	Pass	Pass	Pass	Pass	?	Pass

Q_{TS} = 75 mL/min (molbloc-L_C)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	Pass

Q_{TS} = 50 mL/min (molbloc-L_C)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	Pass	?	?	Pass

Q_{TS} = 25 mL/min (molbloc-L_C)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	[-]	X	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	[-]	X	?	Pass
Criterion D	Pass	Pass	Pass	Pass	?	Pass	[-]	X	?	Pass

Q_{TS} = 10 mL/min (molbloc-L_C)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	Pass	Pass	Pass	Pass	[-]	Pass	Pass	Pass
Criterion B	Pass	Pass	Pass	Pass	Pass	Pass	[-]	Pass	Pass	Pass
Criterion D	Pass	Pass	Pass	Pass	Pass	Pass	[-]	Pass	Pass	Pass

Q_{TS} = 10 mL/min (molbloc-L_D)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	X	Pass	X	[-]	[-]	X	Pass	Pass
Criterion B	Pass	Pass	X	Pass	X	[-]	[-]	X	Pass	Pass
Criterion D	Pass	Pass	X	Pass	X	[-]	[-]	X	?	Pass

Q_{TS} = 5 mL/min (molbloc-L_D)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	Pass	Pass	X	Pass	X	[-]	[-]	Pass	Pass	Pass
Criterion B	Pass	Pass	X	Pass	X	[-]	[-]	Pass	Pass	Pass
Criterion D	Pass	Pass	X	Pass	X	[-]	[-]	?	Pass	Pass

Q_{TS} = 2 mL/min (molbloc-L_D)	INRIM	LNE	PTB	CMI	METAS	KRISS	NMIJ	NMIA	NIST	CMS
Criterion A	X	Pass	Pass	Pass	X	[-]	[-]	Pass	X	X
Criterion B	X	Pass	Pass	Pass	X	[-]	[-]	Pass	X	X
Criterion D	X	Pass	Pass	Pass	X	[-]	[-]	Pass	X	X

10.4 Annex D: 2 mL/min data analysis by Median of Absolute Deviations (MAD)

The note from Cox[1] illustrated that the median can be expected to be more appropriate than the weighted mean if a number up to one third of the institute's measurements can be regarded as discrepant. The data of 2 mL/min in CCM.FF-K6.2017 comparison were matching condition of the note from Cox illustrated at 2002. Therefore, the pilot re-evaluated the KCRV of 2 mL/min by median and the results were shown in annex E. There were seven participants could pass the consistency check that listed in the table of outlier identification. KCRV could calculate by weighted case and un-weighted case, and the expression for the uncertainty in the KCRV were followed the methods from J. Randa[4],[5] . The t-distribution of the expended uncertainty at a 95 % level was 2.45 (degrees of freedom value was 6). Un-weighted Case 1 results were the best results that pilot lab suggested.

OUTLIER IDENTIFICATION

	METAS	PTB	NMIA	CMI	INRIM	CMS	NIST	LNE
x_i (%)	-0.330	0.037	0.072	0.151	0.221	0.244	0.393	0.545
$u(x_i)$ (%)	0.142	0.059	0.037	0.129	0.046	0.063	0.051	0.253
$U(x_i)$ (%)	0.283	0.119	0.075	0.259	0.092	0.127	0.101	0.505
median (x_i) (%)	0.186							
Abs (x_i-median(x_i))	0.516	0.149	0.114	0.035	0.035	0.058	0.207	0.359
MAD(n)	0131							
s(MAD(n)) =k(n) MAD(n)	0.219	0.219	0.219	0.219	0.219	0.219	0.219	0.219
2.5*s(MAD(n))	0.487	0.487	0.487	0.487	0.487	0.487	0.487	0.487
	Outlier	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Multiplier k value from J.Randa[5].

n	2	3	4	5	6	7	8	9	10	11
k(n)	1.773	2.206	2.019	1.800	1.764	1.686	1.671	1.633	1.626	1.602
n	12	13	14	15	20	25	50	100	1000	2000
k(n)	1.596	1.581	1.577	1.566	1.544	1.530	1.507	1.494	1.484	1.483

Weighted Case

$$KCRV = \sum_{i=1}^{N} \frac{x_i}{u^2(x_i)} u^2(KCRV)$$

$$u^2(KCRV) = \frac{1}{\sum_{i=1}^{N} \frac{1}{u^2(x_i)}}$$

KCRV (WM) (%)	0.182							
u^2(KCRV)	0.00046							
	METAS	PTB	NMIA	CMI	INRIM	CMS	NIST	LNE
Abs (*KCRV*-x_i) (%)	0.51	0.15	0.11	0.03	0.04	0.06	0.21	0.36
U_{95} (*KCRV*-x_i) (%)	0.35	0.14	0.08	0.31	0.10	0.15	0.11	0.62
Abs(*KCRV*-x_i)/U_{95} (*KCRV*-x_i)	1.46	1.07	1.47	0.10	0.39	0.43	1.87	0.59

Un-weighted Case 1

$$KCRV = \frac{1}{N}\sum_{i=1}^{N} x_i$$

$$u^2(KCRV) = \frac{1}{N(N-1)}\sum_{i=1}^{N}(x_i - KCRV)^2$$

KCRV (UWM) (%)	0.237							
$u^2(KCRV)$	0.00462							
	METAS	PTB	NMIA	CMI	INRIM	CMS	NIST	LNE
Abs ($KCRV$-x_i) (%)	0.57	0.20	0.17	0.09	0.02	0.01	0.16	0.31
U_{95} ($KCRV$-x_i) (%)	0.39	0.21	0.18	0.32	0.19	0.21	0.20	0.55
Abs ($KCRV$-x_i)/U_{95} ($KCRV$-x_i)	1.47	0.97	0.90	0.27	0.09	0.03	0.79	0.56

Un-weighted Case 2

$$KCRV = \frac{1}{N}\sum_{i=1}^{N} x_i$$

$$u^2(KCRV) = \frac{1}{N^2}\sum_{i=1}^{N} u^2(x_i)$$

KCRV (UWM) (%)	0.237							
$u^2(KCRV)$	0.00192							
	METAS	PTB	NMIA	CMI	INRIM	CMS	NIST	LNE
Abs ($KCRV$-x_i) (%)	0.57	0.20	0.17	0.09	0.02	0.01	0.16	0.31
U_{95} ($KCRV$-x_i) (%)	0.35	0.16	0.13	0.29	0.14	0.17	0.15	0.53
Abs ($KCRV$-x_i)/U_{95} ($KCRV$-x_i)	1.63	1.23	1.25	0.30	0.12	0.04	1.04	0.58