Final report on the key comparison, CCM.P-K15 in the pressure range from 1.0 x 10^{-4} Pa to 1.0 Pa

Christian Wuethrich[1], Kenta Arai[2], Mercede Bergoglio[3], James A. Fedchak[4], Karl Jousten[5], Seung Soo Hong[6], Jorge Torres Guzman[7]

November 2016

[1] *METAS, Lindenweg 50, 3003 Bern Wabern, Switzerland*
[2] *NMIJ-AIST, Tsukuba Central 3, Umezono 1-1-1, Tsukuba, Ibaraki 306-8563, Japan*
[3] *INRIM, Strada delle Cacce 91, 10135 Torino, Italia*
[4] *NIST, 100 Bureau Drive MS 8364, Gaithersburg, MD 20899-8364, USA*
[5] *PTB, Abbestr. 2-12, 10587 Berlin, Germany*
[6] *KRISS, 1 Doryong-Dong, Yuseong-Gu, Taejon 305-340, Rep. of Korea*
[7] *CENAM, km 4.5 carretera a los Cués, El Marqués, Qro, C.P. 76246, MÉXICO*

Abstract

The comparison CCM.P-K15 is a key comparison in pressure involving six laboratories in three regional metrological organizations (RMO). The measurand of the comparison is the accommodation coefficient of two spinning rotating gauge characterized in nitrogen from 0.1 mPa up to 1.0 Pa.

The two transfer standards were circulated from November 2009 until March 2011. The circulation consisted of three loops, one for each RMO, and a new calibration by the pilot between each loop. The stability of one of the transfer standards was poor and was worse than expected based on the previous history of the transfer standard while the other transfer standard demonstrated good stability while circulated in Europe and America and a fair stability while circulated in Asia.

All the participants demonstrated equivalence to the definition of pressure in their respective primary standards.

Content

Index of figures

Index of tables

1 Introduction

At the CCM WG-LP meeting in Paris in April 2008 it was decided to achieve a key comparison in the range 0.1 mPa to 1 Pa in order to assess the equivalence of the definition of this important vacuum range within the different regional metrological organisations (RMOs). This comparison is the successor of the CCM.P-K9, which was made during 1981 to 1987.

At the same CCM WG-LP meeting, it was decided that a new procedure, described in section 2, would be used to designate the participants within each participating RMO.

Three RMOs decided to take part, and for each RMO, as described in section 2, two participating laboratories were selected. APMP was represented through NMIJ and KRISS, CIM was represented through CENAM and NIST and Euramet was represented by PTB, INRIM and METAS which is pilot.

Two spinning rotor gauges (SRG) and one control electronic were used as transfer standard. The circulation of the transfer standard was organised as a loop for each RMO with a measurement by the pilot between each RMO.

A reference value was determined based on a weighted mean of the results. All the participants demonstrated equivalence to the definition of the pressure.

2 Participating laboratories.

Seven laboratories, including the pilot, took part in the comparison. The participants cover three regional metrological organisations (RMOs). The participants were selected, by the chairman of each RMO, based on technical capabilities, as decided at the CCM WG-LP in Paris in April 2008. The pilot laboratory was chosen to be METAS and was designated at the same WG-LP meeting due to a recent experience in a similar comparison in Euramet community.

The following table summarizes the applied methods and the status of the participant's standards at the time of the comparison.

Table 1: Characteristics of the definition of the pressure by the participants.

Laboratory	Standard	Definition	Traceability	CMC
NMIJ	Static expansion system	Primary	Independent	YES
KRISS	Dynamic expansion system	Primary	Independent	YES
CENAM	Static expansion system	Primary	Independent	YES
NIST	Dynamic expansion system	Primary	Independent	YES
INRIM	Continuous and static expansion system	Primary	Independent	YES
PTB	Static expansion system	Primary	Independent	YES
METAS	Static expansion system	Primary	Independent	NO

2.1 NMIJ

2.1.1 Equipment of NMIJ

NMIJ relies on a static expansion system for the definition of the pressure in this comparison.

The system consists of four chambers, a reference chamber (indicated as A), an initial chamber (B), a sub-expansion chamber (C) and a main chamber (D) with 0.2, 6, 10 and 170 L in volume, respectively. All chambers are made of stainless steel. The gauge to be calibrated is attached on the main chamber D. The chambers A, C and D are connected each other by all metal type pneumatic actuated valves controlled by a personal computer. Vacuum pump systems are also connected to the chambers through pneumatically actuated valves.

The sensitivity of the test gauge S is measured by exposure to a standard pressure P_{cal} in the chamber D and a reading of the test gauge P_{test},

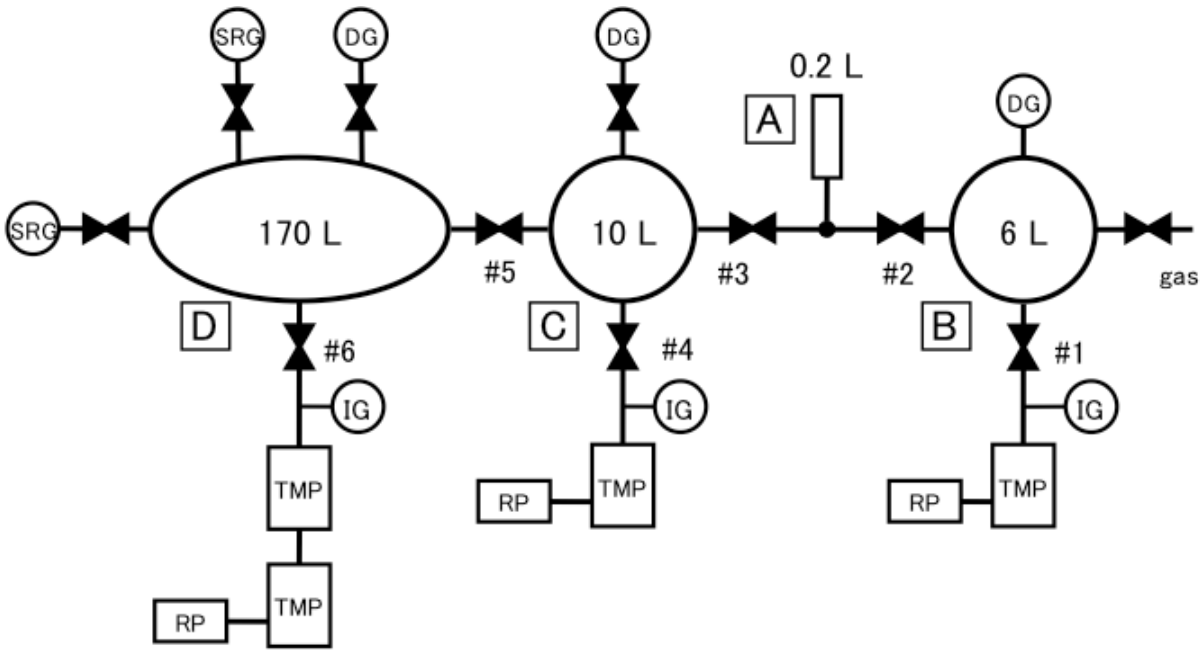

Fig 1: Static expansion system used by the NMIJ for this work.

The standard pressure P_{cal} is generated by various expansion steps. First, a calibration gas is introduced into the chamber A and B. The pressure in these chambers is measured by a capacitance diaphragm gauge mounted on chamber B. The gauge was calibrated by a pressure balance. At the sub-expansion step, the gas in the chamber "A" with volume Va was expanded into chamber "A" and "C" with volume (Va + Vc) followed by the isolation of chamber "A" from "B". The volume ratio R50 of (Va + Vc) / Va is about 50. Depending on the target value of the standard pressure P_{cal}, the sub-expansion step may be skipped or repeated a proper number of times followed by an evacuation of gas in the chamber "C". At the main expansion step, the gas in the chamber "A" is expanded into chamber "C" and "D". The volume ratio R_{900} =(Va + Vc + Vd) / Va and is about 900.

2.1.2 Traceability of the system

Measurement of the volume ratio is performed by the expansion technique using the capacitance diaphragm gauge attached on chamber B. For an accurate measurement the volume changes due to the valve operation (open and closed motion) should be taken into account. The drive side (the side where the volume changes with operation) of the valve #2, #3 and #5 faces toward chamber A, C and D, respectively.

2.1.3 Calibration procedure

The gauges are mounted on the chamber D. The SRG1 is on the middle of the chamber and the SRG2 is on the top of the chamber. The measurement of the residual drag was performed just before the calibration.

2.2 KRISS

2.2.1 Dynamic expansion standard at KRISS

The primary standard for ultra-high vacuum (UHV) at KRISS used for this comparison is an orifice type dynamic expansion system as described in [1]. It consists of two dynamic calibration systems: one for high vacuum from 10^{-5} to 10^{-2} Pa, and the second for UHV from 10^{-7} to 10^{-5} Pa. The UHV system is connected to the HV by a porous plug having a very small conductance ($6.36 \cdot 10^{-3}$ L/s in nitrogen at 23 °C).Gas is supplied to the high vacuum system from a constant pressure type flowmeter, some of which flows through the porous plug into the UHV chamber. The HV system is evacuated using a turbomolecular pump with a pumping speed for nitrogen of 345 L/s, and the UHV system is evacuated using a closed loop helium refrigerator type cryopump with a pumping speed for nitrogen of 1500 L/s.

2.2.2 Measurements at KRISS

The transfer standards were connected on the vacuum chamber on 1 July 2010. The measuring head (drive, magnetic levitation and sensing) provided showed an error and KRISS had to use its own measuring head to perform the measurements.

2.3 CENAM

The static expansion system has 4 volumes, as described in table 2 and shown in Figure 2.

Table 2: SEE-1 volumes.

Identification	Nominal volume
V_1	0.5 L
V_2	50.0 L
V_3	1.0 L
V_4	100.0 L

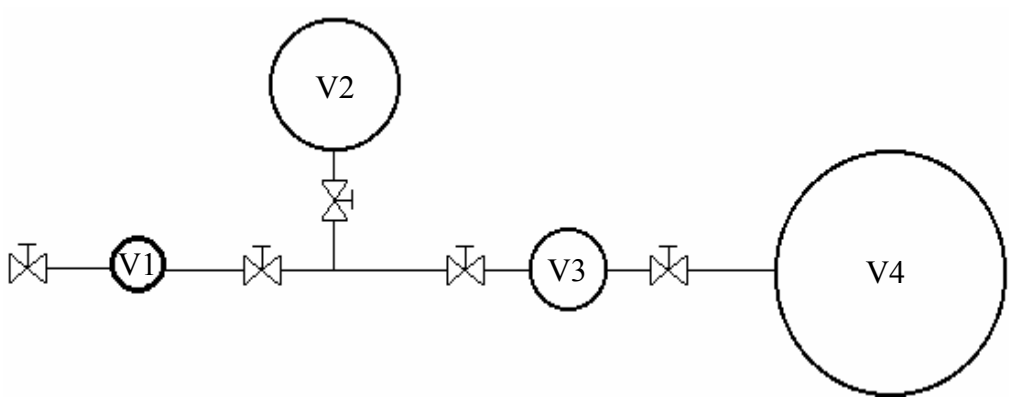

Fig 2: SEE-1 diagram.

In the SEE-1 the calibration chamber is V_4. It is possible to perform various expansions before the calibration pressure is achieved. Table 3 shows the different expansion paths in the SEE-1.

Table 3: Expansion paths identification.

Identification	Expansion path
f_A	$V_1 \rightarrow V_1+V_x+V_2$
f_B	$V_1 \rightarrow V_1+V_x+V_2+V_3$
f_C	$V_3 \rightarrow V_3+V_4$

The expansion paths can be combined. Table 4 shows the pressure ranges which can be obtained by the SEE-1.

Table 4: Expansion paths required according to the pressure range.

Pressure range	Expansion paths
10^{-5} Pa to 10^{-3} Pa	f_A, f_A, f_B, f_C
10^{-3} Pa to 10^{-1} Pa	f_A, f_B, f_C
10^{-1} Pa to 10^{1} Pa	f_B, f_C
10^{1} Pa to 10^{3} Pa	f_C

The devices under test were placed around chamber 4 at mid height.

The determination of the residual drag of the ball was made during the measurement.

2.4 NIST

All of the data were collected with the two SRG transfer standards connected to the NIST mid-range vacuum standard. The NIST mid-range vacuum standard is dynamic-expansion or orifice-flow vacuum standard similar to that described in Tilford *et al.* [2]. The constant pressure flowmeter used for the mid-range standard is described in McCulloh *et al*. [3]. A unique feature of the NIST mid-range standard is that different two orifice plates, of nominal diameters 11 mm and 2 mm, may be used and these may be switched without venting the vacuum chamber. The 11 mm orifice and the constant pressure flowmeter was used to generate pressures range of 10^{-4} Pa to 0.3 Pa. For the pressure point at 1 Pa, the 2 mm orifice was used, but the pressure was realized by a direct comparison to a capacitance diaphragm gauge (CDG).)).For the pressure range of 10^{-4} Pa to 0.3 Pa, the main contributions to the uncertainty in the generated pressure are the uncertainties due to the flow generated from the flowmeter, U_{fm}, the orifice conductance, U_C, the temperature U_T, and the pressure ratio across the orifice in the dynamic expansion chamber U_{PR}. U_{fm} is the largest component and typically ranges from about 0.2 % to 0.15 % for the gas flows required over this pressure range. However, several of the flows determined for the target pressure at 10^{-4} Pa had atypically large statistical contributions that increased the uncertainty to about 0.5 %. For the P-K15 study, U_T = 0.06 %, U_{PR} = 0.02 %, and 0.05% < U_C < 0.1 % for target pressures ranging from 10^{-4} Pa to 0.3 Pa.

To generate a target pressure of 1 Pa, the 2 mm orifice plate was lowered into place, and a calibrated CDG, was connected to the dynamic expansion chamber. The 2 mm orifice has a known conductance, and it could have been used to generate a known pressure using the dynamic expansion technique. However, in this case, a direct comparison to the CDG was preferred since the uncertainty was lower than that of the dynamic-expansion technique with

the 2 mm orifice. The CDG was calibrated using the NIST 140 Pa oil manometer [4]. The particular CDG used in this comparison has a 15 year calibration history at NIST and its uncertainty due to calibration drift is well known. To minimize drift, the CDG is kept in a temperature controlled box and does not experience temperature variations or mechanical shock. The total standard uncertainty of the CDG is about 0.1 % at 1 Pa.

Since two orifice plates were used in this study, the entire range of pressures could not be generated in one day. The pressure range of 10^{-4} Pa to 0.3 Pa was generated on one day and the pressure of 1 Pa was generated on different day. Data was collected on four separated days. The labels “Day 1” or “Day 2” correspond to a full pressure range of data taken over two days. These labels were used to be consistent with the labels given in the data spreadsheets submitted to the pilot lab.

Residual drag (*RD*) data were collected for at least 12 hours before each measurement day. A linear fit of *RD* versus frequency, *f*, was then performed to determine the slope and offset of the *RD* at *f* = 410 Hz. Values of *RD* were measured every day for four consecutive days to determine the repeatability of the measurement. We take the standard deviation of the offset for these four days to be the *k*=1 uncertainty of the *RD* measurement. For SRG 1 we found that *RD* was $7.2413 \times 10^{-7} \pm 6.5 \times 10^{-10}$ s^{-1}, and for SRG 2 we measured $1.6747 \times 10^{-7} \pm 2.4 \times 10^{-10}$ s^{-1}; both are *k* = 1 Type A uncertainties.

Finally, we note that we had some problems keeping SRG 2 suspended at a pressure of 1 Pa. This rotor would periodically become un-suspended over the course of the data acquisition.

2.5 INRIM

INRIM made the measurements on two primary pressure standards according to the pressure range.

2.5.1 Continuous expansion system

The INRIM continuous expansion system [5] works in the range between 1×10^{-6} Pa and 9×10^{-2} Pa. It is mainly composed by a primary flowmeter [6], a calibration chamber and a pumping chamber connected by a conductance C. The flowmeter is based on constant-pressure and variable-volume method and it can generate and measure molar flow rate in the range between 10^{-12} mol/s and 10^{-7} mol/s.

The system is equipped with pressure and temperature transducers referred to INRIM interferometric manobarometer, INRIM static system and to ITS90 scale.

2.5.2 Static expansion system

The INRIM static expansion system works in the pressure range between 0,09 Pa e 1000 Pa [7]; It is formed by three volumes of about 0.05 m^3 (V_1), 5×10^{-4} m^3 (V_3) e 1×10^{-5} m^3 (V_2), a turbo-molecular pumping system (residual pressure of about 10^{-6} Pa) and it is equipped with pressure and temperature transducers referred to INRIM interferometric manobarometer and

to ITS90 scale.

2.5.3 Stability of transfer standards

The rotor 1 (G191993) has shown an evident instability of the measured signal, both inside each day and in different measurement cycles, especially when connected to static system. Many measurements have been performed but the problem has not been solved and we have been unable to find the origin of the problem. We decided to send only results concerning rotor 2 as it was obvious there was an error on the measurement of rotor 1.

2.6 PTB

Two primary standards of the PTB, both realizing pressure using the static expansion method, were involved in the comparison.

The system, called SE1, was the one that covered the whole pressure range. In SE1 the pressures are generated by expanding gas of known pressure from a very small volume V4 of 17 mL directly into a volume of 233 L, or, alternatively, by two successive expansions from a volume V1 = 17 mL into an intermediate volume of 21 L including V4 and then from V4 into the 233 L vessel. The regular operational range of SE1 is 10^{-6} Pa up to 7 Pa.

Due to the relatively high volume ratios, the initial pressure had to be reduced below 1 kPa for some target pressures of this comparison, e.g. at 9 mPa. For this reason the uncertainty of the initial pressure measurement is higher and contributes significantly in an intermediate pressure regime. The system is described in more detail in references [10] and [11]. SE1 underwent a major reconstruction in 2009 and 2010. The calibrations for this comparison were the first after the reconstruction except of an internal comparison with SE2. A beat effect of two membrane pumps caused significant vibrations on SE1 for some hours which lead to an increase in the offset of the SRGs by a factor between 3 and 6. There were more "quiet" times in between, which were used for the measurements for this comparison, but the uncertainty of the measurements was affected nevertheless. Unfortunately, the reason causing the vibrations was discovered only after the measurements were finished.

The system, called SE2 [8, 9], served as a link of this comparison to CCM.P-K4, which was performed for a pressure range from Pa to 1 kPa. SE2 was one of the primary standards compared in CCM.P-K4. Only the target pressure of 1 Pa was measured with SE2. In order to calculate the measured data for 1 Pa more accurately, also some data at 0.09 Pa and 0.3 Pa were taken.

2.7 METAS

2.7.1 Reference system.

METAS realizes the pressure from 0.01 mPa to 100 Pa using a static expansion system with up to four expansion stages. Each stage has a typical expansion ratio of 100 except the last

expansion stage that has a double expansion scheme with expansion ratios of either 50 or 200. The maximum pressure reduction is then 2 x 10^{-8}. The initial pressure is regulated by a pressure generator PPC3 from DH-Instruments, the temperature of all the chambers is measured with an uncertainty of 0.1 K. The expansion process is automated and the closing time of the valves is optimized to avoid dynamic effects.

The expansion ratio of each stage has been characterized using the technique by addition as well as by depletion. [13].

The system is made of stainless steel chambers connected with conflat gaskets. The valves use polymer gaskets for the sealing with the chamber.

2.7.2 Measurements.

The measurements were made on the two SRG used as transfer standards at the same time. One of the SRG was connected to the MKS-SRG2 CE that was circulated and the other SRG was connected to an MKS-SRG2 that remained in METAS. On the second day of measurement the MKS-SRG2 units where swapped so that each SRG would be characterized using two different electronic units. The residual drag has been measured prior each measurement point. The value of the deceleration under pressure has been measured 5 times for pressure up to 30 mPa and 3 times for higher values. A linear regression is applied on the measurement points in order to compensate for possible outgassing at low pressure. The linear regression gives similar results to a mean value at high pressures. A spare SRG has been present on the last chamber during the time of the comparison to assess the stability of the system. Unfortunately the stability of this reference sensor was not better than 1 % for technical reasons that have nothing to do with the stability of the primary standard.

2.8 Uncertainty of the participants on the reference pressure

The participants have similar relative uncertainties in the highest pressures of the comparison. At the lowest pressures there are greater differences, mostly due to potential outgassing problems in the systems based on static expansion. KRISS does not cover all the range of the comparison and has the highest uncertainties.

Table 5: Average of the relative standard uncertainty on the definition of the reference pressure by the participants.

Pressure	NMIJ	KRISS	CENAM	NIST	INRIM	PTB	METAS
Pa							
1.0×10^{-4}	0.0039		0.0061	0.0039	0.0094	0.0044	0.0100
3.0×10^{-4}	0.0025		0.0037	0.0022	0.0082	0.0033	0.0050
9.0×10^{-4}	0.0023	0.0085	0.0035	0.0017	0.0064	0.0032	0.0050
3.0×10^{-3}	0.0026	0.0087	0.0026	0.0015	0.0050	0.0032	0.0030
9.0×10^{-3}	0.0024	0.0083	0.0024	0.0013	0.0039	0.0031	0.0030
3.0×10^{-2}	0.0020	0.0091	0.0023	0.0016	0.0038	0.0031	0.0030
9.0×10^{-2}	0.0022	0.0105	0.0023	0.0016	0.0015	0.0010	0.0030
3.0×10^{-1}	0.0023		0.0021	0.0019	0.0014	0.0009	0.0020
1.0	0.0022		0.0018	0.0010	0.0013	0.0009	0.0018

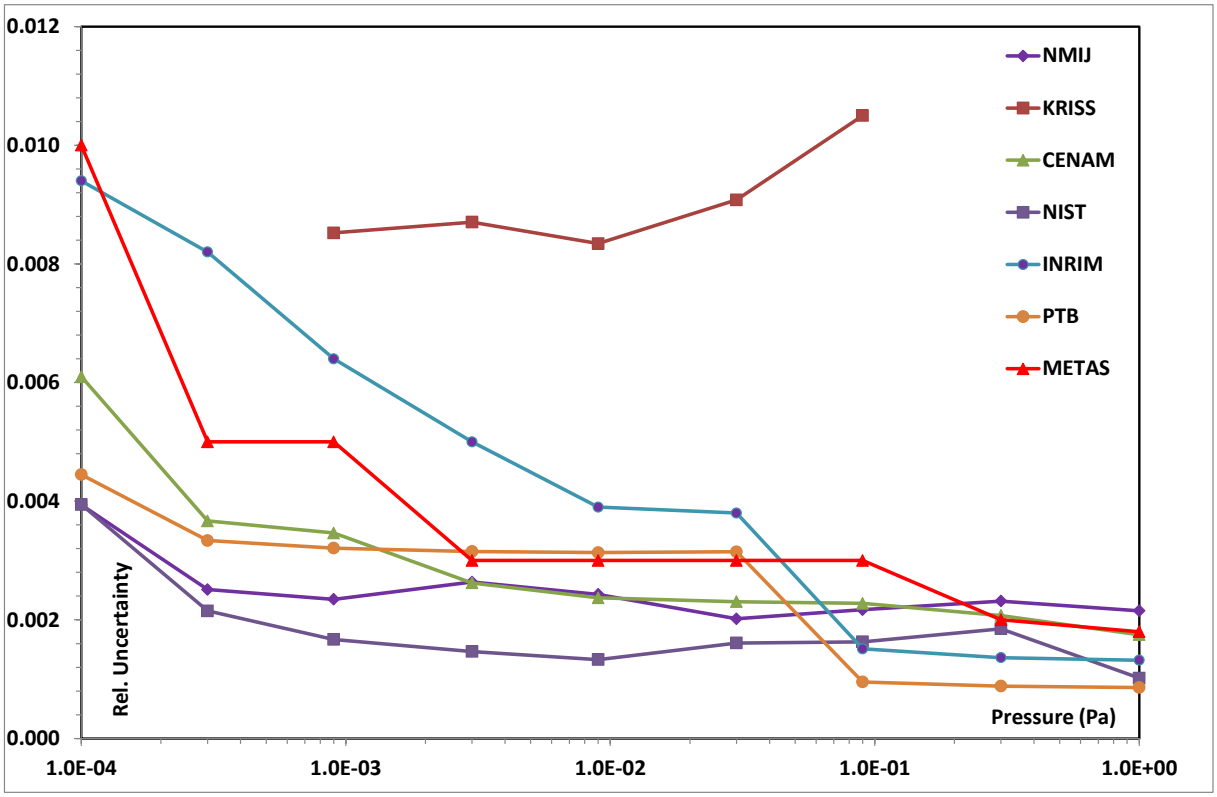

Fig 3: Relative uncertainty on the definition of the reference pressure by the participants plotted versus the pressure in Pa.

3 Transfer standard

The transfer standards consisted of a pair of SRG kept under vacuum using a Varian all-metal valve. The specifications of the transfer standard are listed in the table below. Some of the characteristics were not measured but are nominal values used to determine the accommodation coefficient.

Table 6: Characteristics of the transfer standard.

Transfer Standard	**SRG1**	**SRG2**
Metas Number	006771	006772
SRG Part Number (MKS)	MKS SRG-BF	MKS SRG-BF
SRG Serial Number	G191993	G192004
Valve part number	Varian 9515027	Varian 9515027
Valve Serial Number	LV0908L250	LV0908L249
Dead volume, valve open	120 cm^3 (u=1 cm^3)	120 cm^3 (u=1 cm^3)
Ball diameter (nominal)	4.5 mm	4.5 mm
Ball density (nominal)	7700 kg/m^3	7700 kg/m^3
Rotation frequency	405 – 415 Hz	405 – 415 Hz

A stainless steel spring was mounted on the plate of the valve and immobilised the ball when the valve was closed. In the open position, the spring was far enough from the ball such that the measurement of the residual drag did not show any spurious drag due to an electromagnetic coupling between the ball and the spring.

An electronic readout unit was circulated in conjunction with the transfer standard. The participating laboratories had the choice either to use the readout unit provided or their own unit. The characteristics of the readout unit were as follow:

Part Number (MKS): SRG-2CE
Metas number: 005555
Serial number 500163G

The rotation frequency of the ball was restricted to the range 405 Hz – 415 Hz. Preliminary measurements indicated that the frequency of rotation had a negligible effect on the uncertainty of the residual drag measurement; however this range of frequency is compatible with older electronic readout equipment.

4 Method used for the measurements

4.1 Correction of the residual drag

Two techniques were proposed for the determination of the residual drag of the SRG used for the correction of the deceleration measured under vacuum. The first technique is to measure the residual drag prior to the series of measurements at the different target pressure. The measurement has to be repeated for several values of rotation speed to determine the correct frequency-dependent residual drag. A second technique is to measure the residual drag before each measurement point. This second technique is well adapted to the measurements in a static expansion system as the SRG is under vacuum before each measurement.

4.2 Points of measurement.

The measurements were performed at 9 target pressure using nitrogen.

According to the protocol, the temperature of the system was to stay within 20 °C and 24 °C and the deviation of the actual pressure from the target pressure had to be less than 10% from the nominal value for points lower than 4.0 x 10^{-2} Pa, and less than 5% of the nominal value at higher pressure.

Each laboratory had to repeat each measurement point at least three times in each calibration sequence and the calibration sequence was repeated at least twice, making a total of at least 54 measurement points.

Depending on the target pressure, the duration of the measurement and the number of measurements recorded for the calculation of the average value was changed as shown in Table 7.

Table 7: Measurement method used for each target pressure.

Pressure	Time (sec)	Measurements	Repetitions
1.0 x 10^{-4} Pa	30	10	3
3.0 x 10^{-4} Pa	30	10	3
9.0 x 10^{-4} Pa	30	10	3
3.0 x 10^{-3} Pa	30	10	3
9.0 x 10^{-3} Pa	30	5	3
3.0 x 10^{-2} Pa	30	5	3
9.0 x 10^{-2} Pa	30	5	3
3.0 x 10^{-1} Pa	10	5	3
1.0 Pa	10	5	3

4.3 Circulation of the transfer standard

The circulation of the transfer standard was organised in three loops, one loop for each RMO. Between each loop the transfer standards where measured by the pilot laboratory to assess the stability of the transfer standard. The effective circulation was made according to the following schedule (Table 8).

Table 8: Schedule of the circulation of the transfer standard.

Loop			**Date**	**Laboratory**	**Index *l***
APMP			2010.03	METAS	1
APMP			2010.04	NMIJ	2
APMP			2010.06	KRISS	3
APMP	SIM		2010.07	METAS	4
	SIM		2010.08	CENAM	5
	SIM		2010.09	NIST	6
	SIM	EURAMET	2010.10	METAS	7
		EURAMET	2010.12	INRIM	8
		EURAMET	2011.01	PTB	9
		EURAMET	2011.03	METAS	10

4.4 Collection of the results

The results were collected directly by METAS from all the participants. An electronic work-sheet was provided to the participants to simplify the processing of the data by the pilot laboratory.

The participants had to provide the value of the pressure generated by the standard, the deceleration of the SRG while exposed to the pressure and under residual pressure conditions, the uncertainty of the deceleration under residual pressure (residual drag), the frequency of rotation, the temperature, and the uncertainty on the temperature.

5 Results provided by the participants.

The results of all the participants, including all the measurements made by the pilot are presented in Tables 9 and 10. The accommodation coefficient versus pressure is displayed in Fig 4 for SRG 1 and in Fig 5 for SRG 2. The value is the mean of the measurements made by the participants and the uncertainty is given by equation 25 for all participants, pilot included.

The plot shows a relatively wide spread of the measured values due to the drift of the accommodation coefficient of the SRG with time. The viscous flow effect is responsible for the decrease of the value of the accommodation coefficient at pressure higher than 0.03 Pa.

All participants provided results on the whole range covered by their reference standard except for two special situations.

INRIM did not provide any results for SRG1 as the measurement of the deceleration on SRG1 was unstable and not reproducible for a reason that was not explained during the time the transfer standard was at INRIM.

CENAM did not provide the results for SRG2 below 9.0 x 10^{-3} Pa as it appeared in the calculation of the accommodation coefficient that a leak was obviously present in the chamber once this sensor was mounted. The effect of the leak on the measurement could be neglected for measurement at 9.0 x 10^{-3} Pa and above.

Table 9: Accommodation coefficient (σ_{1l}) measured by the laboratories for the transfer standard SRG1 and uncertainties associated calculated using the Eq. 25.

P_j (Pa)		METAS1	NMIJ	KRISS	METAS4	CENAM	NIST	METAS7	INRIM	PTB	METAS10
1.0 x 10^{-4}	σ_{11}	1.0039	1.0087	-	0.9829	0.9703	0.9815	0.9750	-	0.9596	0.9635
	$u(\sigma_{11})$	0.0153	0.0302	-	0.0151	0.0366	0.0189	0.0151	-	0.0135	0.0150
3.0 x 10^{-4}	σ_{12}	1.0039	1.0063	-	0.9829	0.9854	0.9724	0.9750	-	0.9634	0.9635
	$u(\sigma_{12})$	0.0061	0.0105	-	0.0060	0.0112	0.0102	0.0060	-	0.0061	0.0059
9.0 x 10^{-4}	σ_{13}	1.0039	1.0062	0.9882	0.9829	0.9838	0.9794	0.9750	-	0.9639	0.9635
	$u(\sigma_{13})$	0.0053	0.0047	0.0096	0.0052	0.0044	0.0027	0.0051	-	0.0041	0.0051
3.0 x 10^{-3}	σ_{14}	1.0039	1.0063	0.9867	0.9829	0.9806	0.9768	0.9750	-	0.9626	0.9635
	$u(\sigma_{14})$	0.0032	0.0030	0.0091	0.0032	0.0030	0.0025	0.0032	-	0.0034	0.0031
9.0 x 10^{-3}	σ_{15}	1.0039	1.0061	0.9851	0.9829	0.9814	0.9774	0.9750	-	0.9652	0.9635
	$u(\sigma_{15})$	0.0032	0.0025	0.0090	0.0032	0.0030	0.0016	0.0032	-	0.0034	0.0031
3.0 x 10^{-2}	σ_{16}	1.0039	1.0055	0.9812	0.9829	0.9825	0.9769	0.9750	-	0.9644	0.9635
	$u(\sigma_{16})$	0.0032	0.0020	0.0094	0.0031	0.0023	0.0016	0.0031	-	0.0036	0.0031
9.0 x 10^{-2}	σ_{17}	1.0025	1.0043	0.9726	0.9817	0.9817	0.9761	0.9738	-	0.9620	0.9621
	$u(\sigma_{17})$	0.0032	0.0022	0.0105	0.0031	0.0023	0.0016	0.0031	-	0.0011	0.0031
3.0 x 10^{-1}	σ_{18}	1.0005	1.0005	-	0.9798	0.9782	0.9729	0.9715	-	0.9585	0.9601
	$u(\sigma_{18})$	0.0048	0.0023	-	0.0048	0.0024	0.0019	0.0048	-	0.0010	0.0048
1.0	σ_{19}	0.9870	0.9873	-	0.9674	0.9675	0.9625	0.9591	-	0.9468	0.9487
	$u(\sigma_{19})$	0.0025	0.0021	-	0.0025	0.0023	0.0011	0.0025	-	0.0009	0.0024

Table 10: Accommodation coefficient (σ_{2l}) measured by the laboratories for the transfer standard SRG2 and uncertainties associated calculated using the Eq. 25.

P_j (Pa)		**METAS1**	**NMIJ**	**KRISS**	**METAS4**	**CENAM**	**NIST**	**METAS7**	**INRIM**	**PTB**	**METAS10**
1.0 x 10^{-4}	σ_{21}	1.0029	1.0048	-	0.9917	-	0.9905	0.9948	0.9913	0.9930	0.9877
	$u(\sigma_{21})$	0.0142	0.0255	-	0.0141	-	0.0136	0.0141	0.0150	0.0547	0.0141
3.0 x 10^{-4}	σ_{12}	1.0029	1.0040	-	0.9917	-	0.9922	0.9948	0.9929	0.9875	0.9877
	$u(\sigma_{22})$	0.0055	0.0088	-	0.0055	-	0.0043	0.0055	0.0090	0.0094	0.0055
9.0 x 10^{-4}	σ_{23}	1.0029	1.0049	0.9946	0.9917	-	0.9940	0.9948	0.9926	0.9884	0.9877
	$u(\sigma_{23})$	0.0051	0.0036	0.0092	0.0050	-	0.0021	0.0050	0.0065	0.0052	0.0050
3.0 x 10^{-3}	σ_{24}	1.0029	1.0050	0.9951	0.9917	-	0.9937	0.9948	0.9927	0.9891	0.9877
	$u(\sigma_{24})$	0.0031	0.0028	0.0091	0.0031	-	0.0022	0.0031	0.0050	0.0053	0.0031
9.0 x 10^{-3}	σ_{25}	1.0029	1.0049	0.9938	0.9917	0.9933	0.9942	0.9948	0.9924	0.9898	0.9877
	$u(\sigma_{25})$	0.0031	0.0025	0.0088	0.0030	0.0027	0.0017	0.0030	0.0039	0.0045	0.0030
3.0 x 10^{-2}	σ_{26}	1.0029	1.0044	0.9901	0.9917	0.9918	0.9936	0.9948	0.9921	0.9887	0.9877
	$u(\sigma_{26})$	0.0030	0.0020	0.0095	0.0030	0.0024	0.0016	0.0030	0.0038	0.0048	0.0030
9.0 x 10^{-2}	σ_{27}	1.0016	1.0032	0.9816	0.9902	0.9904	0.9929	0.9937	0.9907	0.9864	0.9864
	$u(\sigma_{27})$	0.0030	0.0022	0.0106	0.0030	0.0023	0.0016	0.0030	0.0015	0.0011	0.0030
3.0 x 10^{-1}	σ_{28}	0.9997	0.9995	-	0.9884	0.9864	0.9899	0.9903	0.9865	0.9831	0.9839
	$u(\sigma_{28})$	0.0020	0.0023	-	0.0020	0.0024	0.0019	0.0020	0.0015	0.0009	0.0020
1.0	σ_{29}	0.9868	0.9868	-	0.9758	0.9764	0.9790	0.9786	0.9730	0.9710	0.9715
	$u(\sigma_{29})$	0.0018	0.0021	-	0.0018	0.0018	0.0012	0.0018	0.0014	0.0009	0.0018

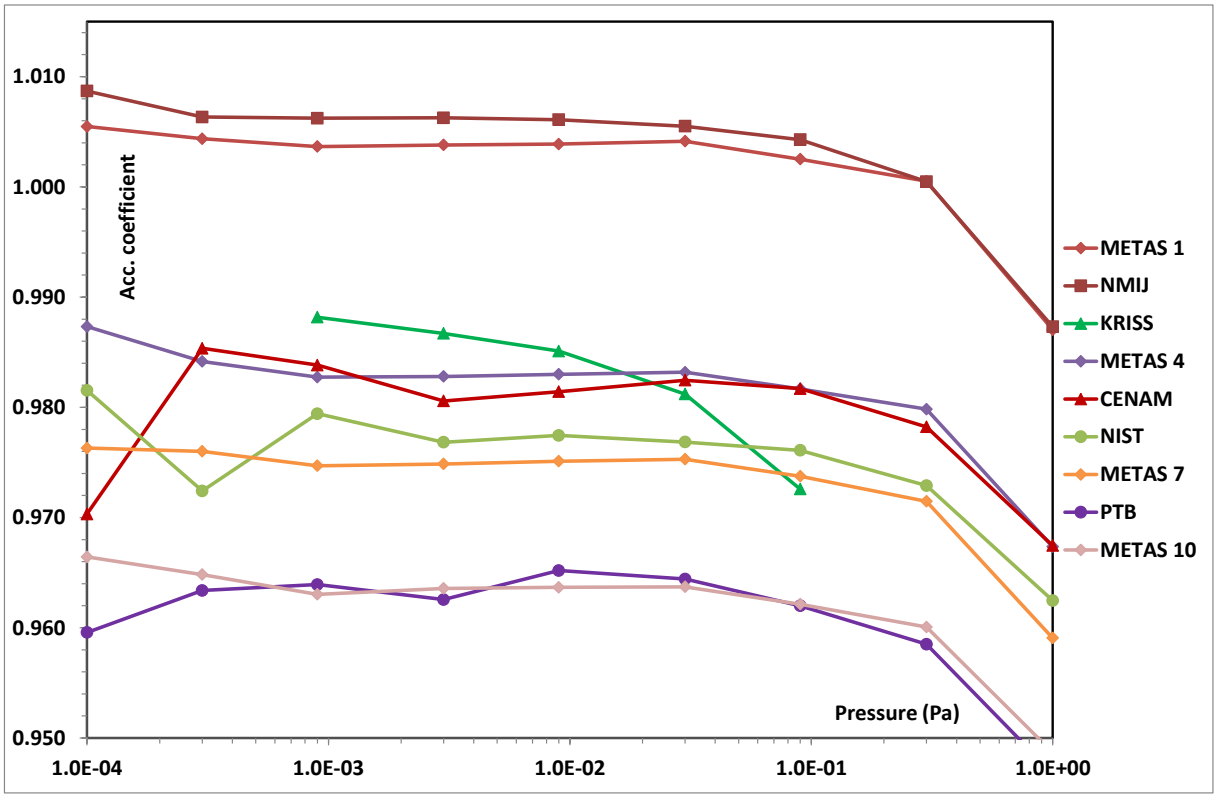

Fig. 4: Accommodation coefficient measured on the SRG1 by the different participants.

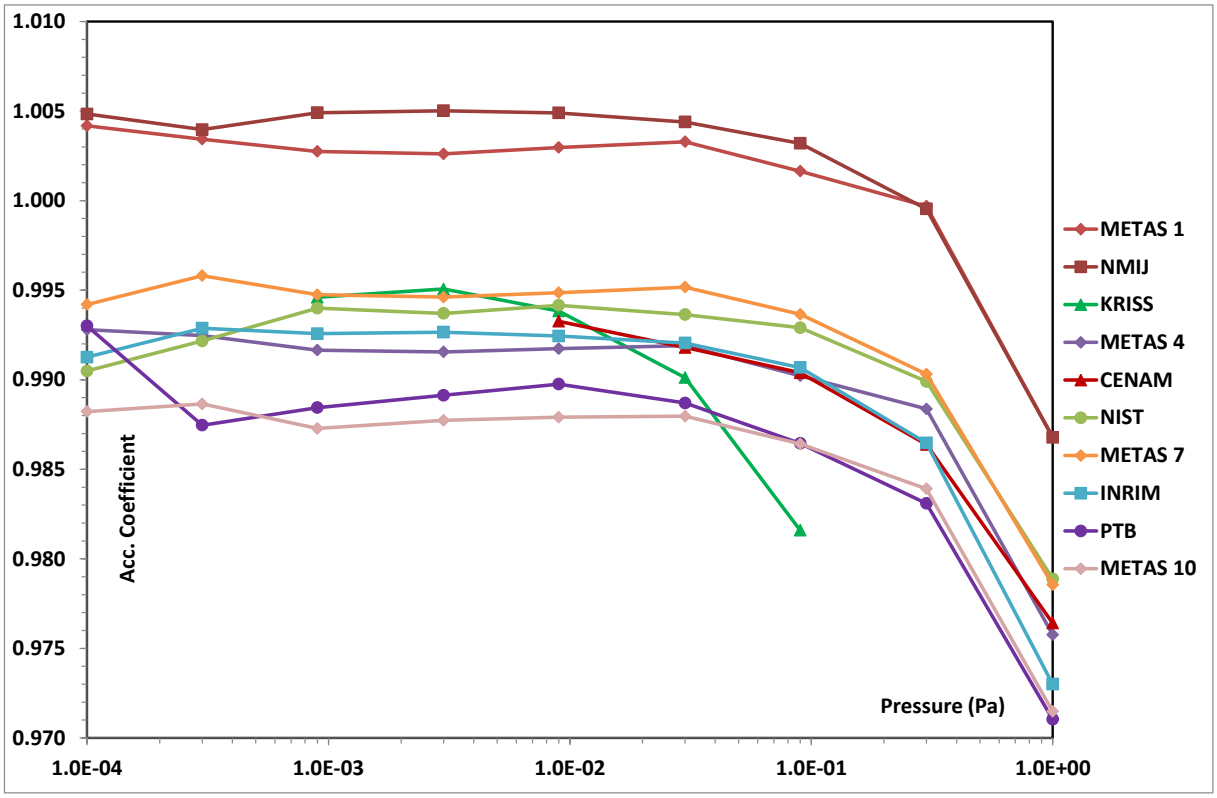

Fig 5: Accommodation coefficient measured on SRG2 by the different participants.

5.1 Measurements of the pilot laboratory and stability.

The transfer standard was measured by the pilot laboratory before the circulation, between each RMO and after the circulation.

The two transfer standards had shown excellent stability over five sets of measurements made before the comparison with a stability better than 0.2 %. Unfortunately SRG 1 exhibited a decrease of the accommodation coefficient larger than 1.5 % in the first loop and a continuous drift during the entire comparison and its final sensitivity dropped by as much as 3.8 %. Further investigation revealed that, during the transport, the ball was not correctly immobilized by the spring mounted on the valve even though the valve was closed. This possibly led to friction during transport and could, presumably, change the surface condition of the ball. SRG 2 had a large change of 0.7 % after the first loop, but remained satisfactory stable for the subsequent loops.

The drift of SRG 1 did not affect the uncertainty of the comparison much due to the weighted mean calculation applied in this work. (Eq. 8 or Eq. 12)

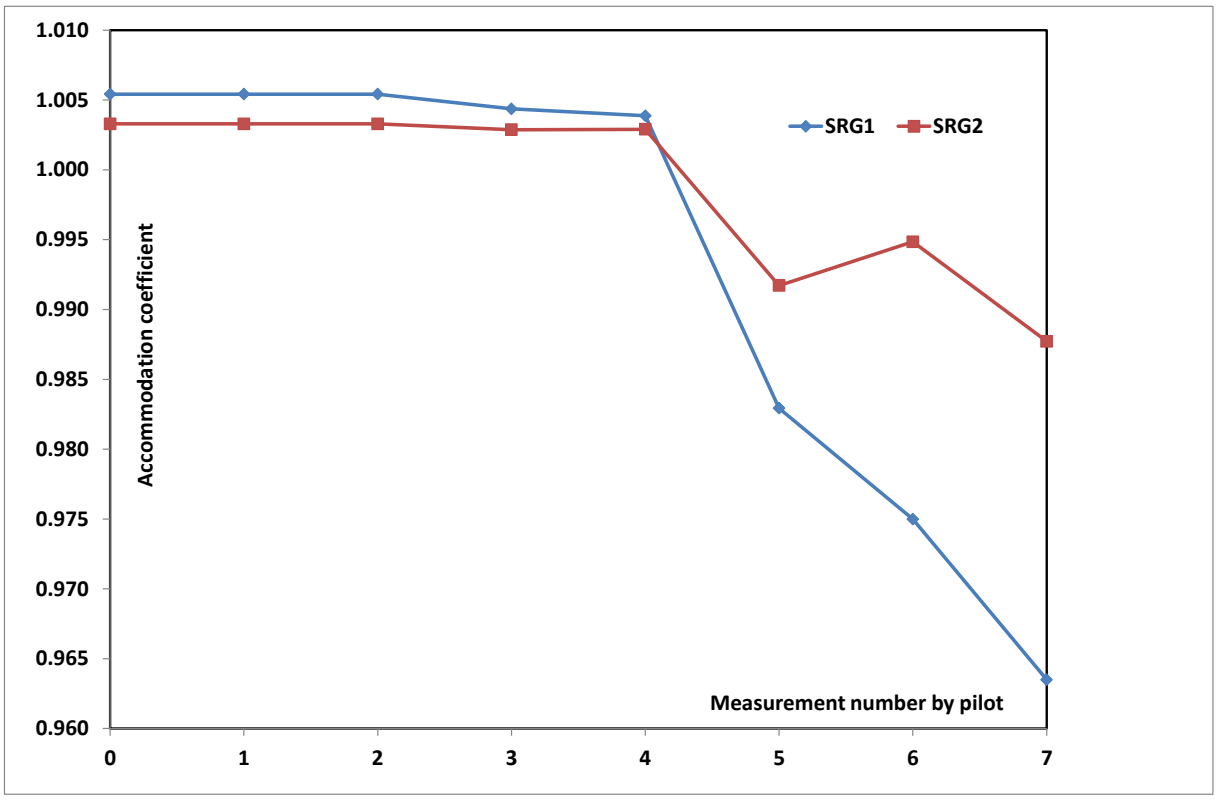

Fig. 6: Accommodation coefficient relative to the initial value for the SRG1 (blue) and SRG2 (red). The numbers on the horizontal axis denote the successive measurements performed by the pilot before and during the time of the comparison. The initial measurement used in this comparison corresponds to number 4. The measurements 4 to 7 are denoted respectively as METAS1, METAS4, METAS7 and METAS10 in the calculation.

6 Method used for the calculation of the reference value.

The measurand of the comparison is the accommodation coefficient of two SRG sensors determined over a range of pressures.

The measured values of the accommodation coefficient, for each participating laboratory, are given by:

$$\sigma_{ijkl}$$

Where

i is the number of the spinning rotating gauge
j is the number of the nominal target pressure
k is the number of the measurement in the series for a specific target pressure
l is the number of the measurement within the comparison (see table 10) and is also used to designate the NMI that did the measurement.

6.1 Influence of the reference pressure in the transition regime.

Since the value of the effective accommodation coefficient is dependent on the pressure in the transition regime, the measured accommodation coefficients have to be corrected to the target value of pressure for measurements made at nominal pressures of $9.0 \cdot 10^{-2}$ Pa and above. A linear regression is then made on the measurement and the slope is used to correct the actual measurement to what would have been obtained at the exact target pressure.

$$\sigma_{ijk} = \hat{\sigma}_{ijk} + \left(\widetilde{P}_j - \hat{p}_{ijk} \right) m_i \qquad (1)$$

Where:

$\hat{p}_{ijk}$ is the actual pressure generated by the standard
$\widetilde{P}_j$ is the target or nominal pressure j
$\hat{\sigma}_{ijk}$ is the accommodation coefficient measured at the pressure $\hat{p}_{ijk}$
m_i is the slope of the linear regression over the set of measurement [$\hat{p}_{ijk}$, $\hat{\sigma}_{ijk}$] for j=1 to 6 and k for the values where $p_k > 3.0 \cdot 10^{-2}$ Pa

The contribution to the uncertainty introduced by this correction will be neglected in the rest of the discussion as the $\hat{p}_{ijk}$ were very close to their respective p_k.

6.2 Correction of the drift of the transfer standard

As the transfer standard was not stable over the entire time of the comparison, it is necessary to make a correction of the drift by using the measurements made by the pilot laboratory at the beginning and at the end of each loop.

Since the value of the accommodation coefficient is not a function of the pressure in the molecular flow regime, it was decided to calculate an average value $\bar{\sigma}_{il}(METAS)$ for all meas-

urements made at pressure values of $3.0 \cdot 10^{-2}$ Pa or lower. This average value is calculated for measurements performed at nominal pressures between $9.0 \cdot 10^{-4}$ Pa and $3.0 \cdot 10^{-2}$ Pa

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{36}\sum_{k=1}^{k=6}\sum_{j=1}^{J=6}\sigma_{ijkl}(METAS) \quad j=1..6,\ l=1,\ 4,\ 7,\ 10 \tag{2}$$

The value of the accommodation coefficient at pressures above 3.0×10^{-2} Pa is calculated for each pressure point.

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{6}\sum_{k=1}^{k=6}\sigma_{ijkl}(METAS) \quad j=7..9,\ l=1,\ 4,\ 7,\ 10 \tag{3}$$

The reference value within a loop is determined by a weighted mean of the pilot laboratory accommodation coefficient values at the beginning and at the end of the loop.

For each participating NMI which is the first in the loop of its RMO, the value of the accommodation coefficient given by the pilot laboratory has been calculated by taking the average value:

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{3}\left(2\overline{\sigma}_{ij(l-1)}(METAS) + \overline{\sigma}_{ij(l+2)}(METAS)\right) \qquad l=2,\ 5,\ 8 \tag{4}$$

A similar way, for each participating NMI which is the last in the loop of its RMO, the value of the accommodation coefficient given by the pilot laboratory has been calculated by taking the average value:

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{3}\left(\overline{\sigma}_{ij(l-2)}(METAS) + 2\overline{\sigma}_{ij(l+1)}(METAS)\right) \qquad l=3,\ 6,\ 9 \tag{5}$$

This way of calculating the reference value is optimal to correct a linear drift of the transfer standard during the loop. It is also more robust than an average value in the case of a sudden shift due to an incident during the measurement by a participant or during the transport between the two participants of the same RMO.

6.3 Pressure value for a participating laboratory

The pressure realized in the participating laboratories is determined from the average response of the sensor measured before and after the participating NMI by the pilot laboratory. In a first step, the average response of the SRG is calculated:

$$\sigma_{ijl} = \frac{1}{6}\sum_{k=1}^{k=6}\sigma_{ijkl} \qquad l=2,\ 3,\ 5,\ 6,\ 8,\ 9 \tag{6}$$

Next, we calculate the effective pressure determined by the participating laboratory if the pressure generated by the laboratory was exactly the target pressure and the drift is removed from the accommodation coefficient:

$$p_{ij} = \widetilde{P}_j \frac{\sigma_{ijl}}{\overline{\sigma}_{ijl}(METAS)} \qquad l=2, 3, 5, 6, 8, 9 \tag{7}$$

6.4 Mean value for a participating laboratory

The mean value for a participating laboratory is the weighted mean of the predicted pressure value for each SRG [12]. It is weighted by the uncorrelated (mostly Type A) uncertainties. This has the advantage that if one SRG had a large drift and the other was stable during the transport, the result of the participating laboratory is less affected by the unstable SRG.

$$p_j = \frac{\sum_{i=1}^{i=2} \frac{p_{ij}}{u_A^2(p_{ij})}}{\sum_{i=1}^{i=2} \frac{1}{u_A^2(p_{ij})}} \tag{8}$$

6.5 Pressure value for the pilot laboratory

The accommodation coefficients determined by the pilot laboratory is determined from the average of the measurements at the beginning and at the end of each loop.

$$\sigma_{ij}(METAS, l, l+3) = \frac{1}{12} \sum_{k=1}^{k=6} \left[\sigma_{ijkl}(METAS) + \sigma_{ijkl+3}(METAS) \right] \qquad l=1, 4, 7 \tag{9}$$

Then the effective pressure determined by the pilot laboratory for each loop of the comparison is given by:

$$p_{ij}(METAS, l, l+3) = P_j \frac{\sigma_{ij}(METAS, l, l+3)}{\overline{\sigma}_{ij}(METAS, l, l+3)} \qquad l=1, 4, 7 \tag{10}$$

It should be mentioned that for nominal pressure above 3.0 x 10^{-2} Pa the value of the pressure of the pilot laboratory is always the nominal pressure.

Finally the pressure value of the pilot laboratory is given by the weighted mean value of the reference pressure for all the loops. The weight coefficient is the combination of all the type A uncertainties.

$$p_{ij}(METAS) = \sum_{l=1,4,7} \frac{\frac{p_{ij}(METAS, l, l+3)}{u_A^2(p_{ij}(METAS, l, l+3))}}{\frac{1}{u_A^2(p_{ij}(METAS, l, l+3))}} \tag{11}$$

6.6 Mean value for the pilot laboratory

The mean value of the pilot laboratory is obtained in a similar way as the participating NMI's, by a weighted mean value in which the weight coefficient is the combination of the type A uncertainties:

$$p_j(METAS) = \frac{\sum_{i=1}^{i=2} \frac{p_{ij}(METAS)}{u_A^2(p_{ij}(METAS))}}{\sum_{i=1}^{i=2} \frac{1}{u_A^2(p_{ij}(METAS))}} \tag{12}$$

6.7 Expected pressure value based on the mean pressure of the pilot laboratory

The expected pressure value based on the mean pressure provided by the pilot laboratory is obtained as a weighted mean value on all the participants.

$$p_j(CCM.P-K15, pilot) = \frac{\sum_l \frac{p(l)}{u^2(p(l))}}{\sum_l \frac{1}{u^2(p(l))}} \tag{13}$$

Where l designate the NMI's selected to provide the reference value according to the number of the loop where the NMI did its measurement.

6.8 Reference value of the comparison

The expected pressure value given in equation 13 is biased because in equation 7 we take into account only the accommodation coefficients defined by the pilot. It is allowed to multiply by the same ratio all the reference pressure for all the participants without affecting the uncertainty of the comparison. This way it is possible to obtain the reference pressure of the comparison as the nominal pressure. The coefficient of normalization is given by the weighted mean value among all the laboratories.

$$c_j = \frac{p_j}{p_j(CCM.P-K15, pilot)} \tag{14}$$

And this way, the reference value of the comparison is equivalent to the value of the target pressure:

$$p_j(CCM.P-K15) = p_j(CCM.P-K15, pilot)c_j \equiv P_j \tag{15}$$

6.9 Relative deviation to the reference value.

Due to the large span of pressures in this comparison, it is more convenient to express the deviation relative to the nominal value rather than in absolute number. This deviation is given by the following expression:

$$d_j = \frac{p_j}{P_j} - 1 \tag{16}$$

6.10 Degree of equivalence

Finally the degree of equivalence is given by the ratio between the deviation and the uncer-

tainty of the deviation. The degree of equivalence is given by:

$$E_j(l) = \frac{d_j(l)}{U(d_j(l))} \tag{17}$$

7 Method used for the determination of the uncertainty.

7.1 Uncertainty on sigma measured by the participants

In the following discussion the measured quantity has, in some places, been replaced by the presumed value of this quantity (for example the effective pressure seen by the sensor has been replaced by the nominal value of the pressure). This is for the simplification of the calculation and has only a negligible effect on the uncertainty calculation as both values are very close.

7.1.1 Equation of the SRG

The value of sigma is determined by using the relation between the deceleration and the pressure including the influence factors (temperature, residual drag)

$$\sigma_{ijk} = \left(DCR_{ijk} - RD_{ijk}(\omega)\right)\frac{\pi a \rho}{10\,\hat{p}_{ijk}}\sqrt{\frac{2RT_{ijk}}{\pi m}} \tag{18}$$

For clarity we will rewrite it by putting all the constants together:

$$\sigma_{ijk} = \left(DCR_{ijk} - RD_{ijk}(\omega)\right)\frac{K}{\hat{p}_{ijk}}\sqrt{T_{ijk}} \tag{19}$$

And the constant K is then given by:

$$K = \frac{\pi a \rho}{10}\sqrt{\frac{2R}{\pi m}} \tag{20}$$

The constant is the same for both SRGs as we have determined it based on the nominal value of mass, density and diameter. This way of doing has no influence on the uncertainty or final value as explained under 7.1.2.

The uncertainty of the sigma measured by the participants is estimated by taking into account the uncertainty of the generated pressure determined by the participants, the uncertainty on the residual drag, the uncertainty on the temperature of the SRG, as well as the standard deviation of the set of sigma measured.

7.1.2 Uncertainty on K

The uncertainty on the constant K has an influence on the calculation of the accommodation factor sigma. The factor K is used by all the participants and this way this uncertainty is correlated over all the participants. It is then cancelled in the calculation of the pressure measured by the participants.

7.1.3 Uncertainty of the reference pressure

The uncertainty of the generated pressure has been provided by the participants for each

measurement point. As the value of the relative uncertainty is not sensitive to small changes of pressure and since the values provided by the participants are similar from one cycle of measurement to another, only one value of uncertainty for each target pressure needs to be calculated. This uncertainty is a type B uncertainty and the sensitivity coefficient evaluated at the nominal pressure P_j is given by:

$$\frac{\partial \sigma_{ij}}{\partial P_j} = \left(DCR_{ij} - RD_{ij}(\omega)\right)\frac{-K}{P_j^2}\sqrt{T_j} \tag{21}$$

7.1.4 Contribution due to the uncertainty on the temperature

The collision rate of gas on the rotor depends on the temperature, therefore the SRG temperature uncertainty needs to be included in the combined uncertainty of the accommodation factor. The sensitivity coefficient of the temperature is given by:

$$\frac{\partial \sigma_{ij}}{\partial T_j} = \left(DCR_{ij} - RD_{ij}(\omega)\right)\frac{K}{2P_j}\sqrt{\frac{1}{T_j}} \tag{22}$$

7.1.5 Contribution due to the uncertainty on the residual deceleration

The residual deceleration at zero pressure is determined and used to correct the deceleration measured when the SRG is exposed to the gas. The sensitivity coefficient of the residual drag is given by:

$$\frac{\partial \sigma_{ij}}{\partial RD_i(\omega)} = -\frac{K}{P_j}\sqrt{T_j} \tag{23}$$

7.1.6 Type A uncertainty for σ

The standard deviation of a set of measured accommodation coefficients for a given SRG and a given nominal pressure is a type A uncertainty. It is generated by the repeatability of the measurement of the DCR due to non-systematic errors. This standard deviation has been corrected for a small sample size as explained by Kacker and Jones [14] to obtain the contribution to the uncertainty of sigma:

$$u(repeatibility) = \sqrt{\frac{n-1}{n-3}}s\left(\sigma_{ij}\right) \tag{24}$$

Where:

n: is the number of measurements
s: is the standard deviation

Finally the uncertainty on the accommodation factor is given by:

$$u^2\left(\sigma_{ij}\right) = \frac{n-1}{n-3}s^2\left(\sigma_{ij}\right) + \left(\frac{\partial \sigma}{\partial P_j}\right)^2 u^2\left(P_j\right) + \left(\frac{\partial \sigma}{\partial T_j}\right)^2 u^2\left(T_j\right) + \left(\frac{\partial \sigma}{\partial RD_i(\omega)}\right)^2 u^2\left(RD_i(\omega)\right) \tag{25}$$

It is useful for the calculation of the weighted mean value to determine the combination of the

uncorrelated uncertainties (mostly type A) to the accommodation coefficient.

$$u_A^{\,2}\left(\sigma_{ij}\right)=\frac{n-1}{n-3}s^2\left(\sigma_{ij}\right)+\left(\frac{\partial\sigma}{\partial T_j}\right)^2 u^2\left(T_j\right)+\left(\frac{\partial\sigma}{\partial RD_i(\omega)}\right)^2 u^2\left(RD_i(\omega)\right) \qquad (26)$$

7.2 Uncertainty on the value of the accommodation coefficient drift correction.

The uncertainty on the reference value used to compensate the drift of the SRG (the $\overline{\sigma}_{ijl}\left(METAS\right)$ as defined in equation 4 or 5) is given by the stability of the transfer standard. The uncertainty due to the stability of the SRG is defined the following way:

$$u\left(\overline{\sigma}_{ijl}\left(METAS\right)\right)=\frac{1}{2}\left|\overline{\sigma}_{ijl}\left(METAS\right)-\overline{\sigma}_{ij(l+3)}\left(METAS\right)\right| \qquad l=1,\ 4,\ 7 \qquad (27)$$

The minimal value for the uncertainty has been set to 0.0015 as a same value before and after a given NMI could also involve some canceling of the drift.

7.3 Uncertainty on sigma measured by the pilot

The uncertainty of the accommodation coefficient determined by the pilot laboratory is slightly different from the uncertainty on the coefficient of the participating NMI's as the value of the pilot is an average value of two measurements. We make the assumption that the value of the uncertainty is the same for each cycle of measurement of the accommodation factor. The terms that correspond to type B uncertainty are unchanged while the terms of type A are slightly reduced due to the larger numbers of measurement:

$$u^2\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)=\left(\frac{\partial\sigma}{\partial P_j}\right)^2 u^2\left(P_j\right)+$$
$$\frac{1}{4}\sum_{L=l,l+3}\left(\frac{n-1}{n-3}s^2\left(\sigma_{ijL}\right)+\left(\frac{\partial\sigma}{\partial T_j}\right)^2 u^2\left(T_j\right)+\left(\frac{\partial\sigma}{\partial RD_i(\omega)}\right)^2 u^2\left(RD_{iL}(\omega)\right)\right) \qquad l=1,\ 4,\ 7 \qquad (28)$$

The combination of the type A uncertainties is given by:

$$u_A^2\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)=$$
$$\frac{1}{4}\sum_{L=l,l+3}\left(\frac{n-1}{n-3}s^2\left(\sigma_{ijL}\right)+\left(\frac{\partial\sigma}{\partial T_j}\right)^2 u^2\left(T_j\right)+\left(\frac{\partial\sigma}{\partial RD_i(\omega)}\right)^2 u^2\left(RD_{iL}(\omega)\right)\right) \qquad l=1,\ 4,\ 7 \qquad (29)$$

It has to be recalled that the mean value of the accommodation coefficient of the pilot laboratory, used to link the measurements within a loop, as defined in equation 4 and 5, is not equivalent to the mean value defined by equation 12 used to link the loops within the comparison. The values are however very close and their respective uncertainties can be considered as equivalent.

$$u^2\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)=u^2\left(\overline{\sigma}_{ijl+1}\left(METAS\right)\right)=u^2\left(\overline{\sigma}_{ijl+2}\left(METAS\right)\right) \qquad l=1,\ 4,\ 7 \qquad (30)$$

Equivilantly, the same assumption is made for the combination of type A uncertainties:

$$u_A^2\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)=u_A^2\left(\overline{\sigma}_{ijl+1}\left(METAS\right)\right)=u_A^2\left(\overline{\sigma}_{ijl+2}\left(METAS\right)\right) \quad l=1,\ 4,\ 7 \qquad (31)$$

7.4 Uncertainty of the reduced pressure for the participants

The uncertainty of the reduced pressure can be treated as an incoherent addition of the relative uncertainty of the accommodation coefficient measured by the participating NMI and given by the pilot laboratory:

$$u\left(p_{ijl}\right)=\widetilde{P}_j\sqrt{\left(\frac{u\left(\sigma_{ijl}\right)}{\sigma_{ijl}}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ijl}\left(METAS\right)\right)}{\overline{\sigma}_{ijl}\left(METAS\right)}\right)^2} \qquad l=2,\ 3,\ 5,\ 6,\ 8,\ 9 \qquad (32)$$

Once again, the calculation of the combination of the type A uncertainty used for the weighted mean is given by:

$$u_A\left(p_{ijl}\right)=\widetilde{P}_j\sqrt{\left(\frac{u_A\left(\sigma_{ijl}\right)}{\sigma_{ijl}}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ijl}\left(METAS\right)\right)}{\overline{\sigma}_{ijl}\left(METAS\right)}\right)^2} \qquad l=2,\ 3,\ 5,\ 6,\ 8,\ 9 \qquad (33)$$

7.5 Uncertainty on the reduced pressure for the pilot, for one loop

The calculation of the uncertainty of the pilot for one loop is similar to the calculation for the participants; the only difference is the definition of the accommodation coefficient determined by the pilot laboratory which is the average value of two measurements:

$$u\left(p_{ij}\left(METAS,l,l+3\right)\right)=\widetilde{P}_j\sqrt{\left(\frac{u\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)}{\sigma_{ij}\left(METAS,l,l+3\right)}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ij}\left(METAS,l,l+3\right)\right)}{\overline{\sigma}_{ij}\left(METAS,l,l+3\right)}\right)^2} \qquad (34)$$

The combination of the type A uncertainties is given by:

$$u_A\left(p_{ij}\left(METAS,l,l+3\right)\right)=\widetilde{P}_j\sqrt{\left(\frac{u_A\left(\sigma_{ij}\left(METAS,l,l+3\right)\right)}{\sigma_{ij}}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ij}\left(METAS,l,l+3\right)\right)}{\overline{\sigma}_{ij}\left(METAS,l,l+3\right)}\right)^2} \qquad (35)$$

Finally, the uncertainty on the reference value of pressure, for a given SRG, obtained by the weighted mean value of the measurements of the 3 loops:

$$u^2\left(p_{ij}\left(METAS\right)\right)=u^2\left(P_j\right)+\frac{1}{\sum_{l=1,4,7}\frac{1}{u_A^2\left(p_{ij}\left(METAS,l,l+1\right)\right)}} \qquad (36)$$

Where the combination of type A uncertainties is:

$$u_A^2\left(p_{ij}\left(METAS\right)\right)=\frac{1}{\sum_{l=1,4,7}\frac{1}{u_A^2\left(p_{ij}\left(METAS,l,l+1\right)\right)}} \qquad (37)$$

7.6 Uncertainty on the mean value of a participant

The uncertainty of the mean pressure of a given NMI for a given step of the comparison is given by:

$$u^2\left(p_j\right)=u^2\left(P_j\right)+\frac{1}{\sum_{i=1}^{i=2}\frac{1}{u_A^2\left(p_{ij}\right)}} \tag{38}$$

7.7 Uncertainty on the mean value of the pilot

The uncertainty of the weighted mean value of the reference pressure obtained with the two SRGs is given by:

$$u^2\left(p_j\left(METAS\right)\right)=u^2\left(P_j\right)+\frac{1}{\sum_{i=1}^{i=2}\frac{1}{u_A^2\left(p_{ij}\left(METAS\right)\right)}} \tag{39}$$

7.8 Uncertainty on the reference value of the comparison

The uncertainty of the reference value of the comparison obtained with Eq.13 is given by Cox [15]:

$$u^2\left(p_j\left(CCM.P-K15\right)\right)=\frac{1}{\sum_l\frac{1}{u^2\left(p(l)\right)}} \tag{40}$$

The uncertainty on the normalized reference value for a target pressure, as given by Eq. 15, is similar to Eq. 40 as the coefficient given by Eq. 14 is close to 1.

7.9 Uncertainty on the relative deviation

The uncertainty on the relative deviation calculated by Eq. 16 is given by Cox [15] and is as follow for the laboratories participating to the definition of the reference value:

$$U\left(d_j(l)\right)=2\sqrt{\left(\frac{u\left(p_j\right)}{p_j}\right)^2-\left(\frac{u\left(p_j\left(CCM.P-K15\right)\right)}{p_j\left(CCM.P-K15\right)}\right)^2} \tag{41}$$

8 Reduction to a reference value

The determination of a key comparison reference value has been made on the basis of all the laboratories who took part to the comparison.

The consistency check defined by Cox [15] using the chi squared function has been applied to all the laboratories contributing to the reference value and it has been fulfilled with success as shown in table 11.

Table 11: Chi2 observed, Chi2 maximal permissible and number of contributors.

Pressure	**Chi2 observed**	**Chi2 maximum**	**Number of contributors**
1.0×10^{-4} Pa	0.48	11.07	5
3.0×10^{-4} Pa	1.21	11.07	5
9.0×10^{-4} Pa	1.62	12.59	6
3.0×10^{-3} Pa	1.67	12.59	6
9.0×10^{-3} Pa	1.40	12.59	6
3.0×10^{-2} Pa	1.80	12.59	6
9.0×10^{-2} Pa	3.31	12.59	6
3.0×10^{-1} Pa	2.23	11.07	5
1.0 Pa	3.21	11.07	5

The reference pressure as given by Eq. 8 and Eq. 12 for the participant and the pilot respectively and is normalized by using Eq. 14. The associated uncertainties as given by Eq. 38 and Eq. 39 are presented on table 12.

Table 12: Normalized value of the participants as given by Eq. 8 and Eq. 12 corrected by the Eq. 14. The associated uncertainty is given by Eq. 40 for the reference value, by Eq. 38 for the participants and by Eq. 39 for the pilot.

P_j (Pa)		**Reference**	**NMIJ**	**KRISS**	**CENAM**	**NIST**	**INRIM**	**PTB**	**METAS**
1.0×10^{-4}	p_1	1.0000×10^{-4}	1.0090×10^{-4}	-	9.9066×10^{-5}	9.9983×10^{-5}	9.9961×10^{-5}	9.9353×10^{-5}	1.0020×10^{-4}
	$u(p_1)$	5.95×10^{-7}	2.04×10^{-6}	-	3.75×10^{-6}	1.16×10^{-6}	1.56×10^{-6}	1.47×10^{-6}	1.05×10^{-6}
3.0×10^{-4}	p_2	3.0000×10^{-4}	3.0195×10^{-4}	-	3.0161×10^{-4}	2.9943×10^{-4}	3.0018×10^{-4}	2.9901×10^{-4}	3.0034×10^{-4}
	$u(p_2)$	8.08×10^{-7}	2.62×10^{-6}	-	3.63×10^{-6}	1.32×10^{-6}	2.91×10^{-6}	2.09×10^{-6}	1.60×10^{-6}
9.0×10^{-4}	p_3	9.0000×10^{-4}	9.0520×10^{-4}	8.9832×10^{-4}	9.0253×10^{-4}	8.9968×10^{-4}	8.9937×10^{-4}	8.9708×10^{-4}	8.9915×10^{-4}
	$u(p_3)$	1.60×10^{-6}	5.38×10^{-6}	9.25×10^{-6}	5.45×10^{-6}	2.24×10^{-6}	6.75×10^{-6}	4.77×10^{-6}	4.66×10^{-6}
3.0×10^{-3}	p_4	3.0000×10^{-3}	3.0195×10^{-3}	2.9963×10^{-3}	3.0005×10^{-3}	2.9989×10^{-3}	3.0001×10^{-3}	2.9913×10^{-3}	2.9991×10^{-3}
	$u(p_4)$	4.78×10^{-6}	1.70×10^{-5}	3.08×10^{-5}	1.53×10^{-5}	7.54×10^{-6}	1.86×10^{-5}	1.56×10^{-5}	9.83×10^{-6}
9.0×10^{-3}	p_5	9.0000×10^{-3}	9.0539×10^{-3}	8.9738×10^{-3}	9.0011×10^{-3}	8.9978×10^{-3}	8.9952×10^{-3}	8.9858×10^{-3}	8.9959×10^{-3}
	$u(p_5)$	1.24×10^{-5}	5.00×10^{-5}	9.00×10^{-5}	2.73×10^{-5}	1.97×10^{-5}	4.80×10^{-5}	4.45×10^{-5}	2.95×10^{-5}
3.0×10^{-2}	p_6	3.0000×10^{-2}	3.0181×10^{-2}	2.9815×10^{-2}	2.9987×10^{-2}	2.9994×10^{-2}	2.9989×10^{-2}	2.9940×10^{-2}	3.0010×10^{-2}
	$u(p_6)$	4.05×10^{-5}	1.61×10^{-4}	3.18×10^{-4}	8.33×10^{-5}	6.61×10^{-5}	1.57×10^{-4}	1.53×10^{-4}	9.79×10^{-5}
9.0×10^{-2}	p_7	9.0000×10^{-2}	9.0582×10^{-2}	8.8817×10^{-2}	8.9995×10^{-2}	9.0054×10^{-2}	8.9977×10^{-2}	8.9768×10^{-2}	9.0028×10^{-2}
	$u(p_7)$	1.16×10^{-4}	4.96×10^{-4}	1.06×10^{-3}	2.55×10^{-4}	2.06×10^{-4}	3.57×10^{-4}	2.96×10^{-4}	2.97×10^{-4}
3.0×10^{-1}	p_8	3.0000×10^{-1}	3.0160×10^{-1}	-	2.9967×10^{-1}	3.0029×10^{-1}	2.9976×10^{-1}	2.9920×10^{-1}	3.0028×10^{-1}
	$u(p_8)$	3.59×10^{-4}	1.66×10^{-3}	-	8.26×10^{-4}	7.13×10^{-4}	1.07×10^{-3}	8.96×10^{-4}	7.36×10^{-4}
1.0	p_9	1.0000	1.0046	-	1.0002	1.0013	0.9968	0.9964	1.0001
	$u(p_9)$	1.07×10^{-3}	5.38 x 10-3	-	2.33×10^{-3}	1.89×10^{-3}	3.90×10^{-3}	3.13×10^{-3}	2.21×10^{-3}

Table 13: Relative deviation respective to the reference value of the comparison as defined by Eq. 16 and relative uncertainty as defined by Eq. 41. The last line is the mean value of the relative deviation expressed in absolute number and depicts some kind of average agreement of the participant with the reference value.

P_j (Pa)		NMIJ	KRISS	CENAM	NIST	INRIM	PTB	METAS
1.0 x 10^{-4}	d_1	0.0090	-	-0.0093	-0.0002	-0.0004	-0.0065	0.0020
	$U(d_1)$	0.0388	-	0.0768	0.0200	0.0288	0.0271	0.0173
3.0 x 10^{-4}	d_2	0.0065	-	0.0054	-0.0019	0.0006	-0.0033	0.0011
	$U(d_2)$	0.0165	-	0.0247	0.0070	0.0187	0.0129	0.0092
9.0 x 10^{-4}	d_3	0.0058	-0.0019	0.0028	-0.0004	-0.0007	-0.0032	-0.0009
	$U(d_3)$	0.0113	0.0203	0.0126	0.0035	0.0146	0.0100	0.0097
3.0 x 10^{-3}	d_4	0.0065	-0.0012	0.0002	-0.0004	0.0000	-0.0029	-0.0003
	$U(d_4)$	0.0108	0.0203	0.0107	0.0039	0.0120	0.0099	0.0057
9.0 x 10^{-3}	d_5	0.0060	-0.0029	0.0001	-0.0002	-0.0005	-0.0016	-0.0005
	$U(d_5)$	0.0107	0.0199	0.0054	0.0034	0.0103	0.0095	0.0059
3.0 x 10^{-2}	d_6	0.0060	-0.0062	-0.0004	-0.0002	-0.0004	-0.0020	0.0003
	$U(d_6)$	0.0104	0.0212	0.0049	0.0035	0.0101	0.0098	0.0059
9.0 x 10^{-2}	d_7	0.0065	-0.0131	-0.0001	0.0006	-0.0003	-0.0026	0.0003
	$U(d_7)$	0.0107	0.0238	0.0050	0.0038	0.0075	0.0061	0.0061
3.0 x 10^{-1}	d_8	0.0053	-	-0.0011	0.0010	-0.0008	-0.0027	0.0009
	$Ud_8)$	0.0108	-	0.0050	0.0041	0.0067	0.0055	0.0043
1.0	d_9	0.0046	-	0.0002	0.0013	-0.0032	-0.0036	0.0001
	$U(d_9)$	0.0105	-	0.0041	0.0031	0.0075	0.0059	0.0039
$\frac{1}{N}\sum_1^N \lvert d_i \rvert$		0.0061	0.0062	0.0051	0.0022	0.0007	0.0008	0.0032

8.1 Difference and uncertainty respective to the reference value.

The relative differences to the reference value as given by Eq. 16 and the associated uncertainty are summarized in table 13. A more visual presentation of the relative deviation is presented on Fig. 7.

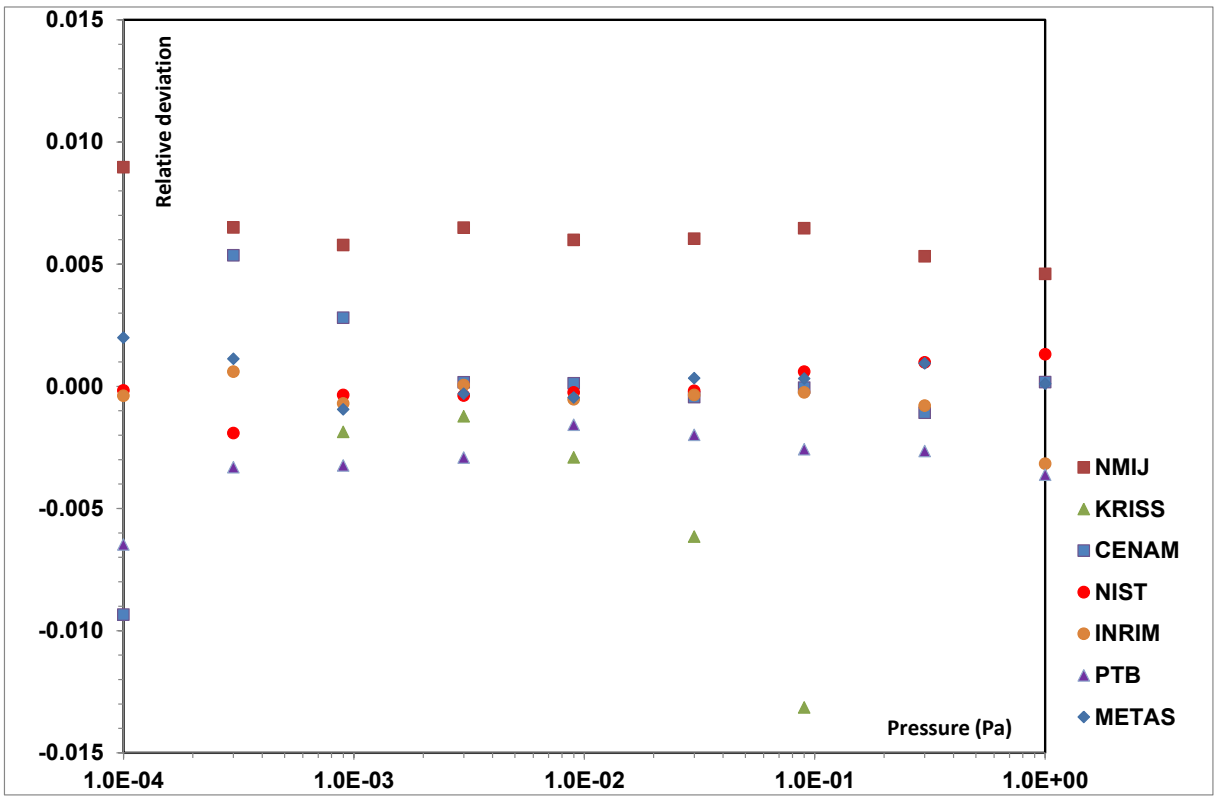

Fig. 7: Relative deviation respective to the reference pressure for all the participants.

8.2 Degree of equivalence.

In order to understand the importance of the deviation relative to the uncertainty it is necessary to determine the ratio between the deviation and the uncertainty. This is done using the Eq. 17 for the determination of the degree of equivalence. The values for all the participants are presented in Table 14. The relative deviations respective to the reference value, and the associated uncertainty, are presented in Fig. 8. At this point it is important to note that all the participants have an equivalent definition of the pressure as the uncertainty bar always crosses the horizontal axis of the graph. The offset as well as the error bars are a combination of the uncertainty of the participating laboratory and the stability of the transfer standard.

Table 14: Ratio between offset and uncertainty as calculated by Eq. 17. All the participants agree with the criterion of equivalence.

P_j (Pa)	**NMIJ**	**KRISS**	**CENAM**	**NIST**	**INRIM**	**PTB**	**METAS**
1.0×10^{-4}	0.23	-	-0.12	-0.01	-0.01	-0.24	0.11
3.0×10^{-4}	0.39	-	0.22	-0.27	0.03	-0.26	0.12
9.0×10^{-4}	0.51	-0.09	0.22	-0.10	-0.05	-0.32	-0.10
3.0×10^{-3}	0.60	-0.06	0.02	-0.10	0.00	-0.29	-0.05
9.0×10^{-3}	0.56	-0.15	0.02	-0.07	-0.05	-0.17	-0.08
3.0×10^{-2}	0.58	-0.29	-0.09	-0.05	-0.04	-0.20	0.06
9.0×10^{-2}	0.61	-0.55	-0.01	0.16	-0.03	-0.42	0.05
3.0×10^{-1}	0.49	-	-0.22	0.24	-0.12	-0.48	0.22
1.0	0.44	-	0.04	0.42	-0.42	-0.61	0.03

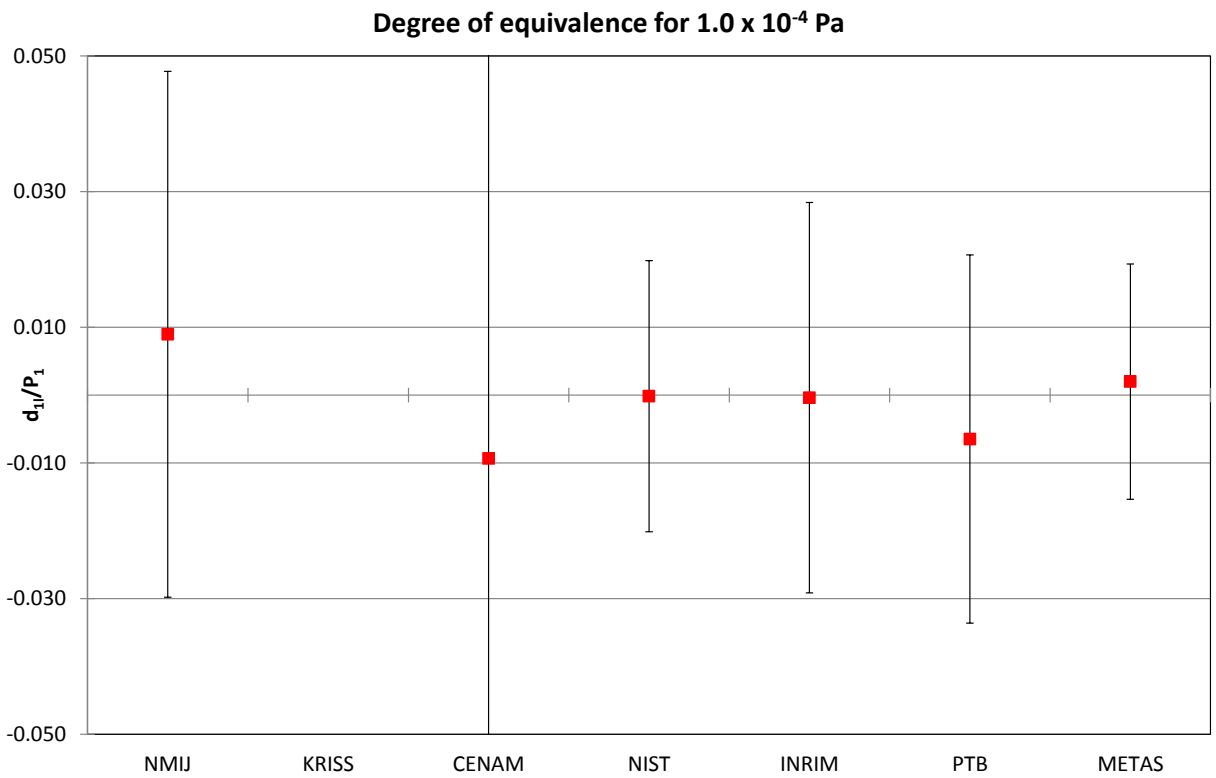

Fig. 8a: Relative degree of equivalence for the target pressure 1.0×10^{-4} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

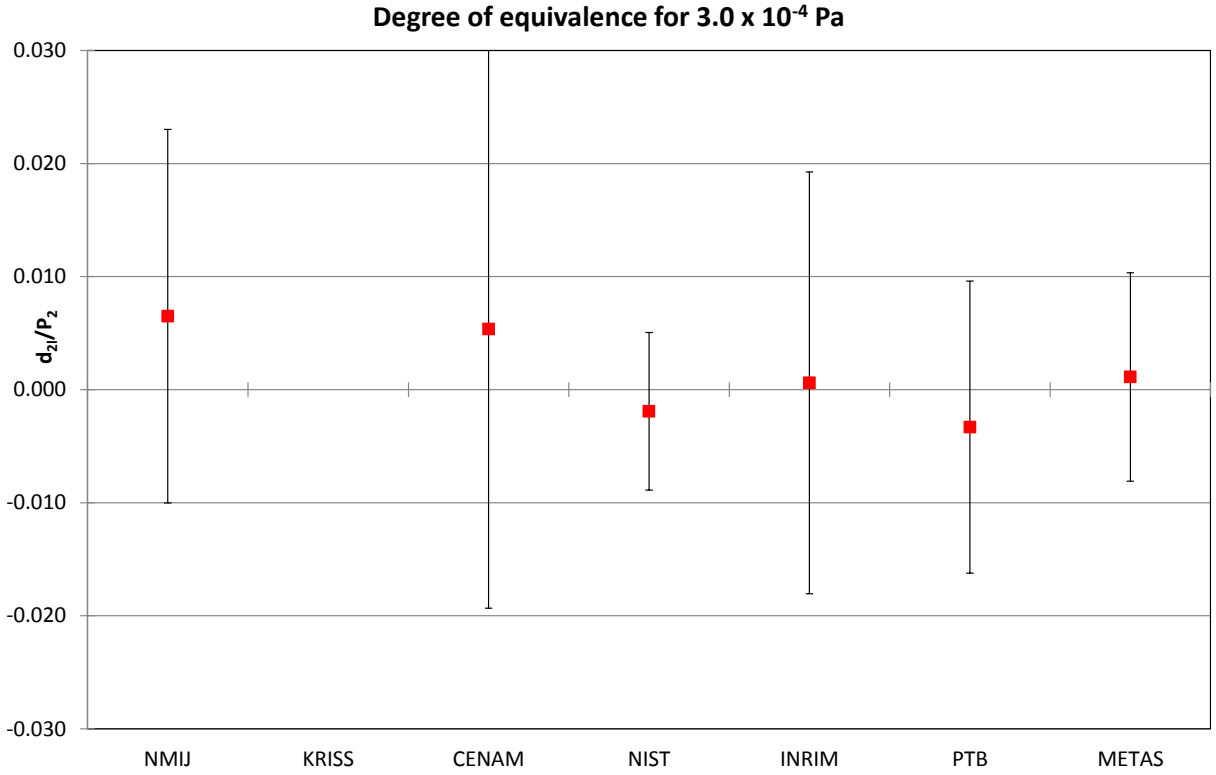

Fig. 8b: Relative degree of equivalence for the target pressure 3.0 x 10^{-4} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

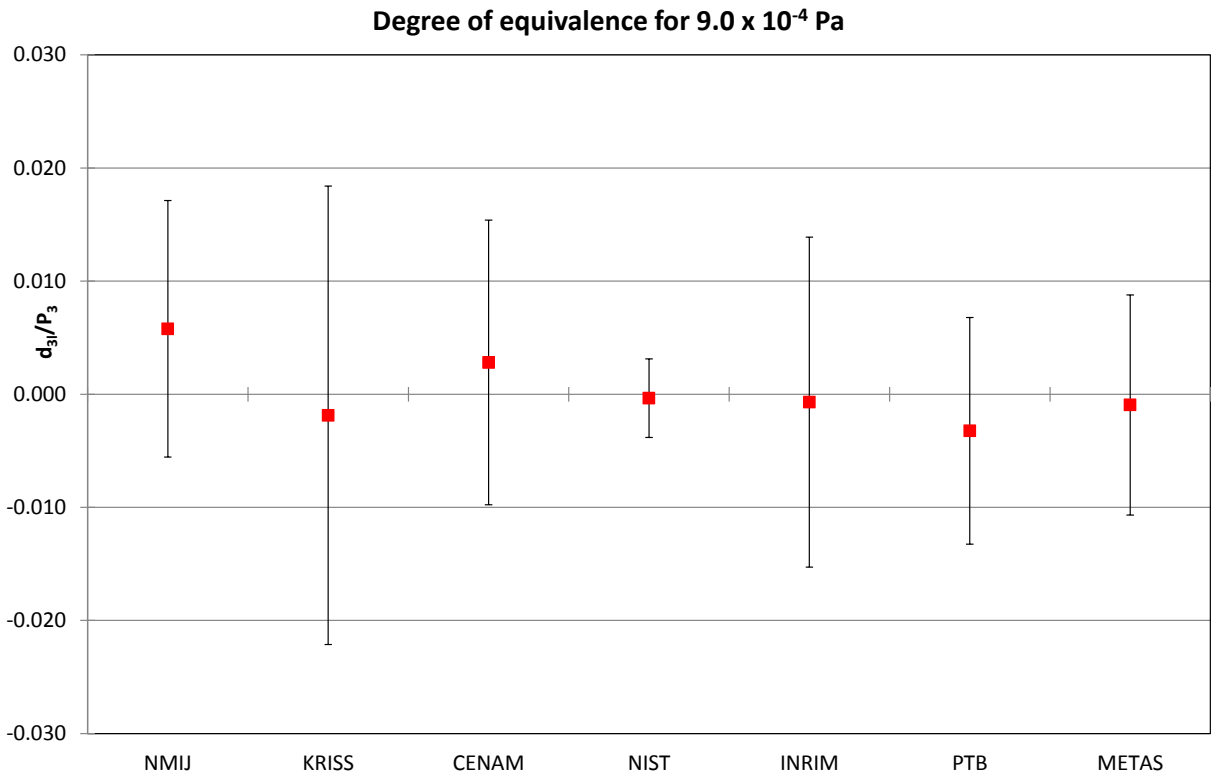

Fig. 8c: Relative degree of equivalence for the target pressure 9.0 x 10^{-4} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

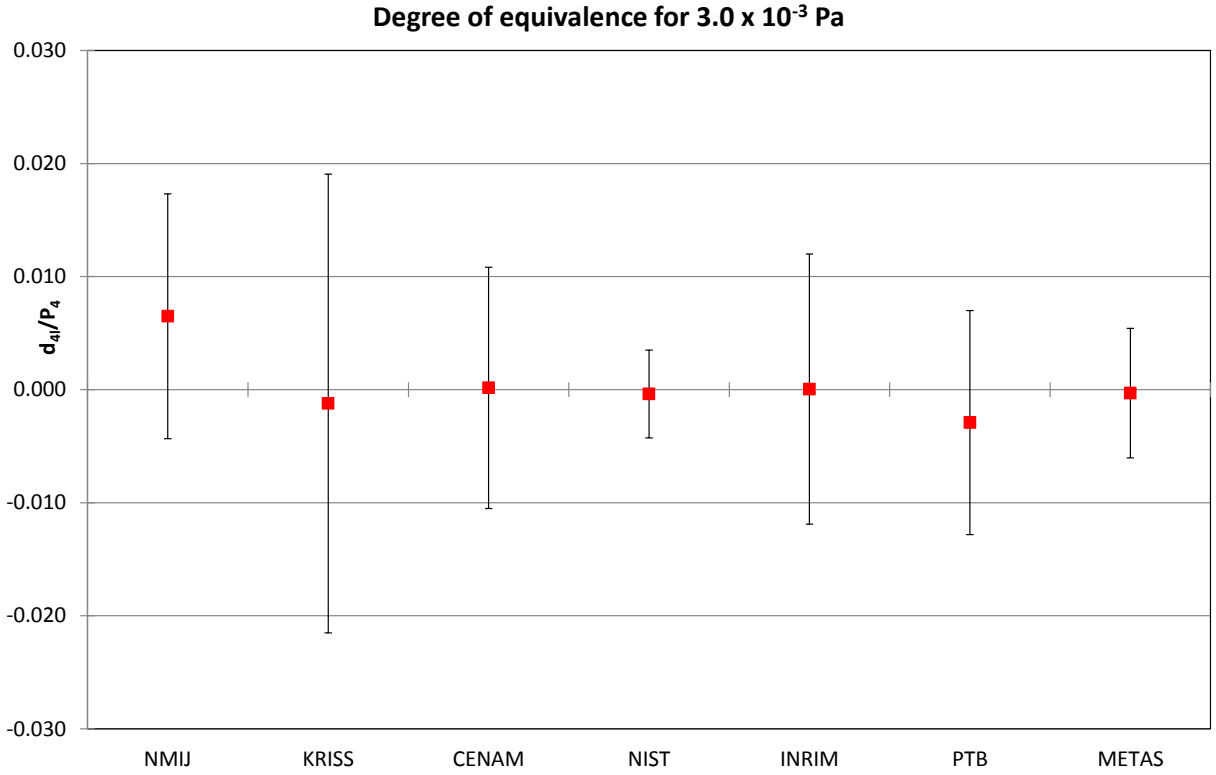

Fig. 8d: Relative degree of equivalence for the target pressure 3.0 x 10^{-3} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

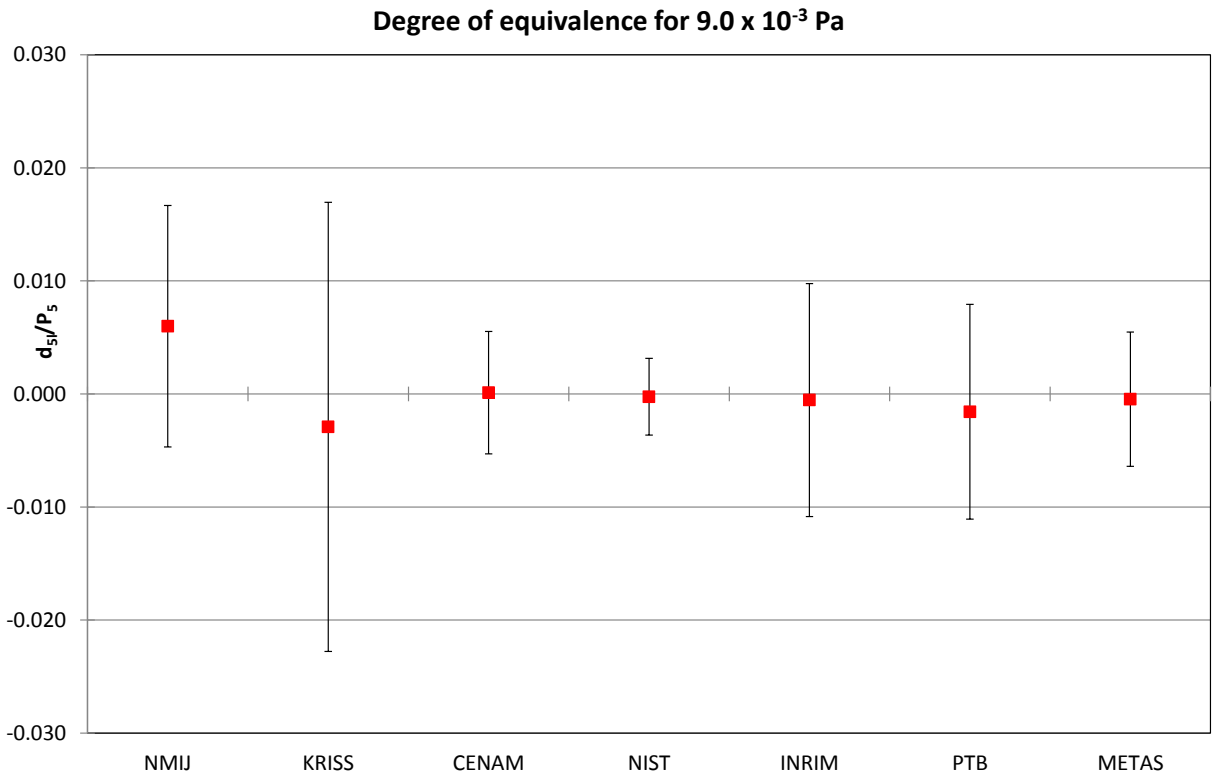

Fig. 8e: Relative degree of equivalence for the target pressure 9.0 x 10^{-3} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

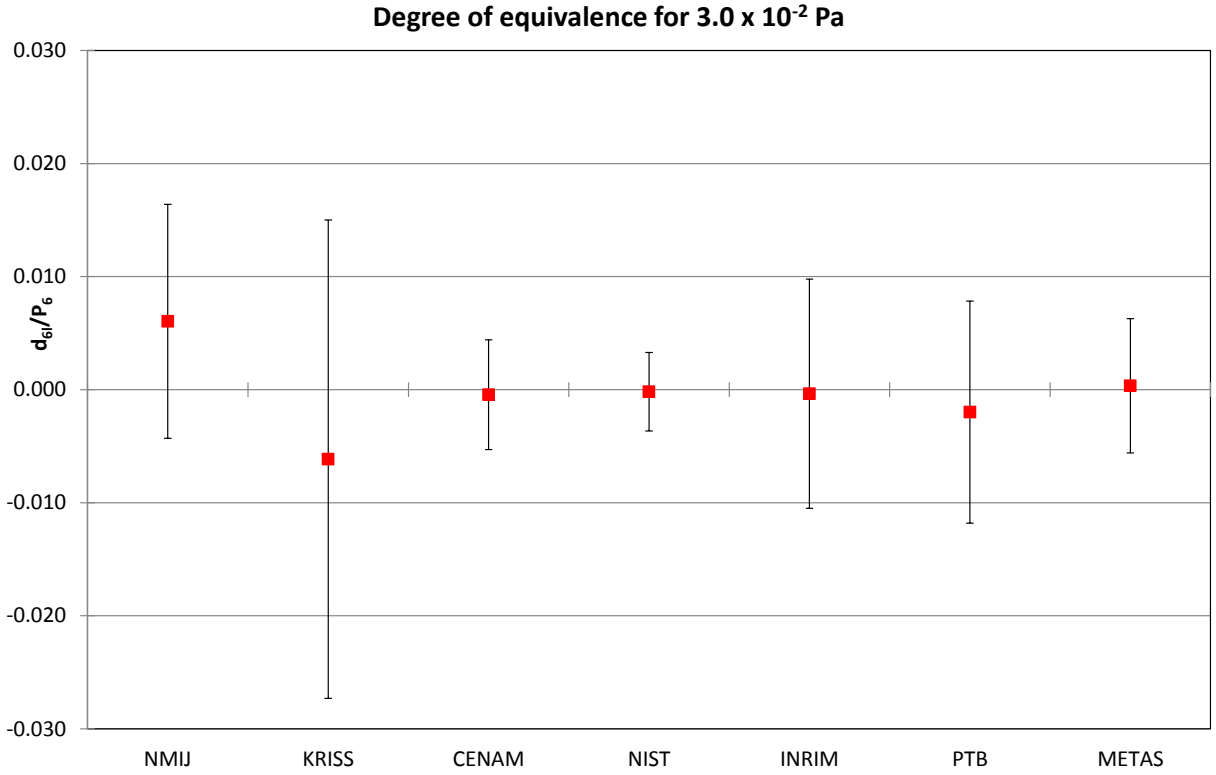

Fig. 8f: Relative degree of equivalence for the target pressure 3.0 x 10^{-2} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

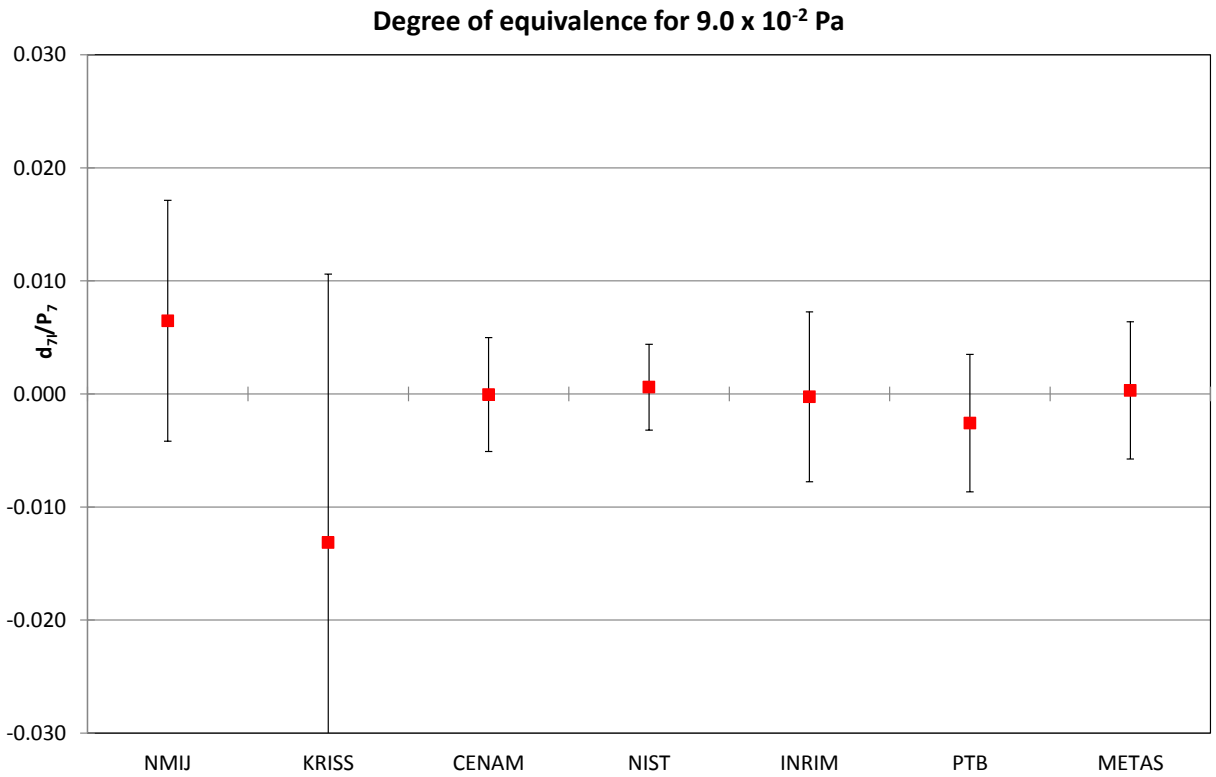

Fig. 8g: Relative degree of equivalence for the target pressure 9.0 x 10^{-2} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

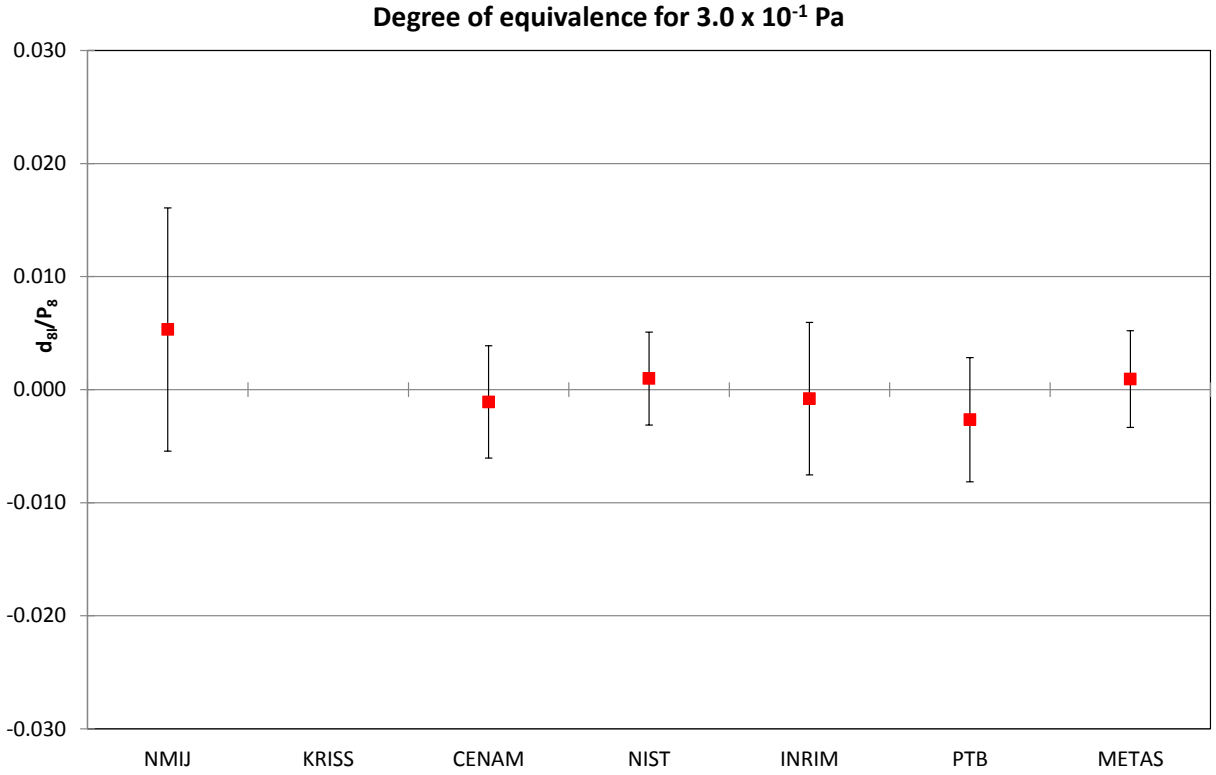

Fig. 8h: Relative degree of equivalence for the target pressure 3.0 x 10^{-1} Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

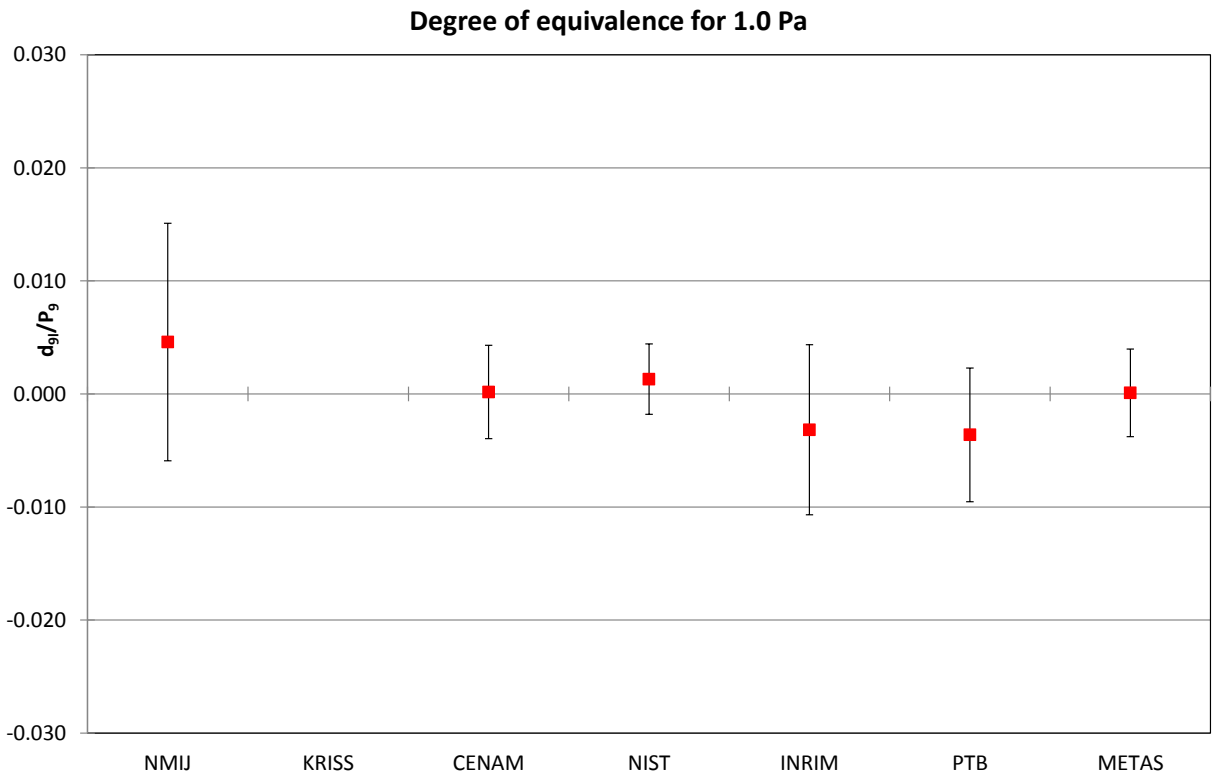

Fig. 8i: Relative degree of equivalence for the target pressure 1.0 Pa. Note that the error bars and the offset are affected by the instability of transfer standard SRGs.

9 Conclusion

The comparison CCM.P-K15 was a key opportunity, 20 years after the comparison CCM.P-K9, to assess the quality of the definition of the pressure scale from 0.1 mPa to 1.0 Pa. The participants came from three different Regional Metrology Organizations (RMO) and each RMO had at least two participants.

The transfer standard, two spinning rotating gauges (SRG) and the readout electronics were circulated from March 2010 until March 2011. One of the SRG exhibited a poor stability; the other one had a good stability while circulated in Europe and America and a fair stability while circulated in Asia.

The reference value has been determined using a weighted mean of the participant's results. The consistency check has demonstrated the validity of this approach. The equivalence of the definition of the pressure has been demonstrated for all the participants and for all the pressure steps. The pair-wise degree of equivalence has been calculated and the conditions of equivalence are fulfilled for all the participants.

This work has demonstrated that the stability of the transfer standard is a key issue in a comparison. Furthemore, the circulation of two transfer standards contributed to the success of this comparison. It is clear that further study for the transportation stability of SRGs is required for the future comparisons among NMIs.

10 References

[1] K. H. Chung, S. S. Hong, Y. H. Shin, J. Y. Lim, S. K. Lee and S. Y. Woo, “Hot cathode ionization gauge calibration with the KRISS ultra-high vacuum standards”, Metrologia, **36** (1999) ,675-679.

[2] Tilford, C.R., Dittmann, S., and McCulloh, K.E., “The National Bureau of Standards high-vacuum standard”, J. Vac. Sci. Technol. **6** (1988), 2853-9.

[3] McCulloh, K.E., Tilford, C.R., Ehrlich, C.D., and Long, F.G “Low-range flowmeter for use with vacuum and leak standards”, J. Vac. Sci. Technol. **5** (1987), 376-81.

[4] *NIST Internal Report of Calibration NC226A IG2L*, prepared by J. Hendricks, NIST, Gaithersburg (2009).

[5] Bergoglio M, Mari D., Vacuum **84** (2009); 270.

[6] Calcatelli A, Raiteri G, Rumiano G. Measurement **34** (2003), 2: 121.

[7] Bergoglio M, Calcatelli A. Metrologia **41** (2004), 278.

[8] W.Jitschin, J.K. Migwi, and G. Grosse, Pressures in the high and medium vacuum range generated by a series expansion standard, Vacuum **40** (1990), 293-304.

[9] W.Jitschin, J.K. Migwi, and G. Grosse, Gauge calibration in the high and medium vacuum range by a series expansion standard, Vacuum **41** (1990), 1799.

[10] K. Jousten, G. Rupschus, The uncertainties of calibration pressures at PTB, Vacuum **44** (1993), 569-572.

[11] K. Jousten, P. Röhl, V. A. Contreras, Volume ratio determination in static expansion systems by means of a spinning rotor gauge, Vacuum **52** (1999), 491-499.

[12] K. Jousten, M. Bergoglio, A. Calcatelli, J. -N. Durocher, J. Greenwood, R. Kangi, J. -C. Legras, C. Matilla and J. Setina, Final report and the regional comparison Euromet.M.P-K1.b in the pressure range from 3.0 x 10^{-4} Pa to 0.9 Pa.

[13] C. Wuethrich and M. Coulibaly, The determination of volume ratio by gas depletion through multiple expansions, Vacuum **81** (2006), 453 - 458

[14] R. Kacker and A. Jones, On use of Bayesian statistics to make the “Guide to the expression of uncertainty in measurement” consistent, Metrologia **40** (2003), 235 - 248

[15] M. G. Cox, The evaluation of key comparison data, Metrologia **39** (2002), 589 - 595

[16] Measurement comparisons in the CIPM MRA, document CIPM MRA-D-05, version 1