Protocol for the CCM comparison CCM.P-K15.1 for absolute pressures in the range from 100 µPa to 1 Pa

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The objective of this work is to link the National Institute of Metrology of China to the reference value define in the comparison CCM.P-K15.1. This comparison is motivated by the fact that the Chinese laboratory was not ready yet at the time of the selection of the participants of APMP. On the other side, one of the participants of APMP did not cover the whole range of CCM.P-K15 and the transfer standard did not exhibit the stability that was expected during the loop of APMP in spring 2010. This extension of CCM.P-K15 had been discussed during the CCM WG-LP in Berlin in April 2011.

Scope

Two spinning rotating gauges (SRG) have to be characterised with the pressure standards of each participating institute in the range from 100 µPa to 1.0 Pa. From these measurements, the accommodation factor of the SRG will be determined at each measurement point by which the generated pressures in the standards can be compared.

Participants

Laboratory	**Address**	**Person responsible for the intercomparison**
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Table 1 – List of Participants

Transfer standard

The transfer standards consist of a pair of SRGs kept under vacuum using a Varian all-metal valve. The specifications of the transfer standard are listed in the table bellow. Some of the characteristics have not been measured properly but will be used as conventional values in order to determine the accommodation coefficient.

Table 1: Characteristics of the transfer standards.

Transfer Standard	SRG-1	SRG-2
Metas Number	To be defined	To be defined
SRG Part Number (MKS)	SRG-BF	SRG-BF
SRG Serial Number	To be defined	To be defined
Valve part number (Varian)	9515027	9515027
Valve Serial Number	To be defined	To be defined
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: 405 Hz max: 415 Hz	min: 405 Hz max: 415 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 when the valve is open, and measurement of the residual drag of the ball has shown no spurious drag due to an electromagnetic coupling between the ball and the spring via the magnetic field of the ball.
One electronic readout unit and one measuring head will be circulated with the transfer standard. The participating laboratories can use this readout unit or their own unit for one SRG, but will need to supply their own unit for the second SRG. The characteristics of the readout unit are as follow:

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

A torque wrench will also be circulated to be used to close the all-metal valve with the specified torque. The following list gives the items that will be circulated:

Table 2: Packing list

Object	Weight	Serial number
SRG mounted on an all metal valve, including the handle and the bolt to fix the measuring head.	2.5 kg	SRG: To be defined Valve: To be defined
SRG mounted on an all metal valve, including the handle and the bolt to fix the measuring head.	2.5 kg	SRG: To be defined Valve: To be defined
Measuring Head for the SRG-SH700	0.8 kg	94187G60
Electronic unit MKS SRG-2CE/0	2.5 kg	500163G
Power cable for European plug	0.2 kg	-
Torque wrench Stahlwille Manoskop 730/5 0 – 50 Nm with ½'' head	0.8 kg	205020179

The two SRGs and the control unit will be shipped in a grey plastic box that is 66 cm x 53 cm x 47 cm in size and a mass of 25 kg. Each SRG shall be packed in a separate cardboard box. The measuring head shall be packed in a third cardboard box. The three cardboard boxes shall be packed into the grey plastic box along with the other items listed in Table 2.

The packing of the objects has to be made the same way as received. The following pictures show how the different objects will be packed together.

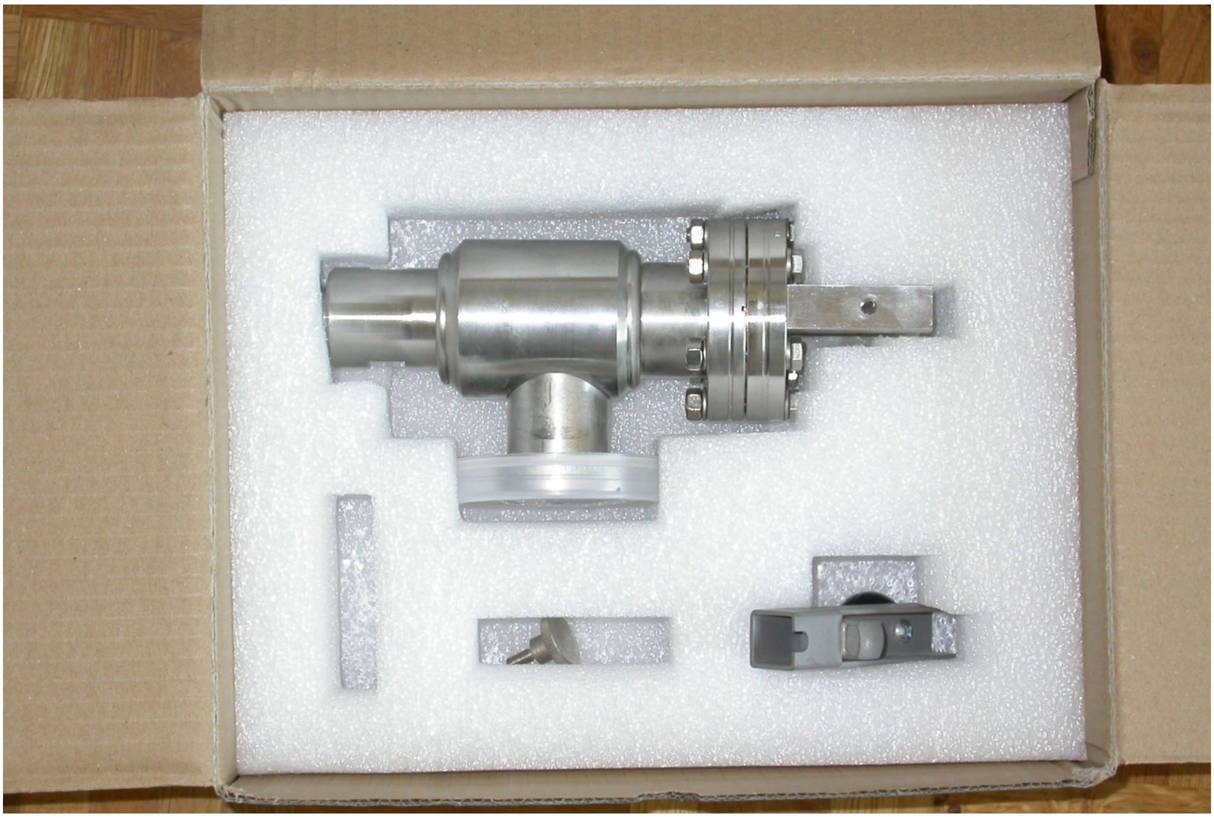

Fig. 1: All-metal valve bolted to the spinning rotating gage, screw to fix the measuring head and handle for the valve. All the items are together in a cardboard box.

Fig. 2: Measuring head in a cardboard box.

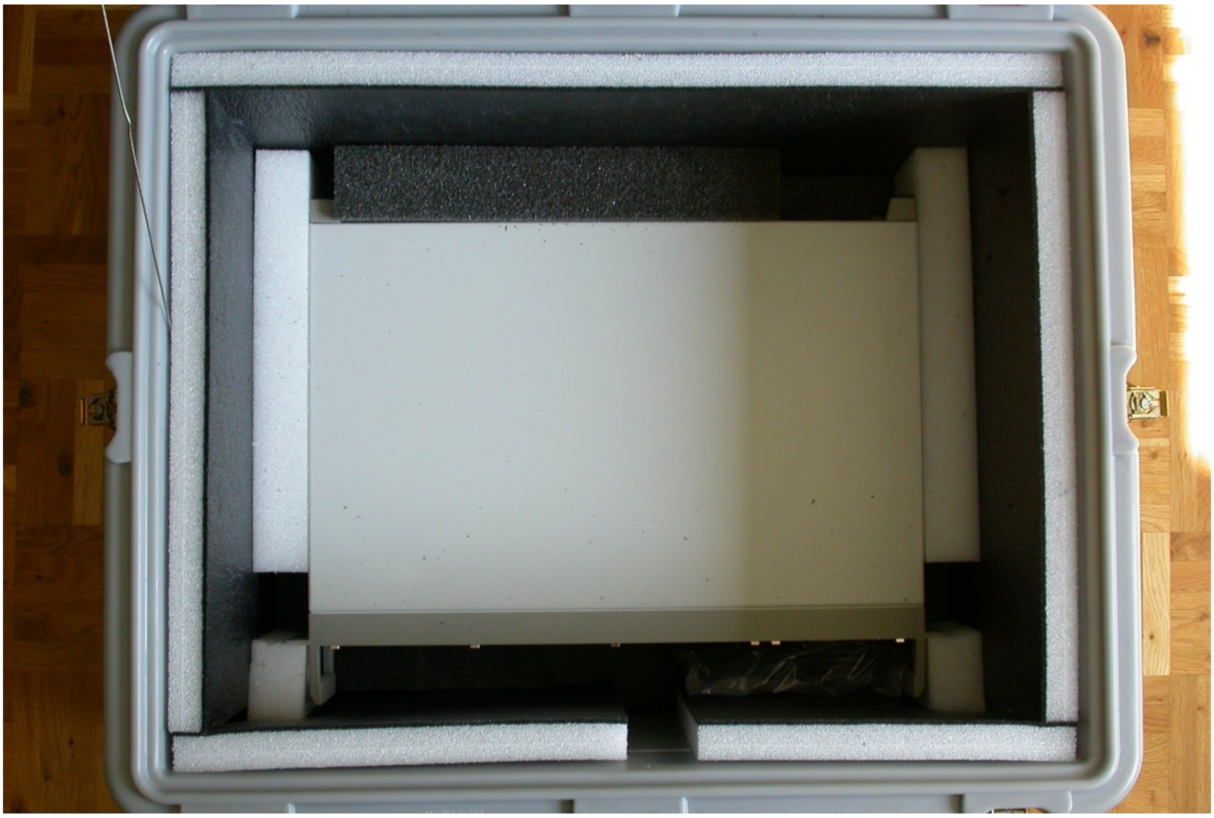

Fig. 3: The electronic unit is placed at the bottom of the box and secured with foam.

Fig. 4: The torque wrench and the three cardboard boxes are placed on the top and secured with foam.

The transfer standards are circulated for the comparison in accordance with the schedule in the Table 3 below..

The laboratory that ships the transfer standard must contact the ship-to laboratory and the pilot laboratory prior to the shipment to make sure that the transfer standard can be received and measured without problem. Complete the departure checklist given in the protocol and email it to the ship-to and pilot laboratories. The receiving laboratory shall inspect the material immediately upon arrival, complete the arrival checklist, and email it to the pilot laboratory. If there is any damage that could prevent the measurements, this must be communicated to the pilot laboratory, and the pilot lab will advise on the next course of action.

The equipment will be accompanied by an ATA carnet where necessary to avoid any tax problem.

Each lab is responsible for its own costs of the measurements, the costs of transportation to the next participant, and any custom charges within its country as well as any damage that may occur within its country.

Table 3: Time schedule

LAB /COUNTRY	RESPONSIBLE FOR THE COMPARISON	DATE FOR PARTICIPATION
Metas / Switzerland	Dr. Christian.Wüthrich	01.07.2011 – 30.09.2011
NIM / China	Dr. Li Yanhua	30.09.2011 – 15.01.2012
Metas / Switzerland	Dr. Christian.Wüthrich	16.01.2012 – 30.03.2012

July and October 2010 are considered as buffer months.

Comparison measurements

Measurand

The measurand of the comparison is the accommodation factor of the ball. The accommodation factor is determined during the comparison according to the following equation:

$$\sigma = \left(\frac{-\dot{\omega}}{\omega} - RD(\omega) \right) \frac{\pi d \rho}{10 p} \sqrt{\frac{2RT}{\pi m}} \tag{1}$$

Where

p generated pressure

RD residual drag at zero pressure due to eddy currents in the ball and in the surrounding metal pieces

ω rotational speed of the ball

$\dot{\omega}$ rate of change of rotational speed of ball

d the diameter of the ball

R gas constant (R=8.314472 J mol^{-1} K^{-1} $U(R)$=0.000014 J mol^{-1} K^{-1} CODATA 2006)

ρ density of the ball

m molar mass of the gas

T temperature

The accommodation coefficient is determined using the nominal ball diameter and ball density given in the description of the transfer standard. The accommodation coefficient is known to be constant in the molecular regime and decreases as the pressure increases in the laminar regime. For the target points > 0.03 Pa, σ_i is significantly dependent on pressure. For this reason, each laboratory must calculate a linear fit function for the three data points at 0.09 Pa, 0.3 Pa and 1 Pa: σ_{ij} versus p_{stj}. With the slope of this fit function it will be possible to calculate a σ_{ij} for each participant for the common value of p_{st}, even if the target pressures were not exactly matched during calibrations.

The measurement shall be performed with a rotation speed between 405 Hz and 415 Hz because the ball characteristics are well known in this range.

The RD (residual drag) and its uncertainty is an important influence factor that has to be determined carefully, especially for measurements performed at low pressure.

Two methods are possible to measure the residual drag, and the participants are free to choose the one that is the most adapted to their instrumentation. For each method, the uncertainty of the measurement shall be estimated.

Technique 1: Determination of *RD* before the measurement.

In this technique the ball is brought into rotation and the deceleration is measured under vacuum at near or less than 1 µPa. The measurement is repeated throughout the range of rotation speed. This measurement has the advantage that it could be performed automatically with only a small computer. A linear approximation of the residual drag versus the frequency is applied to correct the measured drag at the target pressures. This technique can be used if the *RD* is highly reproducible (stable room temperature) because it provides a way of reducing the uncertainty of a single measurement of *RD*.

Technique 2: Determination of *RD* during the measurement.

The residual drag is measured immediately before and/or after the measurement of the deceleration while the ball is exposed to the pressure. This can be accomplished by evacuating the chamber to a pressure below the *RD* pressure before or after the ball is exposed to the measurement pressure. This technique is preferable because the residual drag is a function of the temperature and this influence parameter is expected to remain stable during the small time span needed for the two measurements of the deceleration.

Care of the transfer standard

A few important points have to be observed in order to perform a stable and reproducible measurement and to ensure the stability of the transfer standard for all the time of the comparison.

Mounting the transfer standard on the measurement chamber

Vent the measurement chamber or the load lock and mount the transfer standard with the valve closed using a new copper gasket. Check that the measuring head is within 1 degree of vertical once it is mounted on the finger containing the ball. Once the verticality is good, tighten the bolts, making sure that the flange of the valve does not rotate during tightening. Evacuate the measuring chamber and wait until the pressure is less than 0.1 mPa before opening the all-metal valve. Open the valve completely to make sure that the spring is distant enough from the ball.

Important points to ensure the stability of the accommodation factor:

Do not bake the SRG

The SRG has been prepared by outgassing it properly before the comparison. Please do not bake it further because this could change the accommodation coefficient. It may be necessary to wait one week to get the vacuum conditions needed to achieve a good repeatability at 0.1 mPa.

Use an UPS (uninterrupted power supply) between the mains and the controller

Power failures are more common than often expected because most power failures last only a very short time. However, even a short power failure can crash a rotating ball and change the accommodation factor by a large amount.

Do not leave the ball in rotation without any need

There is almost no energy needed to keep the ball in rotation. However there is still a risk to have a crash.

Do not expose the ball to a strong magnetic field

The residual magnetic field of the ball is strong enough to be detected by the measuring head but relatively weak to keep the *RD* as small as possible. If the ball is exposed to a strong magnetic field, the value of *RD* may be changed, and this has an effect on the repeatability at low pressure.

Start the rotation of the ball 12 h before the measurement

The acceleration of the ball implies heating by eddy currents. The ball needs then 6 h to achieve thermal equilibrium with the vacuum chamber. During this time, there is a significant change of the RD due to the decreasing diameter of the cooling ball.

Check that the rotation speed is within the limit

It has been observed at METAS that the SRG2-CE can loose the rotation of the ball at pressures higher than 0.01 Pa. This is indicated by a rotation speed that is lower than the minimal rotation speed. The ball has then to be brought back into rotation. (Send “STA” to the SRG2-CE through the RS232 interface or hit the “ROTOR” key on the keyboard once.)

<u>Initial checks</u>

Before performing the intercomparison work, verify the following points:

- the residual drag must be of the order of $3 \cdot 10^{-7}$ s^{-1}.
- the signal of the control unit when visualised on an oscilloscope should be stable and clean with the harmonics barely visible.

<u>End of the measurement</u>

Deceleration of the ball

At the end of the measurement the ball has to be properly decelerated in order to avoid any change of the accommodation due to rubbing of the ball on the wall of the tube. This deceleration is performed by pressing the “ROTOR” key twice as described in the manual of the SRG-2CE.

Closure of the valve

The valve can be closed with the provided small handle while the SRG is still under a vacuum better than $1 \cdot 10^{-5}$ Pa. In order to ensure a good tightness of the valve without excessive wear of the valve seat, it has to be tightened with the provided torque wrench with a torque of 40 Nm. When applying this torque hold the valve with a 32 mm wrench on the flat of the body; otherwise a leak could appear from excessive stress in the body of the valve.

Measurements and target points

The measurement will be performed at the following 9 values of pressure of nitrogen in this order:

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

The effective pressure should be within 10% of the nominal pressure for pressures lower than $4\cdot10^{-2}$ Pa, and it should be within 5% for higher pressures.

A gas purity of 99,99% is recommended.

At each target point in each calibration sequence, measure the accommodation coefficient three times. (Note: In static expansion systems and comparison systems pump to zero and re-generate the same point, in continuous expansion systems it is sufficient to re-measure all quantities contributing to the calculated pressure).

It is recommended that both SRGs are calibrated at the same time.

The calibration sequence will be repeated at least once, making a total of at least 54 measurement points for one SRG.

The temperature of the system shall be monitored and should be 22±2 °C.

Apply the following measurement times and repetitions:

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Pressures	Duration of each measurement	Number of measurements at each pressure
$1\cdot10^{-4}$ Pa, $3\cdot10^{-4}$ Pa, $9\cdot10^{-4}$ Pa, $3\cdot10^{-3}$ Pa	30 s	10
$9\cdot10^{-3}$ Pa, $3\cdot10^{-2}$ Pa, $9\cdot10^{-2}$ Pa	30 s	5
0.3 Pa, 1 Pa	10 s	5

The setting of the printer of the MKS-SRG-2 can be used to obtain the correct number of repeat points, or an external computer can be used to achieve the correct number of measurements.

Report of results

The pilot laboratory will provide an Excel spreadsheet template to make the communication of the results uniform.

All pressure values shall be given in the unit of Pa. All values shall be given with 5 significant digits, and absolute uncertainties should cover the value's final digit. For example

p_{NMI} = *0.029938* $\pm$ *0.000046* Pa. Uncertainties shall be given as one standard uncertainty (k=1), as according to GUM.

In the following, all information that must be sent to the pilot lab is listed and, if necessary, explained. Some of the data will not be used for the evaluation, but are important for the correct realisation of the measurements and are therefore meant for your own quality control during the comparison.

1. A short description of your standard (if it is published, send the publication) including a description how the measurements were performed (positions of gauges, which option was used for offset measurement etc.).
2. Date of receipt of the transfer standard set in your institution.
3. Notes of inspection of the package (e.g. Damage? Completeness?).
4. Dates and times when the SRG valve was open.
5. Complete data set of initial offset measurements (Excel table), if applicable. This data set consists of all relevant data entered into the controller, date of measurement, residual pressure in the standard (measured with ion gauge), time of data point, offset in units of DCR (s^{-1}), rotor frequency, mean of all data points, standard deviation about the mean, standard deviation of the mean, linear fit: offset vs. rotor frequency (if applicable).
6. Complete data set of each SRG calibration day. This is an Excel file according to the form as attached. Name the file with your institution and the month and year of your first measurements, for example, "METAS_11.2009". This data set consists of the date of measurement, all relevant data entered into the controller, the results of the offset checks, target pressure, mean temperature of gas in your vessel, mean value of reading of SRG-1, standard deviation of the mean, offset corrected mean value of reading of SRG-1 (if applicable), the latter in Pa, mean value of reading of SRG-2 (if applicable), standard deviation of the mean (if applicable), offset corrected mean value of reading of SRG-2 (if applicable), the latter in Pa, generated pressure in the standard, its standard uncertainty, σ_i as calculated from Eq. (1) and its mean value σ_{ij} for the three measurements at the same nominal target pressure., standard uncertainty in *RD*, and standard uncertainty in temperature
7. The linear fit curve in the range 0.09 Pa to 1 Pa of the determined $(\sigma_{ij})_{\mathrm{det}}$ values vs p_{stj}. The two slopes m_i [Pa^{-1}] of this fit will be needed for further evaluation. Calculate for the nominal target pressures p_{nom} "standardised" values $(\sigma_{ij})_{nom}$ of σ_{ij} as determined with the following formula: p_{nom} are the nominal target pressures 0.09 Pa, 0.3 Pa, 1 Pa and p_{stj} the actually generated pressures close to p_{nom}.

$$(\sigma_{ij})_{nom} = (\sigma_{ij})_{\det} + (p_{nom} - p_{stj}) \cdot m_i \qquad (2)$$

p_{nom} are the nominal target pressures 0.09 Pa, 0.3 Pa, 1 Pa and p_{stj} the actually generated pressures close to p_{nom}. Estimate the uncertainty due to the linear fit.

Collection of data

The participants will communicate the results of the comparison within 6 weeks after all the measurements are finished.

Evaluation of the calibration results and generating a common reference value

The evaluation will mainly follow the procedure as described in [1]. The appropriate model (constant, linear, loopwise, etc.) for the long-term behaviour of the σ-values will be chosen according to the data obtained during the comparison.

Relevant uncertainties

All uncertainties should be given as (one-sigma) standard uncertainties as outlined in the GUM. The indicated value p_i of SRG *i* is uncertain due to the temperature determination, due the determination of the offset *RD*, a possible frequency dependence of the offset, a possible drift of the offset value between its determination and the time of calibration, and the Type A uncertainty of the measurement results. The uncertainty of the temperature entered in the controller shall be reported to the pilot lab. In most cases, however, the uncertainty due to temperature is relatively small.

The uncertainty of the calculated generated pressure p_{stj} has to be determined by each lab at the time of calibration. **Uncertainties of Type A (formerly called random uncertainties) should not be considered for the total uncertainty of p_{stj},** if they appear at immediate repeat calibrations and contribute to the repeatibility of p_i. This is because three immediate repeat measurements are required for each target point.

The uncertainty of σ_{ij} will be determined from the uncertainty of *T, RD* , p_i, and p_{stj} at the time of calibration.

References

1. *Karl Jousten, Mercede Bergoglio, Anita Calcatelli, Jean-Noel Durocher, John Greenwood, Rifat Kangi, Jean-Claude Legras, Carmen Matilla, Janez Setina:* Results of the regional key comparison Euromet.M.P-K1.b in the pressure range from $3\cdot10^{-4}$ Pa to 1 Pa, Technical Supplement to Metrologia, 2005, **42,** Tech. Suppl. 07001.