Final report on the supplementary comparison, Euramet.M.P-S7 (Euramet project 1040) in the pressure range from $1\cdot10^{-4}$ Pa to 0.9 Pa

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Abstract

Many laboratories within Euramet started a calibration service in medium and high vacuum recently and did not have the opportunity to take part to a comparison before.

In order to assess the uncertainty budget and the quality of the measurement of these laboratories, an intercomparison, Euramet 1040 registered as Euramet.M.P-S7, from 0.1 mPa to 0.9 Pa has been organised. The participants are the CMI (Czech republic), EIM (Greece), IMT (Slovenia), INRIM (Italy), IMBIH (Bosnia Herzegovinia) and MIKES (Finland) while METAS (Switzerland) is pilot laboratory. Three laboratories (INRIM, CMI and METAS) involved in this work have a primary definition of the pressure.

Two spinning rotor gauges and a control electronic are used as transfer standard. The circulation of the transfer standard is organised as a succession of loops with a measurement by the pilot between each participant.

A reference value has been determined based on a weighted mean of the results of the primary laboratories. All the participants have demonstrated their equivalence in the definition of the pressure.

This comparison has been used as pilot comparison for the CCM.P-K14 project which covers the same scope with similar transfer standards.

Content

1   Introduction

An intercomparison in the range 0.1 mPa to 1 Pa was agreed at the Euramet meeting in March 2007 in Teddington. The comparison had been proposed by METAS (Switzerland) and it immediately raised interest from MIKES (Finland), EIM (Greece), IMT (Slovenia). The comparison was joined before the circulation of the transfer standard by IMBiH (Bosnia Herzegovina) and INRIM (Italy).

The comparison was registered as project number 1040 by EURAMET and as EURAMET.M.P-S7 by the BIPM.

Previous work in that range of pressure includes the CCM.P-K9 that lasted from 1981 to 1987. The comparison EUROMET.M.P-K-1.b (EURAMET project 442) [1] took place from 2000 to 2002. The participants had all primary pressure standards and it was linked to a key comparison only at 0.9 Pa. Some of the participants had to withdraw at least part of the results due to anomalies. A new comparison in that field was motivated by NMI's working with transfer standards as well as by NMI's which encountered problems in the EUROMET.M.P-K-1 project.

2   Participating laboratories.

Seven laboratories, including the pilot, took part to the comparison. The equipment used by the participants cover all the possibilities available for the definition of the pressure scale of the comparison. Some of the participants had already CMC listed for the domain of the comparison while other participants wanted to claim new CMC based on the result of this work. The following table gives a short form of the situation of the participants at the time of the comparison.

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

Laboratory	Standard	Definition	Traceability	CMC
CMI	Continuous expansion system	Primary	Independent	YES
EIM	Spinning rotor gauge	Secondary	PTB	NO
IMT	Spinning rotor gauge	Secondary	PTB	YES
INRIM	Continuous and static expansion system	Primary	Independent	YES
IMBiH	Spinning rotor gauge	Secondary	PTB	NO
MIKES	Spinning rotor gauge	Secondary	PTB	YES
METAS	Static expansion system	Primary	Independent	NO

2.1   Primary pressure laboratories

2.1.1 INRIM

INRIM has the traceability through two primary pressure standards.
The continuous expansion system (CES) between $1 \cdot 10^{-4}$ Pa and $9 \cdot 10^{-2}$ Pa.
The static expansion system (SES) between $9 \cdot 10^{-2}$ Pa and $9 \cdot 10^{-1}$ Pa.
In the measurements performed by CES, the residual drag was determined by the first option of the protocol, using a polynomial approximation versus the frequency in all the range of the rotation speed.
In the measurements carried out by SES, the residual drag was determined during the measurements, by the second option of the protocol and the frequency was not recorded as the data were recorded by internal printing of control unit.

2.1.1.1 Continuous expansion system

The INRIM continuous expansion system [2] works in the range between $1 \cdot 10^{-6}$ Pa and $9 \cdot 10^{-2}$ Pa. It is mainly composed by a primary flowmeter [3], 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 standard pressure p, generated in the calibration chamber, is given by [1]:

$$p = \frac{q}{S_{eff}} \cdot F_T = \frac{q}{\dfrac{C}{1 + \dfrac{C}{S_p}}} \cdot \frac{T_V}{T_q} \cdot \sqrt{\frac{T_{ref}}{T_V}}$$

[1]

where q is the throughput generated and measured by the flowmeter, S_{eff} is the effective pumping speed, F_T is a correction factor for temperature, S_p is pumping speed. T_V is the mean temperature of the calibration chamber V_1, T_q the mean temperature of the flowmeter and T_{ref} the reference temperature at which the conductance was calculated.

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

The combined standard uncertainty $u(p)$ of the standard pressure generated by the continuous expansion system is:

2.1 10^{-8} Pa $\leq u(p) \leq$ 9.4 10^{-7} Pa, $1 \cdot 10^{-6}$ Pa $\leq p \leq 1 \cdot 10^{-4}$ Pa
9.4 10^{-7} Pa $< u(p) \leq$ 3.6 $\cdot 10^{-4}$ Pa, $1 \cdot 10^{-4}$ Pa $< p \leq 9 \cdot 10^{-2}$ Pa

2.1.1.2 Static expansion system

The INRIM static expansion system works in the pressure range between 0,1 Pa and 1000 Pa [4]. It is formed by three volumes of about 0.05 m^3 (V1), 5×10^{-4} m^3 (V3) and 1×10^{-5} m^3 (V2), 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.

The static expansion system is based on the ideal gas law in isothermal conditions:

$$p = p_0 \frac{V_2}{V_2 + V1} = p_0 \frac{1}{R}$$

[2]

where p_0 is the inlet pressure of gas in V_2, p is the pressure of gas after the expansion of gas in V_1 and R is the expansion ratio between volumes $(V_2 + V_1)$ and V_2, which is periodically determined.

In real conditions the gas expansion is not an isothermal process, so a temperature factor T_V/T_v has to be introduced:

$$p = p_0 \frac{1}{R} \frac{T_V}{T_v}$$

[3]

where T_V and T_v are respectively the temperature of gas in V_1 and V_2.

The combined standard uncertainty $u(p)$ of the standard pressure generated by the static expansion system is:

$u(p)$ / Pa $= 5.0 \times 10^{-5} + 8.5 \times 10^{-4} \, p$, 0.09 Pa $\leq p \leq 10$ Pa

$u(p)$ / Pa = 8.5 x 10^{-3} + 4.1 x 10^{-4} p,   10 Pa < $p \leq$ 1000 Pa

2.1.2 CMI

The comparisons of the two SRG rotors were performed simultaneously. The enclosed unit and head were used for one rotor, our own unit and head were used for the other one. During the second day the heads and units were exchanged. There were observed noticeable shifts of the rotors offsets due to this exchange, but the accommodation coefficients remained unchanged.

The care of the rotors was made according to the enclosed instructions. The rotors were not exposed to the atmosphere and there was a UPS unit used for the SRG2-CE/0 units power supplying.

2.1.2.1 Measurement

The actual SRG offset at the actual conditions was measured every time immediately after the measuring of the response for the measured pressure.

The residual drag was measured at the average rotor frequency ω_{RD}. This frequency differed from the average frequency ω_{DCR} during the measurement of the response to the pressure. So the residual drags presented in the table of the results represent the estimations of the residual drags at the frequency ω_{DCR}:

Deceleration(0) = $RD_{MEASURED}$ + $A_{ROT} \cdot (\omega_{DCR} - \omega_{RD})$

The RD(ω) dependences were studied and the following estimations of "A" were used for the RD correction as described above:

A_{43} = 1,07 · 10^{-9}
A_{72} = 5,23 · 10^{-10}

The temperatures of the rotors was measured with the contact Pt1000 thermometers.

2.1.2.2 The reference standard

The pressure was generated by the dynamic expansion system (orifice flow standard). The admitted flow was measured by two constant pressure flowmeters designed A and B.

The overall expanded uncertainty of the generated pressure depends on the flowmeter used and the method of the orifice conductivity determination:

(1·10^{-4} ÷ 1·10^{-3}) Pa   flowmeter B (small), molecular orifice conductivity
                                      1,7 % of the measured value
(1·10^{-3} ÷ 1·10^{-2}) Pa   flowmeter A (big), molecular orifice conductivity
                                        1,6 % of the measured value
(1·10^{-2} ÷ 9·10^{-1}) Pa   flowmeter A (big), transitional orifice conductivity
                                        1,8 % of the measured value

These expanded uncertainties (the uncertainty of the actual pressure and the uncertainty of the mean value of the deceleration) were stated in the table of results.

2.1.3 METAS

2.1.3.1 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. The last expansion stage has two expand scheme with expansion ratio of either 50 or 200. The maximum pressure reduction ratio is then 2·10^{-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. [5].

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

2.1.3.2 Measurements.

The measurements have been 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 measurements 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 system 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.

2.2 Secondary level laboratories

2.2.1 IMBIH

Measurements were performed on a comparison calibration system by direct comparison with a reference SRG, which is traceable to PTB.

The vacuum system is made of stainless steel components with Con-Flat connection flanges. Volume of calibration chamber is 10 L. System is pumped with a turbomolecular pump and a base pressure of the chamber is below 5×10^{-7} Pa. The system is equipped with a quadrupole mass spectrometer for leak detection and analysis of residual gas composition.

Calibration pressure is established by dynamic equilibrium of a flow of incoming gas (99.999 % pure nitrogen) and effective pumping speed of the pump for pressures below $3 \cdot 10^{-2}$ Pa. Above this pressure a static mode is used (pump is closed and the chamber is filled to the required pressure).

The uncertainty of reference pressure is estimated to:

$$U(p) = \sqrt{(0.011 \times p)^2 + (2 \times 10^{-6}\, Pa)^2}$$

[4]

The main components included in this uncertainty are:
- (i)   uncertainty of calibration at PTB
- (ii)   estimated 1 year stability of accommodation coefficient
- (iii)   uncertainty of temperature of calibration gas
- (iv)   short term stability of offset correction, and
- (v)   type A uncertainty of SRG readings.

2.2.2 MIKES

MIKES has a traceability to PTB using a SRG as transfer standard.

The equipment used by MIKES in this comparison was the spinning rotor gauge SRG-2CE, which is traceable to PTB. Display no. 500082G, finger no. 191493, head no. 94097G, certificate 2000 PTB 09 (during the comparison), certificate 70134 PTB 10 (the newest one). For the travelling standards we used our display SRG-2 no. 20750G and head no. 92167G.

In the 0,1 Pa range every measurement is a single reading (30 single readings for every pressure level). In the lower range every reading is an average of 10 readings (3 x 10 = 30). I wanted to get the same number (30) of readings for every point. But it was not possible to use 10 reading averaging on the range 0,1 Pa because then the ball was accelerated in the middle of the measuring process. I did not want it to accelerate then. And this is the reason for 30 single readings.

2.2.2.1 Estimation of measurement uncertainty

Then estimation of the measurement uncertainty was done according to the principles of GUM. The mathematical model used for the calculation is the spinning rotor gauge pressure equation, which is multiplied with the correction factor based to the calibration certificate. In addition, long time stability of the accommodation factor, uncertainty due to incomplete homogeneity of the vacuum chamber, resolution of the reference SRG and resolution of the transfer standard SRG were added.

$$p = \left[\frac{1}{\sigma} \cdot \frac{\pi}{10} \cdot a \cdot \rho \cdot \sqrt{\frac{(8 \cdot R \cdot T)}{(\pi \cdot M)} \cdot \frac{d\omega}{d\omega t} - offset} \, \right] \cdot f(p_{ind}) + \delta_{accstab} + \delta_{chamber} + \delta_{resol} + \delta_{resol,DUT}$$

MIKES wanted to lower uncertainty values for the range 0.003 Pa to 0.9 Pa. For the lower range, 0.0005 Pa to 0.003 Pa, uncertainty values reported in this comparison are the same as in the present CMC-tables.

Uncertainty of the accommodation factor (σ) is from the reference SRG calibration certificate. Uncertainty of the sphere radius (*a*), sphere density (ρ) and *M* are from literature. Uncertainty for *R* is from NIST Codata.

Uncertainty of temperature (*T*) was estimated by measuring it in the different locations around the vacuum chamber and compared the readings at the ones observed at the measuring head. The biggest difference was taken to calculation with rectangular distribution.

Uncertainty for deceleration ($d\omega/d\omega t$) is a typical observed standard deviation of 10 readings for each pressure point and uncertainty for *offset* is the observed standard deviation in the measurements defining the value of offset.

The correction factor, *f(p_{ind})*, is used for pressures higher than 0,01 Pa. Otherwise the value of the factor is 1. Uncertainty of the correction factor is from the calibration certificate. The accommodation factor long time stability ($\delta_{accstab}$) is based to the results from calibration certificates from last four years.

The biggest uncertainty component is the incomplete homogeneity of the vacuum chamber ($\delta_{chamber}$). The value was investigated by measuring in the different locations and in the different pressure levels. Value of this component is pressure dependent and it varies level 10^{-5} Pa to 10^{-4} Pa, rectangular distribution.

2.2.3 IMT

Calibrations were performed on the newly developed static expansion/comparison calibration system (SEC1) at IMT. This system is mainly intended for calibrations by direct comparison with reference vacuum gauges (SRG and CDG) in the range from 1×10^{-5} Pa to 100 kPa. Main calibration chamber has a cylindrical shape with a diameter of 200 mm and height of 300 mm. Volume of the chamber is approximately 10 L. It has six CF35 connection ports for vacuum gauges, located in one plane in the middle of the chamber. Additional small volume is connected to the calibration chamber, which enables "in-situ" calibration of reference gauges by primary static expansion method. Volume ratio is approximately 200. Initial pressure for expansion is measured with 100 kPa FS Quartz Bourdon Gauge which is traceable to a pressure balance RUSKA 2465A (Slovenian national standard), which is further traceable to PTB. Estimated standard uncertainty (k=1) of the generated pressure by static expansion is 0.17% in the range from 500 Pa down to 2 Pa (single expansion) and 0.3% from 2 Pa down to 0.01 Pa (double expansion).

The calibration method in this intercomparison was direct comparison of transfer standard SRGs with our reference SRG, which has been calibrated "in-situ" by static expansion method just before the intercomparison. Calibration range of the reference SRG was from 0.05 Pa to 1 Pa and the measured values of accommodation coefficient were modelled with a quadratic function. The accommodation coefficient of the reference SRG at any pressure in the interval from 1×10^{-5} Pa to 1 Pa can be estimated from the model.

The two transfer standard SRGs were calibrated at the same time. The calibration pressure for direct comparison was established by dynamic equilibrium for pressures below 0.09 Pa, and statically at 0.09 Pa and above. Pressure points from 1×10^{-4} Pa to 9×10^{-4} Pa were measured with a sampling interval 60 s, from 3×10^{-3} Pa to 9×10^{-2} with sampling interval 30 s and from 0.3 Pa to 0.9 Pa with sampling interval 10 s.

Residual drag was measured with the sampling interval 60 s for pressure points up to 0.03 Pa and with sampling interval 30 s above this pressure. At pressure points below 0.03 Pa the residual drag was re-measured at least before and after each series of three data points at given pressure. A mean of these two values was taken for "offset" correction. At pressure 0.03 Pa and above the uncertainty of residual drag becomes negligible, so it was measured only before 0.03 Pa measurement point and then checked after the highest point of 0.9 Pa.

Rotor frequency was kept in the interval from 405 Hz to 425 Hz.

Each measured SRG value (residual drag or pressure point) in our results table is a mean value of 10 successive readings. Reported uncertainties of deceleration rate are given as standard deviation of the mean value (type A).

The SRG controller for the Rotor G191872 was changed after the first day measurements. The suspension head remained the same and was not removed. There was negligible change in offset, but the effect of changing the controller is clearly seen in our results as a 0.1% change in accommodation coefficient (for the other rotor results are essentially the same for both days).

Since the measurements were performed as direct comparison of SRGs, the same value of temperature was entered into all controllers. The estimated uncertainty due to temperature difference of the reference SRG and transfer gauges is u(T)= 0.2 K (k=1).

All the uncertainties in the results table are given as standard uncertainties (k=1).

2.2.4 EIM

Measurements were carried out through comparison to a reference SRG. Both standards were mounted to a vacuum chamber. The details of the equipment used are provided below:

2.2.4.1 Chamber
Type: CS7 vacuum calibration system
Manufacturer: Leybold
Volume: 37,85 lt

2.2.4.2 Reference standard
Spinning Rotor Gauge (SRG)
Type: SRG-2CE/O
Electronics, Type: SRG-2CE/O-EL
Measuring Head, type: SRG-SH 700
Rotor, type: SRG-BF-CAL
      Ball diameter: 4,50 mm
      Ball density: 7,70 g/cm^3

2.3   Uncertainty of the participants on the reference pressure

The average uncertainty of the participants on the reference pressure is summarised in table 2 and is depicted in Fig.1. Two primary laboratories (INRIM and METAS) have smaller uncertainties than all the other participants. CMI has also a primary definition but has a larger uncertainty. The relative uncertainty at smaller pressure tends to increase due to degassing of the wall of the chambers, the influence of the residual drag of the SRG used as reference and the uncertainty due to multiple expansions.

Table 2: Average of the relative uncertainty on the definition of the reference pressure by the participants.

Pressure Pa	CMI	EIM	IMT	INRIM	IMBiH	MIKES	METAS
1.0E-04	0.0085	0.0177	0.0061	0.0093	0.0116		0.0100
3.0E-04	0.0085	0.0118	0.0056	0.0082	0.0064		0.0050
9.0E-04	0.0085	0.0109	0.0055	0.0066	0.0056	0.0317	0.0050
3.0E-03	0.0080	0.0108	0.0055	0.0049	0.0055	0.0200	0.0030
9.0E-03	0.0080	0.0107	0.0055	0.0039	0.0055	0.0092	0.0030
3.0E-02	0.0090	0.0108	0.0055	0.0038	0.0055	0.0080	0.0030
9.0E-02	0.0090	0.0108	0.0055	0.0014	0.0055	0.0077	0.0030
3.0E-01	0.0090	0.0107	0.0055	0.0010	0.0055	0.0076	0.0020
9.0E-01	0.0090	0.0106	0.0056	0.0009	0.0055	0.0075	0.0020
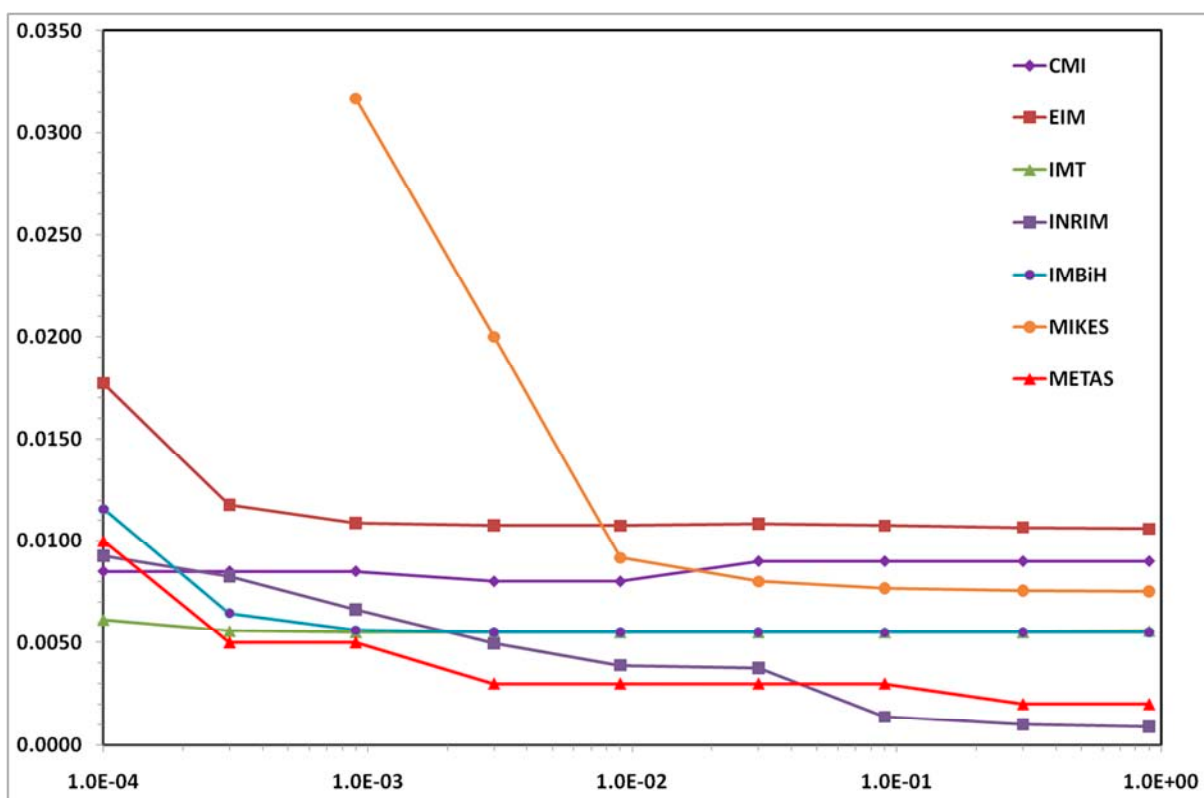
Fig 1: Average of the relative uncertainty on the definition of the reference pressure by the participants plotted versus the pressure in Pa.

3 Transfer standard

The transfer standards consist 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 have not been measured but will be used as conventional values in order to determine the accommodation coefficient.

Table 3: Characteristics of the transfer standard.

Transfer Standard	A	B
Metas Number	006411	006412
SRG Part Number (MKS)	SRG-BF	SRG-BF
SRG Serial Number	G191872	G191943
Valve part number (Varian)	SRG-BF	SRG-BF
Valve Serial Number	LVB90339	LVL70128
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	min: 402 Hz max: 450 Hz	min: 402 Hz max: 450 Hz

A stainless steel spring is mounted on the plate of the valve and will immobilise the ball once the valve is closed. The spring is far enough from the ball once the valve is open and the measurement of the residual drag of the ball has shown no spurious drag due to an electro-

magnetic coupling between the ball and the spring via the magnetic field of the ball.

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

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

A dynamometric wrench was also circulated and had to be used to close the all metal valve with the specified torque.

4   Method used for the measurements

4.1   Correction of the residual drag

Two techniques have been proposed for the determination of the residual drag of the SRG used for the correction of the deceleration measured under vacuum. A first technique is the measurement of the residual drag prior to the series of measurement at the different pressure steps. The measurement has to be repeated for several values of rotation speed to determine the correction to apply due to the rotation speed. A second techniques is the measurement of 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 measurement have been performed at the 9 following value of pressure obtained using nitrogen:

$1 \cdot 10^{-4}$, $3 \cdot 10^{-4}$, $9 \cdot 10^{-4}$, $3 \cdot 10^{-3}$, $9 \cdot 10^{-3}$, $3 \cdot 10^{-2}$, $9 \cdot 10^{-2}$, $3 \cdot 10^{-1}$, $9 \cdot 10^{-1}$ Pa

The deviation of the effective pressure from the nominal pressure had to be less than 10% from the nominal value for points lower than $4 \cdot 10^{-2}$ Pa and less than 5% of the nominal value at higher pressure.

Each laboratory had to repeat each measurement point at least three time in each calibration sequence. The calibration sequence will be repeated at least twice making a total of at least 54 measurement points.

The temperature of the system had to stay within 21 and 23 °C.

4.3   Circulation of the transfer standard

The circulation of the transfer standard was organised with measurement by the pilot laboratory between each participant to assess the stability of the transfer standard. A cycle of measurement by a participant and then the pilot is called a petal. The effective circulation has been made according to the following schedule which differs slightly from the original schedule. The petals of the comparison are numbered according to the calibration number performed by the pilot laboratory. The petal 3 has no participant due to an unexpected problem.

Table 4: Schedule of the circulation of the transfer standard.

Petal Nr.	Date	Pilot	Date	Participant
1	2008.06	METAS	2008.08	CMI
2	2008.09	METAS	2008.10	EIM
3	2008.11	METAS	-	-
4	2009.02	METAS	2009.04	IMT
5	2009.04	METAS	2009.06	INRIM
6	2009.07	METAS	2009.08	IMBiH
7	2009.10	METAS	2009.11	MIKES
8	2010.02	METAS	-	-

4.4 Collection of the results

The results communicated to the pilot laboratory included the residual drag, the uncertainty of the residual drag, the deceleration under the generated pressure, the temperature, the uncertainty on the temperature and the uncertainty on the reference pressure. Some additional values like the rotation speed have been included to make possible the correction of some unexpected influence factors but have not been needed for the determination of the reference value.

The results have been transmitted to a third party laboratory (LNE, Pierre Otal) once all the measurements had been finished in order to demonstrate the independence of all the participants.

5 Method used for the calculation of the reference value.

The mesurand of the comparison is the accommodation factor of two SRG sensor at different value of pressure. However for comparing the value measured by the different laboratories the values will have to be reduced to an unique reference value of pressure for each pressure step of the comparison.

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

$$\sigma_{ijkl}$$

Where

i is the number of the spinning rotating gage

j is the number of the nominal pressure

k is the number of the measurement in the serie at that specific nominal value

l is the number of the petal within the comparison and is also used to designate the NMI that did the measurement in the petal.

5.1 Influence of the reference pressure in the transition regime.

Since the value of accommodation factor is dependent of the pressure in the transition regime, the measured accommodation factors have to be corrected for the nominal value of pressure for measurement at nominal pressure $9.0 \cdot 10^{-2}$ Pa and above. A linear regression is then made on the measurement and the slope is used to correct the measurement.

$$\sigma_{ijk} = \hat{\sigma}_{ijk} + \left(P_j - \hat{p}_{ijk} \right) m_i$$

[5]

Where:

$\hat{p}_{ijk}$ is the effective pressure

P_j is the nominal pressure j

$\hat{\sigma}_{ijk}$ is the accommodation factor measured at the effective 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 $\hat{p}_{ijk}$ are very closed from their respective p_k.

5.2 Correction of the drift of the transfer standard

As the transfer standard is not stable during the whole time of the comparison, it is necessary to make the correction of the drift by using the measurements made by the pilot laboratory. Ideally it would be required to perform at the same time as the measurement in each laboratory a measurement of the SRG by the pilot laboratory. As this is not possible one has to rely on the average value of the measurement made by the pilot laboratory, before and after each participating NMI.

The value of the accommodation does not change in the molecular regime. It has been decided to calculate an average value $\bar{\sigma}_{il}(METAS)$ used for all measurements made at a nominal pressure of $3.0 \cdot 10^{-2}$ Pa or lower. This average value is calculated on the measurement performed at a nominal pressure between $9.0 \cdot 10^{-4}$ Pa and $3.0 \cdot 10^{-2}$ Pa

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{24} \sum_{k=1}^{k=6} \sum_{j=3}^{J=6} \sigma_{ijkl}(METAS) \qquad j\text{=1..6} \qquad [6]$$

The value of the accommodation factor at pressure above $3.0 \cdot 10^{-2}$ Pa is calculated for each pressure point.

$$\overline{\sigma}_{ijl}(METAS) = \frac{1}{6} \sum_{k=1}^{k=6} \sigma_{ijkl}(METAS) \qquad j\text{=7..9} \qquad [7]$$

For each participating NMI a value for the accommodation factor given by the pilot laboratory has been calculated my taking the average value:

$$\overline{\sigma}_{ij}(METAS,l,l+1) = \frac{1}{2}\left(\overline{\sigma}_{ijl}(METAS) + \overline{\sigma}_{ij(l+1)}(METAS)\right) \qquad [8]$$

5.3 Pressure value for a participating laboratory

The pressure realized in the participating laboratories has been calculated based on 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:

$$\sigma_{ij} = \frac{1}{6} \sum_{k=1}^{k=6} \sigma_{ijk} \qquad [9]$$

Then the measured pressure is:

$$p_{ij} = P_j \frac{\sigma_{ij}}{\overline{\sigma}_{ij}(METAS,l,l+1)} \qquad [10]$$

Where l is the number of the petal where the NMI is involved.

5.4 Reference value for a participating laboratory

The reference value for a participating laboratory is the weighted mean value of the reference value for each SRG. It is weighted by the combination of the type A uncertainties. This way of doing has the advantage that if an 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.

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

[11]

5.5 Pressure value for the pilot laboratory

The pressure determined by the pilot laboratory is taken as an average value before and after each participating NMI.

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

[12]

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

$$p_{ij}(METAS, l, l+1) = P_j \frac{\sigma_{ij}(METAS, l, l+1)}{\overline{\sigma}_{ij}(METAS, l, l+1)}$$

[13]

It should be mentioned that for nominal pressure above $3.0 \cdot 10^{-2}$ 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 petals. The weight coefficient is the combination of all the type A uncertainties.

$$p_{ij}(METAS) = \sum_{l=1}^{5} \frac{\dfrac{p_{ij}(METAS, l, l+1)}{u_A^2(p_{ij}(METAS, l, l+1))}}{\dfrac{1}{u_A^2(p_{ij}(METAS, l, l+1))}}$$

[14]

$$p_{ij}(METAS) = \frac{1}{5}\sum_{l=1}^{5} p_{ij}(METAS, l, l+1)$$

[15]

5.6 Reference value for the pilot laboratory

The reference value of the pilot laboratory is obtained like for the participating NMI's, through a weighted mean value in which the weight coefficient is the combination of the type A uncertainties:

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

[16]

5.7 Reference value of the comparison
The reference value of the comparison is obtained as a weighted mean value on all the participants that have a primary definition of the pressure.

$$p_j(EURAMET1040) = \frac{\sum_l \dfrac{p(l)}{u^2(p(l))}}{\sum_l \dfrac{1}{u^2(p(l))}}$$

[17]

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

5.8 Normalization of the reference value

The reference value given in equation 17 is slightly biased because in equation 10 we take only the accommodation coefficient 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 have as reference pressure of the comparison the nominal pressure. The coefficient of normalization is given by the weighted mean value among the laboratories who take part to the definition of the reference value.

$$p_{jc} = \frac{p_j}{p_j(EURAMET1040)}$$

[18]

And this way, the new reference value is equivalent to the nominal value:

$$p_j(EUR1040) \equiv P_j$$

[19]

5.9 Relative deviation to the reference value.

Due to the large span of value of pressure 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:

$$d_j = \frac{p_{jc}}{P_j} - 1$$

[20]

5.10 Degree of equivalence

Finally the degree of equivalence gives the ratio between the deviation and the uncertainty of the deviation. The degree of equivalence is given by:

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

[21]

6   Method used for the determination of the uncertainty.

6.1   Uncertainty on sigma measured by the participants

In the following discussion the measured quantity has sometimes 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 much closed.

6.1.1   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)

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

[22]

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

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

[23]

And the constant K is then given by:

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

[24]

The constant is the same for both SRG as it is some kind of conventional value that will affect the accommodation factor a similar way for all the participants.
The uncertainty of the sigma measured by the participants is estimated by taking into account the uncertainty of the generated pressure as provided 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.

6.1.2   Uncertainty on K

The uncertainty on the constant K has an influence on the calculation of the accommodation factor sigma. The factor K is however 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.

6.1.3   Uncertainty of the reference pressure

The uncertainty on the generated pressure has been provided by the participants for each measurement point. As the value of the relative uncertainty is almost insensible to slight change of pressure and as the values provided by the participants are similar from one cycle of measurement to another, an absolute uncertainty for each nominal pressure has been calculated. This uncertainty is a type B uncertainty and the sensitivity coefficient evaluated at the nominal pressure P_j is given by:

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

[25]

6.1.4   Contribution due to the uncertainty on the temperature

The collision rate is dependant of the temperature, an uncertainty on the temperature of the

SRG will affect the uncertainty of the accommodation factor. The sensitivity coefficient of the temperature is given by:

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

[26]

6.1.5 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:

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

[27]

6.1.6 Standard deviation of the value calculated for sigma
The standard deviation of a set of sigma 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 as explained by Kacker and Jones [6] to obtain the contribution to the uncertainty of sigma:

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

[28]

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

Finally the uncertainty on the accommodation factor is given by:

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

[29]

It is useful for the calculation of the weighted mean value to determine the combination of the type A uncertainties to the accommodation coefficient.

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

[30]

6.2   Uncertainty on the value of sigma used to correct the drift.

The uncertainty on the reference value of sigma used to compensate the drift of the SRG (the $\overline{\sigma}_{ij}(METAS, l, l+1)$ defined in equation XX) is given by the stability of the transfer standard. The uncertainty due to the stability of the SRG is defined the following way:

$$u\left(\overline{\sigma}_{ij}(METAS, l, l+1)\right) = 0.5\left|\overline{\sigma}_{ijl}(METAS) + \overline{\sigma}_{ij(l+1)}(METAS)\right|$$

[31]

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.
This definition has also already been used in previous comparisons. [x,y]

6.3   Uncertainty on sigma measured by the pilot

The uncertainty of the accommodation coefficient determined by the pilot laboratory is slightly different from 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 number of measurement:

$$u^2\left(\sigma_{ij}\left(METAS,l,l+1\right)\right)=\left(\frac{\partial\sigma}{\partial P_j}\right)^2 u^2\left(P_j\right)+$$

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

[32]

The combination of the type A uncertainties is given by:

$$u_A^2\left(\sigma_{ij}\left(METAS,l,l+1\right)\right)=$$

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

[33]

6.4   Uncertainty on the reduced pressure for the participants

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

$$u\left(p_{ij}\right)=P_j\sqrt{\left(\frac{u\left(\sigma_{ij}\right)}{\sigma_{ij}}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ij}\left(METAS,l,l+1\right)\right)}{\overline{\sigma}_{ij}\left(METAS,l,l+1\right)}\right)^2}$$

[34]

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

$$u_A\left(p_{ij}\right)=P_j\sqrt{\left(\frac{u_A\left(\sigma_{ij}\right)}{\sigma_{ij}}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ij}\left(METAS,l,l+1\right)\right)}{\overline{\sigma}_{ij}\left(METAS,l,l+1\right)}\right)^2}$$

[35]

6.5   Uncertainty on the reduced pressure for the pilot, for one petal

The calculation of the uncertainty of the pilot on one petal is similar to the calculation for the participants, the only difference is the definition of the accommodation factor determined by the pilot which is the average value of two measurements.

$$u\left(p_{ij}\left(METAS,l,l+1\right)\right)=P_j\sqrt{\left(\frac{u\left(\sigma_{ij}\left(METAS,l,l+1\right)\right)}{\sigma_{ij}\left(METAS,l,l+1\right)}\right)^2+\left(\frac{u\left(\overline{\sigma}_{ij}\left(METAS,l,l+1\right)\right)}{\overline{\sigma}_{ij}\left(METAS,l,l+1\right)}\right)^2}$$

[36]

The combination of the type A uncertainties is given by:

$$u_A\big(p_{ij}(METAS,l,l+1)\big) = P_j \sqrt{\left(\frac{u_A\big(\sigma_{ij}(METAS,l,l+1)\big)}{\sigma_{ij}}\right)^2 + \left(\frac{u\big(\overline{\sigma}_{ij}(METAS,l,l+1)\big)}{\overline{\overline{\sigma}}_{ij}(METAS,l,l+1)}\right)^2} \quad [37]$$

Finally, the uncertainty on the reference value of pressure, for a given SRG obtained, obtained by the weighted mean value of the measurements of the 5 petals

$$u^2\big(p_{ij}(METAS)\big) = u^2(P_j) + \sum_{l=1}^{5} \frac{1}{\dfrac{1}{u_A^2\big(p_{ij}(METAS,l,l+1)\big)}} \quad [38]$$

Where:

$$u_A^2\big(p_{ij}(METAS)\big) = \sum_{l=1}^{5} \frac{1}{\dfrac{1}{u_A^2\big(p_{ij}(METAS,l,l+1)\big)}} \quad [39]$$

Is the combination of the uncertainties of type A.

6.6 Uncertainty on the reference value of a participant

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

$$u^2(p_j) = u^2(P_j) + \frac{1}{\displaystyle\sum_{i=1}^{i=2} \frac{1}{u_A^2(p_{ij})}} \quad [40]$$

6.7 Uncertainty on the reference value of the pilot

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

$$u^2\big(p_j(METAS)\big) = u^2(P_j) + \frac{1}{\displaystyle\sum_{i=1}^{i=2} \frac{1}{u_A^2\big(p_{ij}(METAS)\big)}} \quad [41]$$

6.8 Uncertainty on the reference value of the comparison

The uncertainty of the reference value of the comparison obtained with Eq.17 is given by Cox [7]:

$$u^2\big(p_j(EURAMET1040)\big) = \frac{1}{\displaystyle\sum_{l} \frac{1}{u^2(p(l))}} \quad [42]$$

The uncertainty on the normalized reference values given by Eq. 19 is similar to Eq. 42 as the coefficient is closed to 1.

6.9   Uncertainty on the relative deviation

The uncertainty on the relative deviation calculated by Eq. 19 is given by Cox [7] and is as follow for the laboratories participating to the definition of the reference value:

$$U(d_j(l)) = 2\sqrt{\left(\frac{u(p_{jc})}{p_{jc}}\right)^2 - \left(\frac{u(p_j(EUR1040))}{p_j(EUR1040)}\right)^2} \qquad [43]$$

While for the participants not contributing to the reference value the uncertainty on the relative deviation is :

$$U(d_j(l)) = 2\sqrt{\left(\frac{u(p_{jc})}{p_{jc}}\right)^2 + \left(\frac{u(p_j(EUR1040))}{p_j(EUR1040)}\right)^2} \qquad [44]$$

7   Results provided by the participants.

The results of all the participants, including all the measurements made by the pilot are presented in table 5 and 6. The accommodation coefficient versus pressure is displayed in Fig 2 and Fig 3 for respectively the SRG A and B in circulation. The value is the mean of the measurements made by the participants and the uncertainty is given by equation 29 for all participants, pilot included.

The plot shows a relatively wide spread of the measured values due to the drift of the accomodation coefficient of the SRG with time. The viscous effect above 0.03 Pa is also responsible for a decrease of the value of the accommodation coefficient.

Table 5: Accommodation coefficient measured by the laboratories for the transfer standard A and uncertainties associated calculated using the Eq. 29.

P_i (Pa)		METAS1	CMI	METAS2	EIM	METAS3	METAS4	IMT	METAS5	INRIM	METAS6	IMBiH	METAS7	MIKES	METAS8
1.0E-04	σ_{11}	1.0105	1.0149	1.0117	1.0399	1.0120	1.0126	1.0193	1.0133	1.0121	1.0257	1.0332	1.0278	-	1.0293
	$u(\sigma_{11})$	0.0144	0.0113	0.0144	0.0198	0.0144	0.0144	0.0073	0.0144	0.0134	0.0145	0.0134	0.0145	-	0.0145
3.0E-04	σ_{12}	1.0105	1.0197	1.0117	1.0197	1.0120	1.0126	1.0191	1.0133	1.0153	1.0257	1.0279	1.0278	-	1.0293
	$u(\sigma_{12})$	0.0061	0.0109	0.0061	0.0121	0.0061	0.0061	0.0058	0.0061	0.0094	0.0062	0.0073	0.0062	-	0.0062
9.0E-04	σ_{13}	1.0105	1.0220	1.0117	1.0156	1.0120	1.0126	1.0188	1.0133	1.0152	1.0257	1.0283	1.0278	1.0371	1.0293
	$u(\sigma_{13})$	0.0052	0.0096	0.0052	0.0111	0.0052	0.0052	0.0057	0.0052	0.0069	0.0053	0.0058	0.0053	0.0334	0.0053
3.0E-03	σ_{14}	1.0105	1.0198	1.0117	1.0136	1.0120	1.0126	1.0189	1.0133	1.0152	1.0257	1.0278	1.0278	1.0317	1.0293
	$u(\sigma_{14})$	0.0031	0.0085	0.0031	0.0109	0.0031	0.0031	0.0057	0.0031	0.0061	0.0032	0.0058	0.0032	0.0207	0.0032
9.0E-03	σ_{15}	1.0105	1.0202	1.0117	1.0127	1.0120	1.0126	1.0189	1.0133	1.0157	1.0257	1.0283	1.0278	1.0298	1.0293
	$u(\sigma_{15})$	0.0030	0.0086	0.0030	0.0109	0.0030	0.0030	0.0057	0.0030	0.0042	0.0031	0.0057	0.0031	0.0095	0.0031
3.0E-02	σ_{16}	1.0105	1.0212	1.0117	1.0121	1.0120	1.0126	1.0184	1.0133	1.0157	1.0257	1.0284	1.0278	1.0283	1.0293
	$u(\sigma_{16})$	0.0030	0.0095	0.0030	0.0110	0.0030	0.0030	0.0057	0.0030	0.0039	0.0031	0.0057	0.0031	0.0082	0.0031
9.0E-02	σ_{17}	1.0087	1.0201	1.0099	1.0108	1.0108	1.0108	1.0173	1.0118	1.0134	1.0240	1.0274	1.0263	1.0270	1.0280
	$u(\sigma_{17})$	0.0030	0.0095	0.0030	0.0109	0.0030	0.0030	0.0057	0.0030	0.0019	0.0031	0.0057	0.0031	0.0079	0.0031
3.0E-01	σ_{18}	1.0065	1.0177	1.0088	1.0066	1.0078	1.0085	1.0134	1.0100	1.0091	1.0218	1.0246	1.0245	1.0230	1.0258
	$u(\sigma_{18})$	0.0020	0.0099	0.0020	0.0108	0.0020	0.0020	0.0056	0.0020	0.0012	0.0021	0.0057	0.0021	0.0077	0.0021
9.0E-01	σ_{19}	0.9952	1.0064	0.9967	0.9972	0.9973	0.9973	1.0024	0.9985	0.9975	1.0101	1.0131	1.0124	1.0121	1.0139
	$u(\sigma_{19})$	0.0020	0.0097	0.0020	0.0107	0.0020	0.0020	0.0057	0.0020	0.0010	0.0020	0.0056	0.0020	0.0076	0.0020

Table 6: Accommodation coefficient measured by the laboratories for the transfer standard B and uncertainties associated calculated using the Eq. 29.

| P_i (Pa)		METAS1	CMI	METAS2	EIM	METAS3	METAS4	IMT	METAS5	INRIM	METAS6	IMBiH	METAS7	MIKES	METAS8
1.0E-04	σ_{21}	0.9924	0.9982	0.9901	1.0262	0.9888	0.9869	0.9888	0.9867	0.9827	0.9856	0.9943	0.9851	-	0.9814
	$u(\sigma_{21})$	0.0122	0.0139	0.0122	0.0191	0.0122	0.0122	0.0064	0.0122	0.0104	0.0122	0.0149	0.0122	-	0.0121
3.0E-04	σ_{22}	0.9924	1.0003	0.9901	1.0015	0.9888	0.9869	0.9892	0.9867	0.9862	0.9856	0.9891	0.9851	-	0.9814
	$u(\sigma_{22})$	0.0058	0.0121	0.0058	0.0120	0.0058	0.0058	0.0056	0.0058	0.0091	0.0058	0.0067	0.0058	-	0.0057
9.0E-04	σ_{23}	0.9924	1.0011	0.9901	0.9932	0.9888	0.9869	0.9892	0.9867	0.9837	0.9856	0.9873	0.9851	0.9876	0.9814
	$u(\sigma_{23})$	0.0051	0.0137	0.0051	0.0108	0.0051	0.0051	0.0055	0.0051	0.0067	0.0051	0.0056	0.0051	0.0331	0.0051
3.0E-03	σ_{24}	0.9924	0.9998	0.9901	0.9914	0.9888	0.9869	0.9892	0.9867	0.9840	0.9856	0.9868	0.9851	0.9863	0.9814
	$u(\sigma_{24})$	0.0030	0.0086	0.0030	0.0107	0.0030	0.0030	0.0055	0.0030	0.0051	0.0030	0.0055	0.0030	0.0198	0.0030
9.0E-03	σ_{25}	0.9924	0.9998	0.9901	0.9909	0.9888	0.9869	0.9891	0.9867	0.9834	0.9856	0.9873	0.9851	0.9870	0.9814
	$u(\sigma_{25})$	0.0030	0.0084	0.0030	0.0106	0.0030	0.0030	0.0055	0.0030	0.0044	0.0030	0.0054	0.0030	0.0091	0.0030
3.0E-02	σ_{26}	0.9924	1.0006	0.9901	0.9902	0.9888	0.9869	0.9887	0.9867	0.9843	0.9856	0.9875	0.9851	0.9861	0.9814
	$u(\sigma_{26})$	0.0030	0.0094	0.0030	0.0108	0.0030	0.0030	0.0054	0.0030	0.0038	0.0030	0.0055	0.0030	0.0079	0.0030
9.0E-02	σ_{27}	0.9907	0.9995	0.9883	0.9892	0.9874	0.9856	0.9877	0.9851	0.9843	0.9842	0.9866	0.9835	0.9851	0.9800
	$u(\sigma_{27})$	0.0030	0.0092	0.0030	0.0107	0.0030	0.0030	0.0054	0.0030	0.0014	0.0030	0.0054	0.0030	0.0076	0.0030
3.0E-01	σ_{28}	0.9886	0.9972	0.9869	0.9855	0.9845	0.9836	0.9841	0.9834	0.9799	0.9822	0.9840	0.9813	0.9815	0.9779
	$u(\sigma_{28})$	0.0021	0.0092	0.0021	0.0105	0.0021	0.0021	0.0054	0.0021	0.0015	0.0021	0.0054	0.0021	0.0074	0.0021
9.0E-01	σ_{29}	0.9778	0.9865	0.9757	0.9742	0.9745	0.9731	0.9739	0.9726	0.9691	0.9715	0.9733	0.9706	0.9718	0.9671
	$u(\sigma_{29})$	0.0020	0.0093	0.0020	0.0104	0.0020	0.0020	0.0054	0.0020	0.0010	0.0020	0.0054	0.0020	0.0073	0.0020
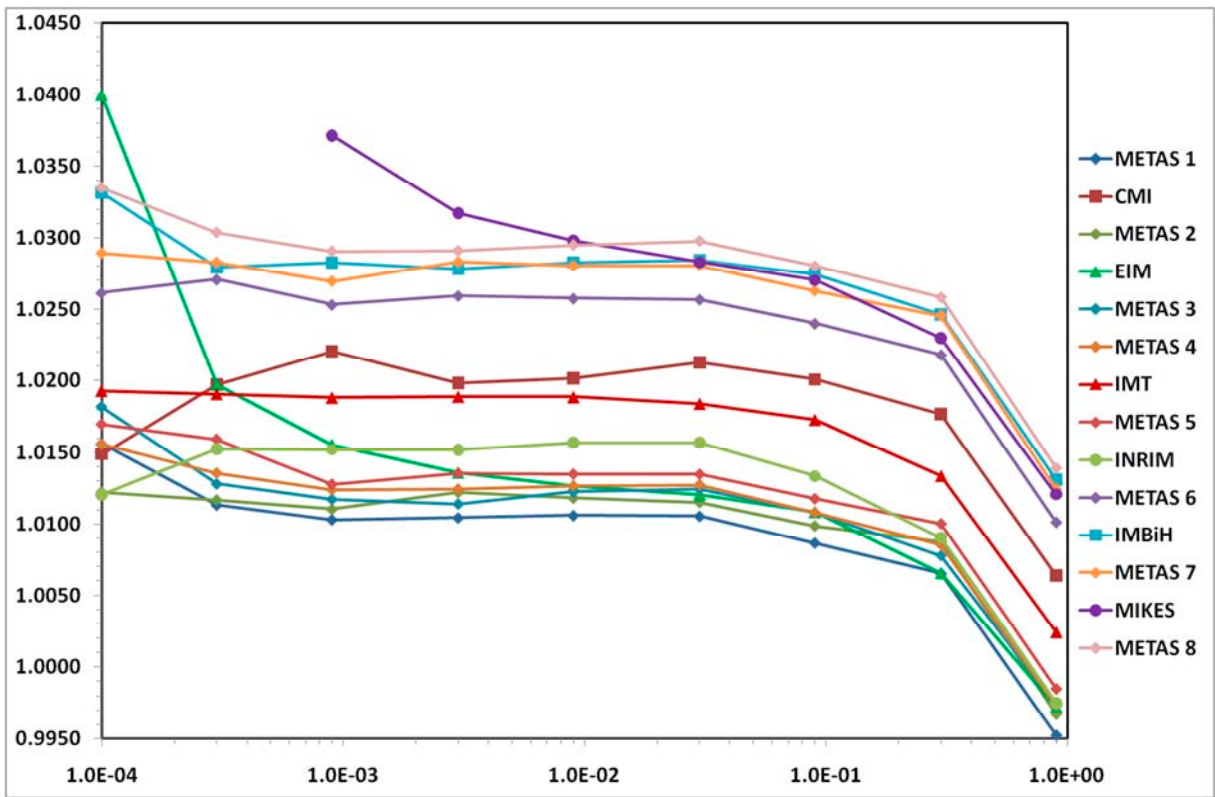
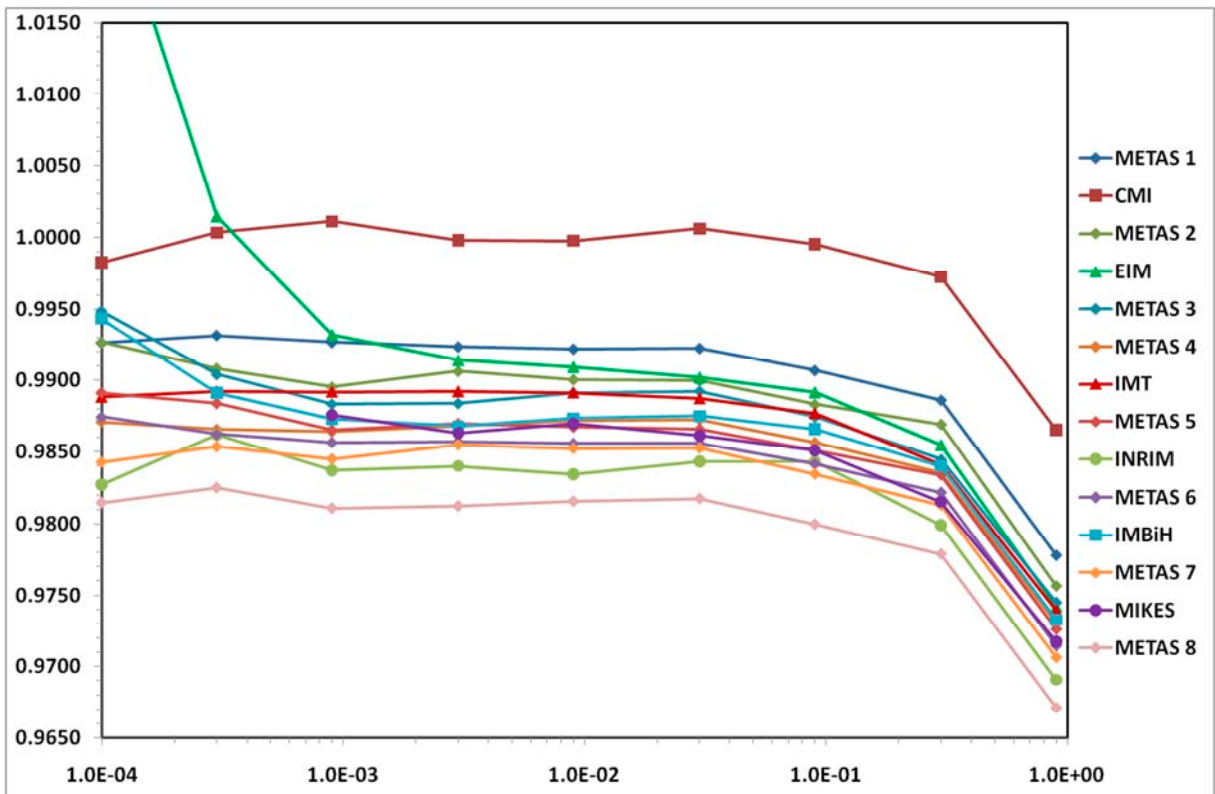**Fig. 2:** Accomodation coefficient measured on the SRG A by the different participants.

Fig 3: Accommodation coefficient measured on SRG B by the different participants.

7.1 Measurements of the pilot laboratory and stability.

The transfer standard has been measured by the pilot laboratory before the circulation, between each participant and after the circulation. It was not possible to extend the stability measurement before the comparison as the two SRG originally planned for this work had been dropped while in rotation due to a failure of the controller.

The stability of the transfer standard is presented in relative unit respective to the value of the initial measurement in Fig.1. The stability over a period of two years is in the order of 2 %. The stability between two successive measurement by the pilot laboratory is 10 times better, in the order of 0.2 %. A large change in the accommodation coefficient of the transfer standard A (SRG G191872) between April and July 2009 has some influence on the uncertainty of the reference value used in that petal. The uncertainty on the reference value of the laboratory involved in that petal, is however only marginally diluted due to the weighted mean and the uncertainty as given by equation 40.
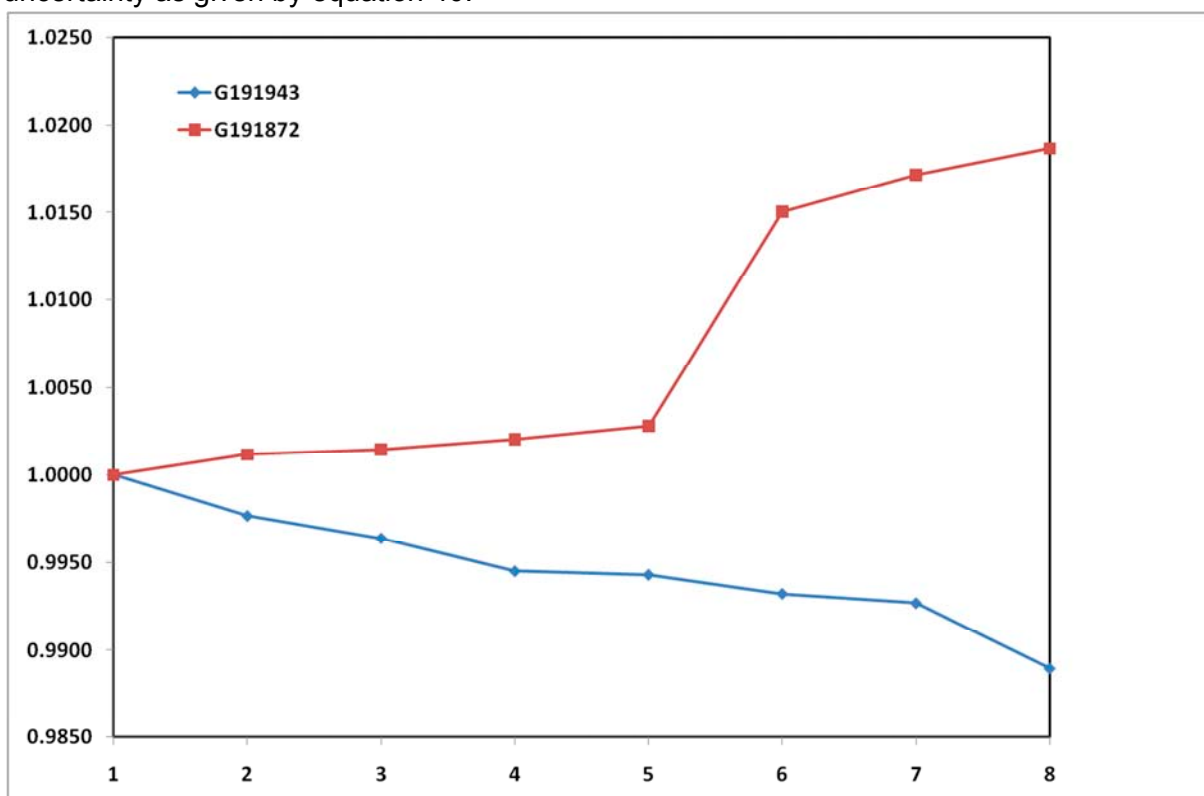

Fig. 4:   Accommodation coefficient relative to the initial value for the SRG A (red) and B (blue). The numbers on the horizontal axis denote the successive measurements performed by the pilot during the time of the comparison.

8   Reduction to a reference value

The determination of a reference value has been made on the base of the three primary laboratories of the comparison (CMI, INRIM and METAS). The relatively large difference between their respective uncertainties should not be a problem as the weighted mean should not lead to a dilution of the reference value.

The consistency check defined by Cox [7] using the chi squared function has been applied to the three laboratories contributing to the reference value and it has been fulfilled with success.

The normalised reference pressure as given by Eq. 18 for each participant and the associated uncertainties as given by Eq. 40 and 41 are presented on table 7.

Table 7: Normalized value of the participants as given by Eq. 18 and the associated uncertainty given by Eq. 40 and 41. The first column gives the normalized uncertainty of the comparison which by definition is equivalent to the nominal pressure and the associated uncertainty given by Eq. 42.

P_j (Pa)		Reference	CMI	EIM	IMT	INRIM	IMBiH	MIKES	METAS
1.0E-04	p_1	1.0000E-04	1.0008E-04	1.0307E-04	9.9937E-05	9.9186E-05	1.0032E-04	-	1.0069E-04
	$u(p_1)$	6.00E-07	1.05E-06	1.86E-06	6.51E-07	1.05E-06	1.29E-06	-	1.02E-06
3.0E-04	p_2	3.0000E-04	3.0156E-04	3.0208E-04	3.0014E-04	2.9861E-04	2.9971E-04	-	3.0004E-04
	$u(p_2)$	1.21E-06	3.01E-06	3.61E-06	1.71E-06	2.72E-06	2.01E-06	-	1.52E-06
9.0E-04	p_3	9.0000E-04	9.0955E-04	9.0329E-04	9.0354E-04	8.9751E-04	9.0143E-04	9.0656E-04	8.9861E-04
	$u(p_3)$	3.36E-06	8.49E-06	9.86E-06	5.07E-06	6.22E-06	5.17E-06	2.89E-05	4.52E-06
3.0E-03	p_4	3.0000E-03	3.0244E-03	3.0041E-03	3.0103E-03	2.9913E-03	3.0021E-03	3.0076E-03	2.9996E-03
	$u(p_4)$	7.55E-06	2.49E-05	3.26E-05	1.69E-05	1.60E-05	1.69E-05	6.03E-05	9.11E-06
9.0E-03	p_5	9.0000E-03	9.0767E-03	9.0083E-03	9.0330E-03	8.9712E-03	9.0131E-03	9.0179E-03	9.0022E-03
	$u(p_5)$	2.18E-05	7.45E-05	9.71E-05	5.06E-05	4.13E-05	5.05E-05	8.33E-05	2.73E-05
3.0E-02	p_6	3.0000E-02	3.0286E-02	3.0008E-02	3.0098E-02	2.9932E-02	3.0048E-02	3.0020E-02	3.0007E-02
	$u(p_6)$	7.08E-05	2.77E-04	3.28E-04	1.69E-04	1.23E-04	1.69E-04	2.43E-04	9.11E-05
9.0E-02	p_7	9.0000E-02	9.0938E-02	9.0086E-02	9.0365E-02	8.9950E-02	9.0229E-02	9.0125E-02	9.0004E-02
	$u(p_7)$	1.51E-04	8.26E-04	9.74E-04	5.06E-04	1.86E-04	5.03E-04	6.98E-04	2.73E-04
3.0E-01	p_8	3.0000E-01	3.0333E-01	3.0016E-01	3.0107E-01	2.9942E-01	3.0097E-01	3.0029E-01	3.0041E-01
	$u(p_8)$	4.32E-04	2.78E-03	3.22E-03	1.69E-03	6.19E-04	1.68E-03	2.29E-03	6.16E-04
9.0E-01	p_9	9.0000E-01	9.1067E-01	9.0100E-01	9.0387E-01	8.9849E-01	9.0333E-01	9.0198E-01	9.0148E-01
	$u(p_9)$	1.20E-03	8.38E-03	9.63E-03	5.12E-03	1.60E-03	5.05E-03	6.85E-03	1.85E-03

Table 8: Relative difference respective to the reference value of the comparison as defined by Eq. 20 and relative uncertainty as defined by Eq. 43 and 44. 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)		CMI	EIM	IMT	INRIM	IMBiH	MIKES	METAS
1.0E-04	d_1	0.0008	0.0307	-0.0006	-0.0081	0.0032	-	0.0069
	$u(d_1)$	0.0172	0.0379	0.0176	0.0173	0.0283	-	0.0163
3.0E-04	d_2	0.0052	0.0069	0.0005	-0.0046	-0.0010	-	0.0001
	$u(d_2)$	0.0182	0.0252	0.0140	0.0163	0.0156	-	0.0061
9.0E-04	d_3	0.0106	0.0037	0.0039	-0.0028	0.0016	0.0073	-0.0015
	$u(d_3)$	0.0171	0.0231	0.0135	0.0117	0.0137	0.0642	0.0068
3.0E-03	d_4	0.0081	0.0014	0.0034	-0.0029	0.0007	0.0025	-0.0001
	$u(d_4)$	0.0157	0.0222	0.0123	0.0094	0.0123	0.0404	0.0034
9.0E-03	d_5	0.0085	0.0009	0.0037	-0.0032	0.0015	0.0020	0.0002
	$u(d_5)$	0.0157	0.0221	0.0122	0.0078	0.0122	0.0191	0.0037
3.0E-02	d_6	0.0095	0.0003	0.0033	-0.0023	0.0016	0.0007	0.0002
	$u(d_6)$	0.0177	0.0224	0.0122	0.0067	0.0122	0.0168	0.0038
9.0E-02	d_7	0.0104	0.0010	0.0041	-0.0006	0.0025	0.0014	0.0000
	$u(d_7)$	0.0179	0.0219	0.0117	0.0024	0.0116	0.0159	0.0051
3.0E-01	d_8	0.0111	0.0005	0.0036	-0.0019	0.0032	0.0010	0.0014
	$u(d_8)$	0.0181	0.0217	0.0116	0.0030	0.0116	0.0156	0.0029
9.0E-01	d_9	0.0119	0.0011	0.0043	-0.0017	0.0037	0.0022	0.0016
	$u(d_9)$	0.0182	0.0216	0.0117	0.0024	0.0115	0.0154	0.0031
	$\frac{1}{N}\sum_1^N \lvert d_i \rvert$	0.0085	0.0052	0.0030	0.0031	0.0021	0.0019	0.0014

8.1 Difference and uncertainty respective to the reference value.

The relative differences to the reference value as given by Eq. 20 and the associated uncertainty are summarised in table 8. A more visual presentation of the relative deviation is presented on Fig. 5. It is obvious that for most of the participants it is challenging to keep a small relative deviation at small pressure.
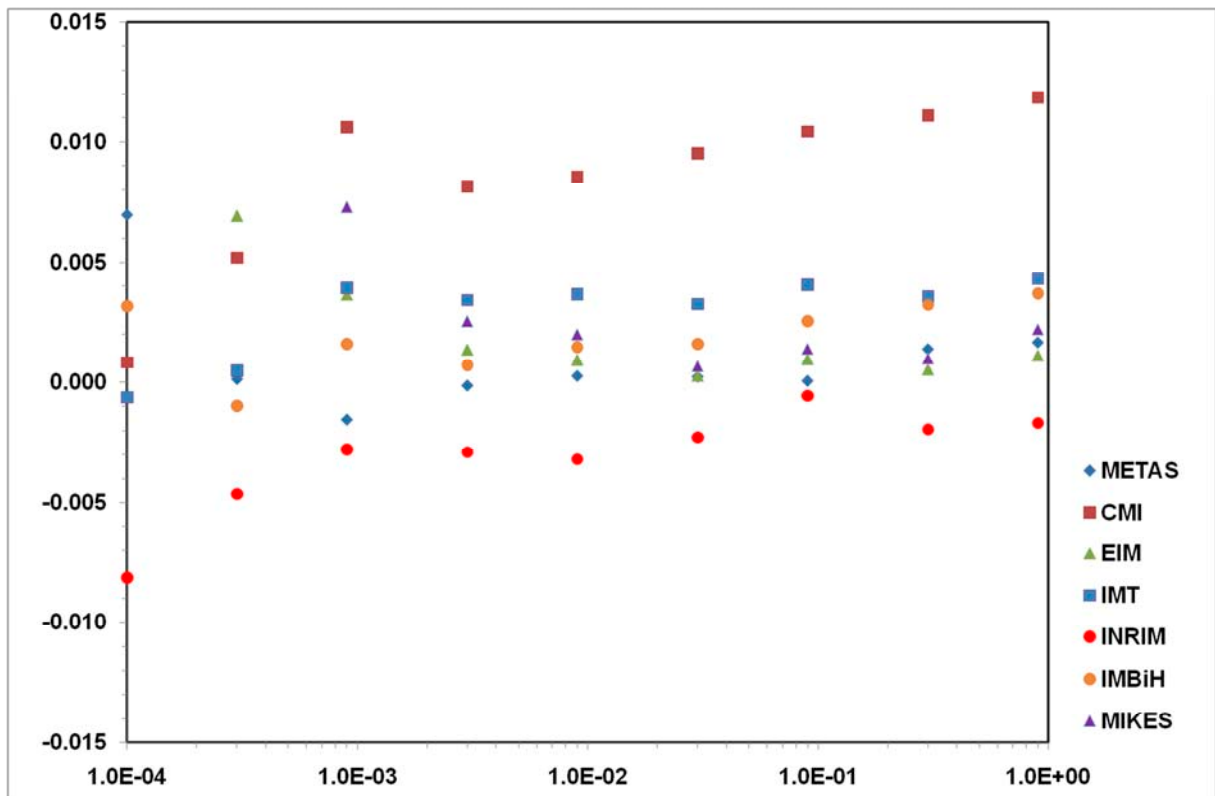
Fig. 5: Relative deviation respective to the reference pressure for all the participants. The deviation of EIM at 0.1 mPa is not present on the graph.

8.2 Degree of equivalence.

In order to understand the importance of the deviation relatively to the uncertainty it is necessary to determine the ratio between the deviation and the uncertainty. This is done using the Eq. 21 for the determination of the degree of equivalence. The values for all the participants are presented in table 9 while the same results are presented in Fig. 6. At this point it is important to note that all the participants have an equivalent definition of the pressure as the value for the degree of equivalence lies within -1 and 1.

Table 9: Degree of equivalence of the participants as calculated by Eq. 21. All the participants agree with the criterion of equivalence.

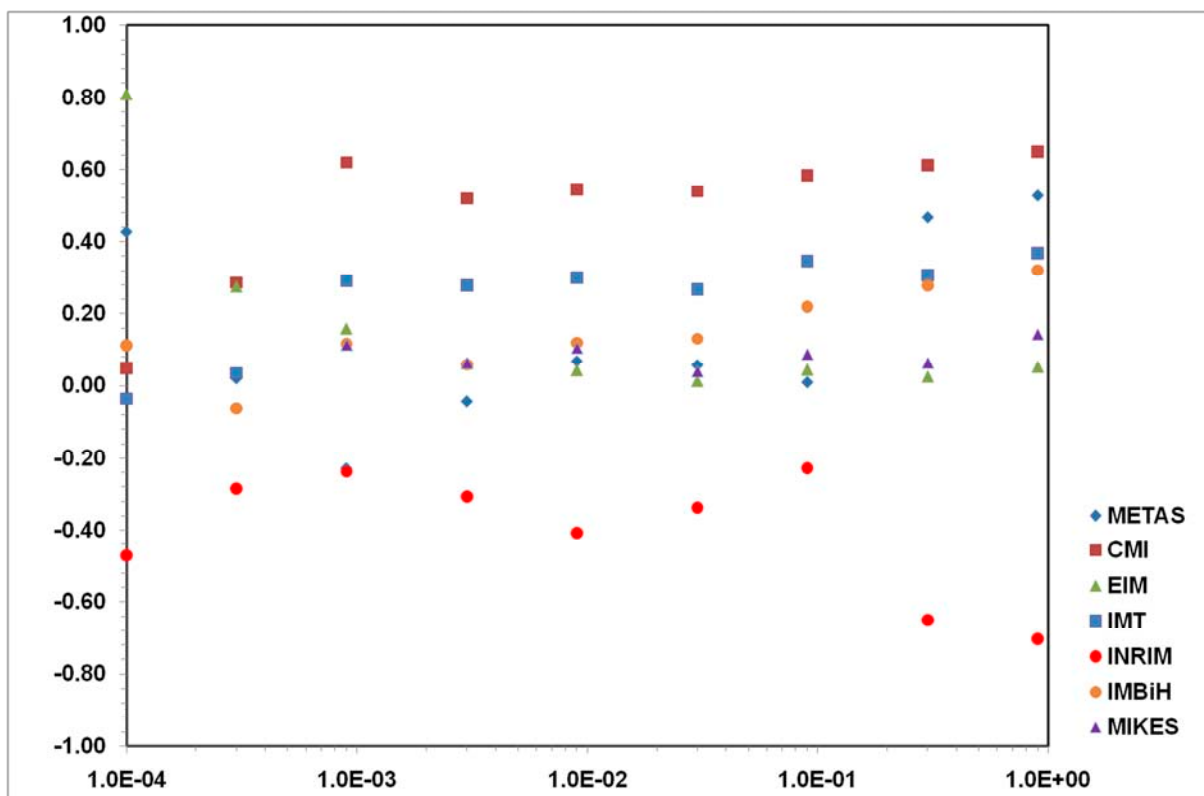| P_i (Pa)	CMI	EIM	IMT	INRIM	IMBiH	MIKES	METAS
1.0E-04	0.05	0.81	-0.04	-0.47	0.11	-	0.43
3.0E-04	0.29	0.28	0.03	-0.29	-0.06	-	0.02
9.0E-04	0.62	0.16	0.29	-0.24	0.12	0.11	-0.23
3.0E-03	0.52	0.06	0.28	-0.31	0.06	0.06	-0.04
9.0E-03	0.54	0.04	0.30	-0.41	0.12	0.10	0.07
3.0E-02	0.54	0.01	0.27	-0.34	0.13	0.04	0.06
9.0E-02	0.58	0.04	0.35	-0.23	0.22	0.09	0.01
3.0E-01	0.61	0.02	0.31	-0.65	0.28	0.06	0.47
9.0E-01	0.65	0.05	0.37	-0.70	0.32	0.14	0.53

Fig. 6: Ratio between the deviation relative to the reference and the associated expanded uncertainty (i.e. plot of the degree of equivalence). All the values for all the participants are visible on this plot demonstrating the equivalence of the definition of the pressure by all the laboratories.

9 Link to key comparison.

This comparison is a pilot comparison for the project CCM.M.P.K-14 as defined in the document CIPM MRA-D-05 [8] and the experience gained in this work has been profitable to the success of CCM.P.K-14. It is planned to link the reference value of this comparison to the reference value of CCM.P.K-14 once the report will be published. This will however not change the official status of this work as supplementary comparison for the CCM.

10 References

[1]   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 $3 \cdot 10^{-4}$ Pa to 0.9 Pa.

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

[3]   Calcatelli A, Raiteri G, Rumiano G. Measurement 2003; 34, 2: 121.

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

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

[6]   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

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

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