Bilateral Key Comparison between the National Pressure Standards of RISE and VTT MIKES in the range 15 kPa to 175 kPa (gauge pressure)

Technical Protocol

1. Introduction

This key comparison was initiated by the Swedish NMI for Pressure and Vacuum at RISE in order to demonstrate the metrological equivalence of their national standard in the range of 3.5 kPa to 170 kPa. The comparison will be piloted by RISE, and VTT MIKES who took part in the previous EURAMET key comparison (EURAMET.M.P-K8 from 2012) has agreed to provide the reference value.

The parameter to be compared is the determined effective area of a Piston Cylinder Assembly (PCA-unit) for each selected pressure level in the range 15 kPa to 175 kPa, gauge pressure in gas.

This technical protocol specifies the procedures to be followed in the comparison.

2. Participants

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Reference laboratory:	**VTT MIKES**
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3. Transfer standard

The transfer standard consists of a Ruska 2465-754 equipped with its low range PCA-unit (nominal pressure range 1.4 kPa to 170 kPa) and has been selected by RISE to be used for the comparison. The unit is provided by Siemens Energy AB through an agreement with RISE, in which Siemens receives a calibration of their pressure balance funded by RISE.

Transfer standard	Piston Cylinder Assembly
Manufacturer	Ruska
Model	2465-725
Serial number	TL-1523
Custom marking	-
Nominal pressure range	1.4 kPa to 170 kPa
Nominal effective area	336 mm^2
Thermal expansion coefficient	$1.5 \cdot 10^{-5}$ °C^{-1}
Piston mass *(k=2)*	47.193 06 g (*U*=0.10 mg)
Piston density	7700 kg/m^3
	Base unit
Model	2465-754
Serial number	39212
Supply voltage (engine for rotation)	110 V
	Temperature sensor
Model	PT100
Serial number	TC13332
Coefficients	
R0	99.9285 ohm
A	0.0039191
B	$-6.5381820 \cdot 10^{-7}$

Coefficient values for the PT100 sensor was provided by Siemens Energy AB in the calibration certificate no.1853-224035

		Weight set	
Model		-	
Serial number		39094	
Custom marking		TC110480	
Weight number	Mass value [g]	Uncertainty [g] *(k=2)*	Density
1 (mass carrier)	500.02052	0.0008	7800
2	1000.0129	0.0016	7800
3	1000.0327	0.0016	7800
4	1000.0321	0.0016	7800
5	1000.0397	0.0016	7800
6	1000.0295	0.0016	7800
7	500.00606	0.0008	7800
8	300.00433	0.0008	7800
9	200.00726	0.0003	7800
10	100.00716	0.00016	7800
11	50.00726	0.00010	7800
12	30.00689	0.00010	7800
13	20.005606	0.00008	7800
14	10.009804	0.00006	7800

The mass of the piston, mass carrier and set of weights have been calibrated by the NMI for Mass at RISE prior to the comparison. Mass values and uncertainties summarized here can be found in certificate *105102-190311-K02*, dated 2022-02-13--15.

4. Time schedule

The preliminary proposed time schedule is as follows:

Week of year 2022	Dates	Action
9*	28th Feb – 4th Mar 2022	Measurements (first calibration) of TS at RISE
37	12th Sep – 16th Sep 2022	Transportation of TS to VTT MIKES
40-42	3rd Oct – 21th Oct 2022	Measurements (calibration) of TS at VTT MIKES
43	24th Oct – 28th Oct 2022	Transportation of TS to RISE
44	31th Oct – 4st Nov 2022	Measurements (second calibration) of TS at RISE
43-52	7th Nov – 30th Dec 2022	Preparation and publication of the comparison report

**A previous calibration of the transfer standard, according to what has been specified in this technical protocol, was performed at RISE during March 2022 and will be used in the comparison to evaluate potential drift of the TS between the calibrations.*

The pilot laboratory will inform the participant and the participant must also inform the pilot laboratory in case of any delay.

Kommentiert [DS1]: Updated the schedule. TS is now at MIKES and they are planning to start their measurements Week 40

5. Transportation of the equipment and cost

The shipping costs of the equipment are covered by RISE.

The laboratory receiving the measurement equipment is responsible for post-delivery inspection to verify if any damage has been caused by the transport. Any anomaly should be reported, and then both laboratories will make a joint decision on how to proceed.

The cost of the calibration performed by VTT MIKES will be covered by RISE.

6. Laboratory standards

6.1 RISE

RISE national standard in the given pressure range is a Pressure Balance Ruska 2465-754 (serial number 53592). In addition it is equipped with its low range PCA-unit model 2465-725 (serial number TL-1194). It is traceable to the SI through regular calibrations at PTB in Germany.

The CMC uncertainty of this standard within the given pressure range is equal to
at 3.5 kPa $\le p \le$ 17 kPa: 0.2 Pa + $2.5\cdot10^{-5}p$
at 17 kPa $\le p \le$ 170 kPa: 1.0 Pa + $3.0\cdot10^{-5}p$

The standard uncertainty of this standard is estimated to be
at 3.5 kPa $\le p \le$ 170 kPa: 0.2 Pa + $2.5\cdot10^{-5}p$

6.2 VTT MIKES

VTT MIKES national standard in the given pressure range is a DHI pressure balance PG-7607 (No 397) with a piston cylinder unit (No 451) from DHI. The effective area (about 19.6 cm^2) of the PCU is traceable to Finnish national standards.

The CMC uncertainty of this standard within the given pressure range is equal to
at 15 kPa $\le p \le$ 170 kPa: 0.5 Pa + $2.2\cdot10^{-5}$p

7. Measurement procedures

The preparation of the measurements, as well as, their execution and reporting should be done according to procedures based on EURAMET cg-3 methodology (including measurement uncertainty estimation), and include the following specified steps:

7.1 Preparation

- Visual inspection of the instrument. The piston and cylinder shall also be inspected with respect to potential damage or deformations, which could influence the measurement results. Cleaning of the PCA-unit should be performed if deemed necessary.
- Reassembly and initial pressure balance functionality test including leak rate, and evaluation of piston fall rate and piston rotation speed.
- Alignment of reference levels between the transfer standard and the reference standard of the laboratory (if required).

As gas medium, dry nitrogen (N2) is to be used.

(Calibration in terms of mass for all reference weights and the piston/cylinder-assembly associated with the transfer standard have been performed by the NMI for mass at RISE prior to the comparison).

7.2 Calibration

The comparison measurements are to be carried out in four measurement series, two series of increasing pressure and two series of decreasing pressure (up, down, up, down) in the nominal pressure levels specified below. Each measurement series shall include one repetition at each pressure level (24 measurements in total):

Number	Nominal pressure level [kPa]	Transfer Standard weight combinations
1	15.1	8, 10, 11, 13
2	35.0	1, 7, 10, 11
3	70.1	1, 2, 7, 8, 11
4	104.9	1, 2, 3, 4, 12, 14
5	140.3	1, 2, 3, 4, 5, 9, 11
6	175.1	1, 2, 3, 4, 5, 6, 8, 10, 12, 14

If applicable, the direction of rotation for both pistons shall be changed during the second measurement series.

7.3 Stability check at RISE

Prior to the comparison (March 2022) a calibration of the transfer standard was performed at RISE, which will serve as the stability check. The calibration followed the same procedures as those specified in this technical protocol, but was limited to one measurement series with three repetitions at each of the six pressure levels (18 measurements in total). Also, the lowest nominal pressure level was changed from 7 kPa to 15 kPa to adapt to the lower limit used by VTT MIKES on their pressure standard. The results of these measurements will be used verify that the transfer standard maintained its functionality during transports and that no significant drift has occurred between the time of the calibrations and the stability check. In the case that the instrument proves to have drifted, uncertainty due to this drift will be included in the uncertainty of the reference value.

Kommentiert [DS2]: Cleaning requirement changed to "if nessesary"

Kommentiert [DS3]: Specified gas medium

Kommentiert [DS4]: MIKES suggested to perform the calibrarion in four measurment series (up, down, up, down) to suit there standard procedure.

8. Reporting

The participants should report the data of their own pressure standards filling the table given in Annex 1, as well as giving any additional information, if useful.

It should be reported how TS was connected to LS and how their pressure equilibrium was achieved and controlled.

Results of individual measurement series and a summary of the 4 measurement series should be reported using forms given in Annexes 2 and 3.

In addition, a list of main uncertainty sources and their contributions to $u(A_p)$ for minimum and maximum pressure must be reported.

Reports with the results of the measurements should be prepared as a WORD file with the tables from Annexes 2 and 3 being additionally provided in an EXCEL file.

The reports should initially be sent to the head of the EURAMET TC-M Subcommittee Pressure as an impartial instance by e-mail to

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within two months after finishing the measurements. Then they will be forwarded to the pilot laboratory. The pilot laboratory will evaluate the measurement data and prepare a report.

9. Evaluation

The reference value of the comparison is provided by the referens laboratory including associated uncertainties, and (if required) including influence due to drift.

The results will be compared in accordance with the statistical approach presented in the standard ISO/IEC 17043:2010 “Conformity assessment – General requirements for proficiency testing”. The degree of conformity will be described by the number E_n calculated from the mean value of determined effective area at each pressure level, according to the following equation

$$E_n = \frac{x - X}{\sqrt{{U_{\mathrm{lab}}}^2 + {U_{\mathrm{ref}}}^2}}$$

where:

x	is the value of the effective area determined by RISE (participant’s result)
X	is the value of the effective area determined by MIKES (reference value)
U_{lab}	is the estimated expanded uncertainty of the participant’s result (RISE)
U_{ref}	is the estimated expanded uncertainty of the reference value (MIKES)

The pressure standards of RISE and VTT MIKES will be considered as metrologically equivalent and the comparison’s performance as satisfactory when the condition of $|E_n| \leq 1$ is fulfilled for every pressure level.

9. Link to KC EURAMET.M.P-K8

The results of the present KC will be linked to the reference values of EURAMET.M.P-K8 using results of VTT MIKES obtained in the two KCs. The link will be performed at pressure points (25, 50, 75, 100, 125, 150 and 175) kPa. To do this, the results of the present KC will be linearly interpolated to the pressure points of the link.

Annex 1. Laboratory standards and measurement conditions

Manufacturer	
Measurement range in kPa	
Material of piston	
Material of cylinder	
Operation mode, free-deformation or controlled-clearance	
Zero-pressure effective area (A_0) at reference temperature in cm^2	
Relative uncertainty of A_0 in 10^{-6}	
Pressure distortion coefficient (λ) in MPa^{-1}	
Uncertainty of λ in MPa^{-1}	
Relative uncertainty of mass pieces in 10^{-6}	
Linear thermal expansion coefficient of piston (α_p) in °C^{-1}	
Linear thermal expansion coefficient of cylinder (α_c) in °C^{-1}	
Reference temperature (t_0) in °C	
Local gravity acceleration (g) in m/s^2	
Relative uncertainty of g in 10^{-6}	
Height difference between laboratory standard (LS) and TS (h), positive if LS is higher than TS, in mm	
Uncertainty of h in mm	

In addition, traceability of LS to SI units should be explained. Details of how A_0 and λ and their uncertainties were determined should be reported.

Annex 2. Results in individual measurement series

Date (period):

Average relative air humidity and its uncertainty:

Meas. no.	p_{nom} / kPa	t_{LS} / °C	t_{amb} / °C	p_{amb} / Pa	rh_{amb} / %	p / kPa	t / °C	A_p / cm^2
1	15.1							
2	35.0							
3	70.1							
4	104.9							
5	140.3							
6	175.1							
7	175.1							
8	140.3							
9	104.9							
10	70.1							
11	35.0							
12	15.1							
13	15.1							
14	35.0							
15	70.1							
16	104.9							
17	140.3							
18	175.1							
19	175.1							
20	140.3							
21	104.9							
22	70.1							
23	35.0							
24	15.1							

Kommentiert [DS5]: We usually measure the relative humitidy as well as ambient temperature and ambient pressure at everey pressure level, so I added a column for this

p_{nom} is nominal pressure,

t_{LS} is temperature of LS,

t_{amb} is temperature of ambient air,

p_{amb} is ambient pressure,

rh_{amb} is relative ambient humidity

p is pressure generated by LS at the TS reference level,

t is temperature of TS,

A_p is effective area of TS at the reference temperature of 20 °C.

The formula to calculate p must be reported.

Annex 3. Summary of all measurement series

p_{nom} / kPa	$\langle A_p \rangle$ / cm²	$\sigma(A_p)/\langle A_p \rangle$ / 10^{-6}	$u(p)/p$ / 10^{-6}	$u(t)$ / °C	$u(A_p)/\langle A_p \rangle$ / 10^{-6}
15.1					
35.0					
70.1					
104.9					
140.3					
175.1					

$\langle A_p \rangle$ is average of the A_p values measured at the same nominal pressure,
$\sigma(A_p)/\langle A_p \rangle$ is standard deviation of the A_p mean value,
$u(p)/p$ is type B uncertainty of the pressure at the reference level of TS,
$u(t)$ is uncertainty of the TS temperature,
$u(A_p)/\langle A_p \rangle$ is combined uncertainty of the A_p mean value.

In addition, a list of main uncertainty sources and their contributions to $u(A_p)$ for pressures of 15.1 kPa and 175.1 kPa must be presented.

All the uncertainties should be expressed as standard ones.