EURAMET.M.P-S9 / EURAMET 1170, LOOP2
Comparison in the negative gauge pressure range -950 to 0 hPa
FINAL REPORT

P. Otal[1], F. Boineau[1], N. Medina[2], D. Pražák[3], C. Wüthrich[4], S. Saxholm[5], W. Sabuga[6], I. Kocas[7], Y. Durgut[7]

ABSTRACT

This report gives the results of a comparison of pressure standards of seven European National Metrology institutes in the range of negative gauge pressure from -950 hPa to 0 hPa. This comparison was piloted by LNE and was carried out from January 2011 to March 2012. This work is a part of the EURAMET project 1170 and is registered as a supplementary comparison EURAMET.M.P-S9. The transfer standard used was a pressure monitor RPM4 A160Ks manufactured by DH Instruments Inc, with a resolution of 0.1 Pa. The reference values have been determined from the weighted mean of the deviations reported by the participants for each specified pressure. Seventy three of the seventy seven values (96%) reported by the laboratories agree with the reference values within the expanded uncertainties with a coverage factor k = 2.

[1] Laboratoire National de Métrologie et d'Essais, France (LNE)
[2] Centro Español de Metrología, Spain (CEM)
[3] Czech metrology institute, Czech Republic (CMI)
[4] Bundesamt für Metrologie, Switzerland (METAS)
[5] Centre for metrology and accreditation, Finland (MIKES)
[6] Physikalisch-Technische Bundesanstalt, Germany (PTB)
[7] Ulusal Metroloji Enstitüsü, Turkey (UME)

Contents

1. INTRODUCTION

This comparison is a part of the EURAMET project n° 1170 and is registered as a supplementary comparison EURAMET.M.P-S9. The project consists in two loops: loop 1 piloted by MIKES and Loop 2 piloted by LNE. The present report describes the results obtained by the participants in loop 2. The transfer standard was a pressure monitor type RPM4 A160Ks from DHI with a resolution of 0.1 Pa

The nominal pressure points for the comparison were 0 kPa, -20 kPa, -40 kPa, -60 kPa, -80 kPa, -95 kPa, -95 kPa, -80 kPa, -60 kPa, -40 kPa, -20 kPa and 0 kPa.

2. PARTICIPANTS

Seven laboratories have participated in the comparison from January 2011 to March 2012.
The list of the laboratories is given below in chronological order (Table 1):

Table 1. Comparison P1170-loop2 participants

Country	Institute	Measurement date
France	Laboratoire National d'Essais (LNE), initial	Week 1/2011 (January)
Spain	Centro Español de Metrología (CEM)	Week 3/2011 (January)
Czech Republic	Czech metrology institute (CMI)	Week 5/2011 (February)
Germany	Physikalisch-Technische Bundesanstalt (PTB)	Week 7/2011 (February)
France	LNE, intermediate	Week 9/2011 (March)
Switzerland	Bundesamt für Metrologie (METAS)	Week 11/2011 (March)
Turkey	Ulusal Metroloji Enstitüsü (UME)	Week 15/2011 (April)
Finland	Centre for metrology and accreditation (MIKES)	Week 17/2011 (April)
France	LNE, final	Week 19/2011 (April)

Customs issues occurred during delivery of the transfer standard to the Magyar Kereskedelmi Engedélyezési Hivatal (MKEH), Hungary, who finally could not participate in this comparison as initially planned.

3. LABORATORY STANDARDS AND MEASUREMENT METHODS

Each laboratory provided the pilot laboratory with the information related to the laboratory standard. Three methods emerge for negative gauge calibrations. First, the negative gauge pressure is generated under the bell jar of a pressure balance. This method is used by CEM, CMI, METAS and MIKES. The second method, used by PTB, consists in measuring the negative gauge pressure by mean of a piston-cylinder assembly mounted upside-down in a pressure balance. The last method is based on the measurement of an absolute pressure associated with the measurement of the atmospheric pressure and is used by UME and LNE.

It was recommended that each laboratory should use a data sheet reporting the data obtained at each comparison point. The measurements were performed for three cycles. The laboratories were also required to report the standard uncertainty of the deviation

3.1. Measurement standard of CEM

Reference standard: Ruska 2465 pressure balance with a low range piston cylinder assembly.
The method used by CEM for the calibration is described as follows. The gauge was connected to the bell of the pressure balance via a valve. The valve is used to isolate the gauge when the masses of the pressure balance are manipulated. In this way, one can reach the measurement target points with the gauge always in contact with nitrogen just as specified in the guide. Prior to opening the valve, for each measurement target point, we have purged the bell, at least twice, to guarantee that the fluid used is nitrogen.
On the other hand, since the high atmospheric pressure in Madrid does not allow one to generate -950 hPa, the gauge has been located in an isobaric chamber maintained at a constant pressure of –1000 hPa. The chamber at the pressure port of the pressure balance has been connected. The pressure value in the chamber is controlled with a Ruska 7000 pressure controller at 1000 hPa (absolute value). The fluid inside the chamber is air.
The pressure in the bell is regulated by means of a pressure source (nitrogen bottle), a vacuum pump, two valves and a variable volume.

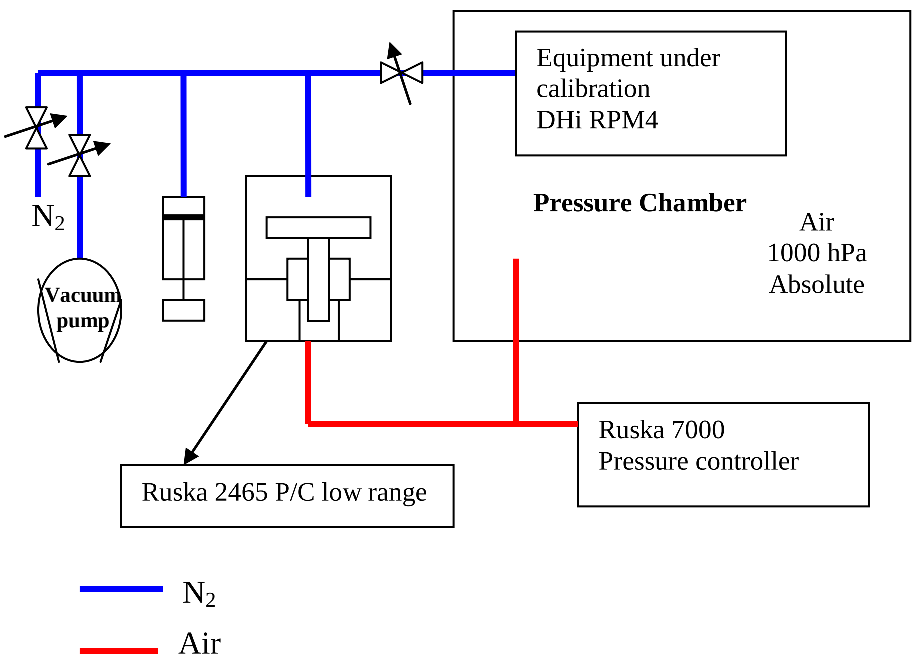

Fig.1: Schematic diagram of the experimental set-up used by CEM.

3.2. MEASUREMENT STANDARD OF CMI

The laboratory standard used was the same as for Project 1131, i.e. a DH-Instruments PG7601 pressure balance s/n 127 equipped with a piston-cylinder unit s/n 368, see [1, 2]. The effective area of this piston-cylinder unit, nominally 980 mm^2, is traceable to a piston-cylinder unit s/n 248 of the same nominal effective area which was evaluated from the dimensional measurements (latest calibration certificate 6013-KL-P0077-10 from 7th December 2010). The automated mass handling system with weight set s/n 2189 was used (latest calibration certificate 6012-KL-H052-08 from 1st December 2008).
Due to repair work being carried out on air-conditioning facilities at CMI-Brno, the temperature fluctuated by ± 1 °C around 21 °C. We decided to perform the measurements in spite of this. The time for temperature stabilisation before the first measurements was 30 hours.
The negative gauge pressure was generated in the bell jar of the pressure balance, while the atmospheric pressure acted upon the bottom of the piston, see [1]. The laboratory standard was connected to TEST(+) port. VENT and TEST(-) ports were kept open to the atmosphere. Except during zeroing, when TEST(+) and TEST(-) ports were interconnected. The head-pressure was negligible during the measurements. The pressure medium was nitrogen. Manual acquisition of data from the transfer standard was used.
Two measurements were performed – the first on 2nd February, the second from 3rd to 4th February. A non-negligible shift of the transfer standard occurred between these two measurements. We declare the results from the second measurement as the data for the comparison. However, we also enclose the results of the first measurement as information for the pilots.

The standard uncertainty of the deviation was calculated as

$$u_{\mathrm{C}} = \sqrt{u_{\mathrm{A}}{}^2 + u_{\mathrm{B}}{}^2} = \sqrt{u_{\mathrm{A}}{}^2 + u_{\mathrm{LS}}{}^2 + u_{\mathrm{res}}{}^2}\ ,$$

where
$u_{\mathrm{LS}} = u_{\mathrm{CMC}}$ = 0.005 hPa + 0.0006 % of the measured value,
$u_{\mathrm{res}} = \frac{2}{\sqrt{3}} \cdot 0.001$ hPa.

3.3. MEASUREMENT STANDARD OF LNE

LNE uses two different methods to define low and negative differential pressures. Procedure *b* was used for the EURAMET project 1170 loop 1.

Procedure *a*
The best uncertainties are achieved with two absolute pressure balances. First, equilibrium is performed between the pressure balances at a pressure close to the atmospheric pressure in order to “zero” the balances. Then, negative gauge pressures are defined by decreasing the pressure in one of them.

Procedure *b*
For daily calibrations, the same method is used with the difference that the variations of atmospheric pressure are measured with a barometer. This barometer is zeroed by comparison

with the absolute pressure balance. In addition to calculating the offset, this method allows us to reduce the uncertainty in the absolute pressure defined by the piston gauge by reducing the contributions on vacuum measurements and on the mass load corresponding to the static pressure. The only remaining mass uncertainty is the relative uncertainty on the differential mass load corresponding to the differential pressure. Concerning the reference barometer, only the resolution and the short-term stability contribute to the uncertainty in the negative gauge pressure.

The gauge pressure ΔP is calculated using the following equation:

$$\Delta P = P\mathrm{bal}_{abs} - P_0(t) - P_{0offset}\,, \qquad (1)$$

where $P\mathrm{bal}_{abs}$ is the absolute pressure defined by pressure balance, $P_0(t)$ is the current atmospheric pressure and $P_{0offset}$ is the barometer offset determined by comparison with the pressure balance.

The expanded uncertainty on the gauge pressure is estimated to be:

$U(\Delta P) = 0{,}20\ \mathrm{Pa} + 1{,}1 \cdot 10^{-5} \times |p|$.

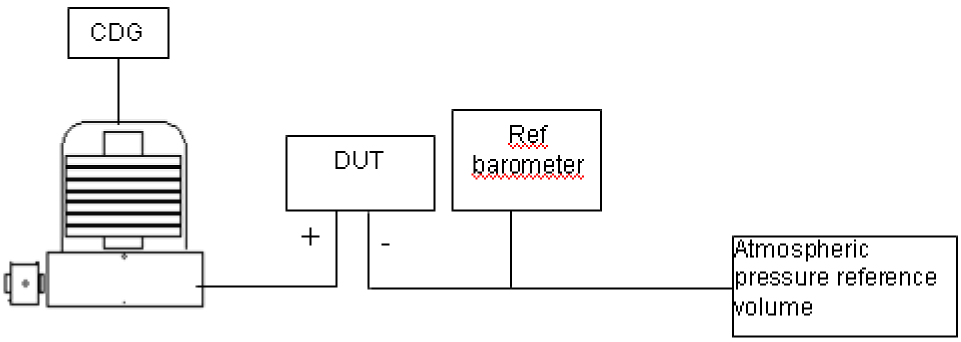

Fig.2: Set-up used for differential mode pressure measurement at LNE

The pressure balance used is a DHI PG7601 with an AMH-38 automated mass handling system. The pressure balance is equipped with a DH piston-cylinder assembly of 10 cm² nominal effective area with serial number 246. Note that for this comparison all measurements were performed in the automatic mode.

3.4. MEASUREMENT STANDARD OF METAS

<u>Experimental set-up.</u>

The depression is realised by floating a piston cylinder of an absolute pressure balance whose bell is partially evacuated, and the space under the piston is connected to the atmosphere. The pressure balance is a DHI PG-7601 (S/N 328) with a piston-cylinder of 10 cm^2 effective area (S/N 608).
The experimental set-up is depicted in Figure 2. A valve V3 is used to isolate the RPM4 from the environment during the venting and change of the mass. The Thommen Manometer is used to make a rough measure of the pressure and to monitor the opening of the valve V3 at the correct value of pressure.

Valves V1 and V2 are needle valves allowing a fine adjustment of the pressure under the bell jar. Valve V4 is normally left open. It is closed at value of -950 mbar to avoid the travelling of the piston to the full upper or lower positions. When V4 is closed, the pressure under the piston is monitored with the DPI142 and the pressure is kept within 5 Pa of actual atmospheric pressure and the rate of change of pressure is kept below 0.3 Pa/min.

Data acquisition

The data acquisition has been made through the GPIB interface of the RPM4. For each step of pressure four measurements have been made keeping the system at equilibrium. The value given in the report is the average of the four measurements.

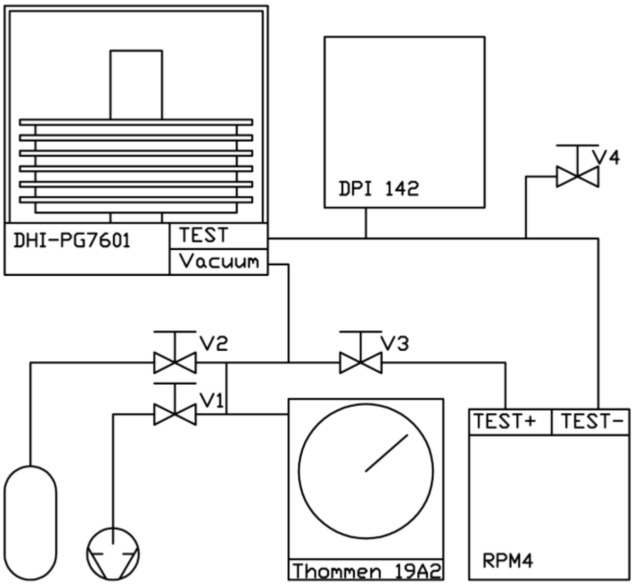

Fig. 3: Schematic diagram of the experimental set-up used by METAS.

Problem due to the altitude of the laboratory

METAS is located at an altitude of 520 m and the standard pressure is about 950 hPa. Fortunately the pressure was unexpectedly high the week of the measurement with maximum close to 970 hPa making the depression measurement at 950 hPa possible.

Temperature of the laboratory

The temperature in the laboratory is kept at 20 °C and the records during the measurement showed a maximal deviation of 0.2 °C from the reference temperature. The measurement system including the transfer standard was left all the week-end for thermal stabilization.

3.5. MEASUREMENT STANDARD OF MIKES

The measurement standard of MIKES was Fluke / DH Instruments pressure balance, type PG7607, no. 397 with piston cylinder assembly no. 451. Negative gauge pressures were generated under the bell jar of the pressure balance, test port open to atmosphere. The nominal effective area of the piston cylinder assembly is 1960 mm^2 and it was determined with dimensional measurements at MIKES and LNE as well as with cross-floating at MIKES and LNE. The effective area has been traceable to LNE for almost twenty years, but after the

dimensional determination of the effective area at MIKES showing good agreement with the results of LNE, the effective area can be considered traceable to MIKES' dimensional measurements [3].
There is only one comparison registered for the negative gauge pressure range before this project: MIKES participated to the comparison EURAMET project no. 1131 / EURAMET.M.P-S8 in 2009, range -950 hPa to +950 hPa.

3.6. MEASUREMENT STANDARD OF PTB

The PTB pressure standard (LS) used in this KC is a piston-cylinder assembly (PCA) manufactured by *Maihak*, Germany, mounted upside-down in a pressure balance manufactured by *Budenberg*, UK, fabric No. 8073/14. The properties of the pressure balance and the PCA are presented in Table 2.

Table 2. PTB pressure balance and measurement conditions

Manufacturer	*Maihak* and *Budenberg*
Measurement range of gauge pressure in kPa	-100 to -5; 5 to 100
Material of piston	steel
Material of cylinder	bronze
Operation mode	gauge
Effective area (A_p) at reference temperature and pressures 0 to 100 kPa in cm^2	5.000853
Relative uncertainty of A_p in 10^{-6}	14.4
Relative uncertainty of main mass pieces	$2 \cdot 10^{-6}$
Linear thermal expansion coefficient of PCA ($\alpha_p + \alpha_c$) in °C^{-1}	$2.9 \cdot 10^{-5}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.812533
Relative uncertainty of g in 10^{-6}	0.53
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in cm	0.18
Uncertainty of h in mm	0.5

The effective area (A_p) of this assembly is traceable to primary PCU whose zero-pressure effective area (A_0) is based on dimensional measurements. This traceability is achieved by cross-float measurements in positive gauge pressure mode, although control cross-float have also been carried in the negative gauge pressure mode against another PCA (Ruska), which have showed no difference of the effective areas in the positive and negative pressure modes.

The TS and LS were connected directly. The pressure measured in the reference level of TS (p_e) was calculated from the well-known formula:

$$p_e = -\frac{g\sum m_i\left(1-\rho_a/\rho_{m_i}\right)}{A_p\left[1+\left(\alpha_p+\alpha_c\right)\cdot\left(t-t_0\right)\right]} + g\left(\rho_f-\rho_a\right)h \text{, where} \qquad (2)$$

m_i are masses of the piston, the weight carrier and the mass pieces placed on the weight carrier,
ρ_i are densities of the parts with masses m_i,

ρ_f is density of the pressure-transmitting medium, which was air,
ρ_a is air density,
t is temperature of PCA,
and other symbols as previously defined.
The air density was calculated from the temperature, pressure and humidity, the latter taken as 60% ± 40%, of the ambient air using the equation given in [1].
The measurements were carried out in an air-conditioned room with the temperature maintained between 20.0 and 20.2 °C during all measurements. The temperature of the PCA changed between 20.0 and 20.4 °C.
The ambient pressure during the experiments was rather unstable with typical variations of about 0.05 hPa within 30 s. When TS was isolated from the ambient air by closing valves, the variations of the indicator were about ± 0.002 hPa around zero.
The uncertainty budget of the pressure in the reference level of TS is presented in Table 3.

Table 3. Type B uncertainty budgets for the minimum and maximum pressures of -950 hPa and -200 hPa. Uncertainty sources contributing less than $1 \cdot 10^{-8}$ to the relative uncertainty of p_e are not listed

Quantity	Uncertainty		$\lvert u_B(p_e)/p_e \rvert \times 10^6$ at -200 hPa	$\lvert u_B(p_e)/p_e \rvert \times 10^6$ at -950 hPa
Gravity acceleration g	$5.3 \cdot 10^{-6}$	m/s^2	0.54	0.54
Air density equation	$1.3 \cdot 10^{-4}$	rel.	0.02	0.02
Ambient pressure	$1.0 \cdot 10^{-4}$	hPa	0.02	0.02
Ambient temperature	0.5	°C	0.34	0.29
Air humidity	0.4	rel.	0.32	0.28
Height difference	0.5	mm	0.06	0.06
PT-100 in LS	0.1	°C	2.90	2.90
Temperature inhomogeneity	0.2	°C	5.80	5.80
Thermal expansion coeff.	$2.0 \cdot 10^{-6}$	$°C^{-1}$	0.82	0.36
LS verticality	1.0	mm/m	0.50	0.50
Mass of piston & weight carrier	$3.0 \cdot 10^{-5}$	kg	2.95	0.62
Density of piston & weight carrier	$2.7 \cdot 10^{-2}$	g/cm^3	0.41	0.09
Mass of main weights	$2.1 \cdot 10^{-5}$	kg	4.91	4.33
Density of main weights	$2.5 \cdot 10^{-2}$	g/cm^3	0.35	0.46
Trim mass	$1.6 \cdot 10^{-6}$	kg	1.07	0.00
Effective area	$1.44 \cdot 10^{-5}$	rel.	14.40	14.40
Combined type B uncertainty			16.88	16.42

The results of the measurements are presented in Table 3. The standard uncertainty of the deviation given there was calculated combining differences between the deviations observed in cycles 1 to 3, instability of the indication of TS and uncertainty of the reference pressure.

3.7. MEASUREMENT STANDARD OF UME

Ruska 2465 model pneumatic pressure balance was used as a reference standard. Table 4 shows the reference standard specifications. Transfer standard was connected to the reference standard directly. The calibration procedure was applied following the technical protocol of this comparison. Atmospheric pressure value was read from the DPM1 at each pressure point.

Absolute reference pressure was calculated

$$p_{\text{ref(abs)}} = \frac{(\Sigma m + V \cdot \rho_{\text{f}}) \cdot g}{A_0 \cdot (1 + \lambda \cdot p) \cdot [1 + (\alpha + \beta) \cdot (t - 20)]} + p_{\text{res}} + (\rho_{\text{f}} - \rho_{\text{a}}) \cdot g \cdot \Delta h \qquad (3)$$

Where,

m is the true mass of the floating elements,
A_0 is the effective area at atmospheric pressure and reference temperature of 20 °C,
λ is the elastic distortion coefficient,
ρ_a is the calculated ambient density,
ρ_f is the calculated fluid density,
V is piston's additional volume which is submerged into fluid and requires a correction due to fluid buoyancy,
g is the local gravity value,
p is the pressure value,
t is the temperature of piston-cylinder unit,
α is the thermal expansion coefficient of piston,
β is the thermal expansion coefficient of cylinder,
Δh is the height difference between reference and test and
p_{res} is the residual pressure.

Error values were calculated according to the formula:

$$\text{Error} = p_{\text{test}} - (p_{\text{atm}} - p_{\text{ref(abs)}}) \qquad (4)$$

Table 4. Laboratory standards and measurement conditions.

Manufacturer	Ruska, serial. no. TL-1283
Measurement range in MPa	0.0014 to 0.172
Material of piston	440 C stainless steel
Material of cylinder	tungsten carbide
Zero-pressure effective area (A_0) at reference temperature in m^2	$3.356984 \cdot 10^{-4}$
Relative uncertainty of A_0 in 10^{-6}	$1.6 \cdot 10^{-5}$ (1)
Pressure distortion coefficient (λ) in Pa^{-1}	0
Relative uncertainty of mass pieces in 10^{-6}	$6 \cdot 10^{-7}$
Linear thermal expansion coefficient of piston cylinder ($\alpha_p + \alpha_c$) in °C^{-1}	$15 \cdot 10^{-6}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.80231036
Relative uncertainty of g in m/s^2	$1.1 \cdot 10^{-7}$
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in m	0.058
Uncertainty of h in mm	2

(1)Reference standard was calibrated against the UME reference standard which is traceable to LNE (PG7601,Serial number 178/329, LNE,F014386/1)

Table 5. Uncertainty budget for pressure point -950 hPa

Quantity	Estimate	Width of distribution 2a	Divisor	Uncertainty	Sensitivity coefficient	Uncertainty contribution Pa	Variance Pa^2
Resolution	p_{test}	1.00E-2 hPa	$\sqrt{3}$	2.89E-03	1	2.89E-01	**8.3E-02**
Zero deviation(max)	$\delta p_{zero\ dev}$	3.00E-2hPa	$\sqrt{3}$	8.66E-03	1	8.66E-01	**7.5E-01**
Repeatability	$\delta p_{repeatability}$	1.25E-2hPa	$\sqrt{3}$	3.60E-03	1	3.60E-01	**1.3E-01**
Hysteresis	$\delta p_{hysteresis}$	3.15E-2hPa	$\sqrt{3}$	9.08E-03	1	9.08E-01	**8.2E-01**
Standard	$p_{reference}$	1.56E-2 hPa	2	7.80E-03	1	7.80E-01	**6.1E-01**
Temperature	t	1.00E-1 ºC	$\sqrt{3}$	2.89E-02	-1.4E-02	-4.12E-02	**1.7E-03**
Thermal linear expansion coefficient	$\alpha+\beta$	1.50E-06 1/ ºC	$\sqrt{3}$	4.33E-07	-2.5E+03	-1.07E-01	**1.1E-02**
Acceleration due to gravity	g_{mass}	1.10E-07 m/s^2	$\sqrt{3}$	3.18E-08	-97	-3.08E-04	**9.5E-08**
Determination of density difference	air	5.95E-02 kg/m^3	$\sqrt{3}$	1.72E-02	5.61E-01	9.76E-03	**9.5E-05**
	nitrogen	2.18E-03 kg/m^3	$\sqrt{3}$				
Determination of acceleration due to gravity	g_{height}	1.10E-07 m/s^2	$\sqrt{3}$	3.18E-08	-6.64E-02	-2.11E-09	**4.5E-18**
Determination of difference of altitute	h	2.00E-03m	$\sqrt{3}$	5.77E-04	-1.12E+01	-6.48E-03	**4.2E-05**
Mass	m	1.90E-06 kg	$\sqrt{3}$	5.54E-07	-7.47E+03	-4.14E-01	**1.7E-01**
DPM1	p_{atmp}	4.50E-2 hPa	2	2.25E-02	1	2.25E+00	**5.1E+00**
Reproducibility	p_{rep}	2.60E-2	$\sqrt{3}$	7.51E-3	1	7.51E-1	**5.6E-1**
						u^2 (Pa^2)	**8.2**
					Standard uncertainty (k=1)	**u (Pa)**	**2.9**
					Standard uncertainty (k=1)	**u (hPa)**	**0.03**

4. TRANSFER STANDARD

4.1. IDENTIFICATION

The transfer standard is a pressure monitor RPM4 A160Ks, serial No. 689, operating with nitrogen. It has a resolution of 0.1 Pa. The manufacturer is DH Instruments, Inc.

4.2. OPERATING PRINCIPLE

The manometer is composed of an absolute Quartz Reference Pressure Transducer (Q-RPT) and of an internal barometer.

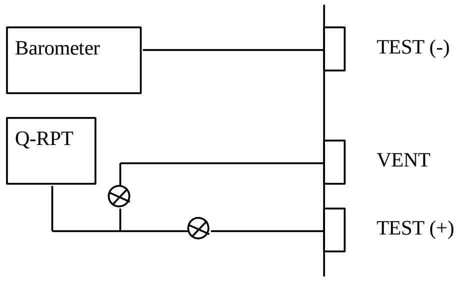

Fig. 4: RPM4 pneumatic schematics

The negative pressure indicated by the RPM4 at the instant t, PRPM (t), is expressed by the following equation:

$$P_{RPM}(\mathrm{t}) = P_Q(t) - P_Q(0) - (P_{baro}(t) - P_{baro}(0)), \tag{5}$$

with the following expressions:

$P_Q(t)$: Indication of the Q-RPT absolute pressure at the instant t

$P_Q(0)$: Indication of the Q-RPT at the atmospheric pressure at the time of zeroing execution

$P_{baro}(0)$: Indication of the barometer at the atmospheric pressure, at the time of zeroing execution

$P_{baro}(\mathrm{t})$: Indication of the barometer at the atmospheric pressure, at the instant *t*.

4.3. CALIBRATION PROCEDURE

Specific using of the transfer standard is given in the Annex A of this report.

The calibration of the transfer standard had to be performed after a warm-up time of at least twelve hours in an air conditioned room at 20 °C. It was asked to perform six pressure points in an descending ascending and then ascending sequence, repeated three times, at the following nominal gauge pressures: 0 hPa, -200 hPa, -400 hPa, -600 hPa, -800 hPa and -950 hPa of nitrogen, with a reference pressure equal to the nominal pressure with a tolerance of ± 5 hPa. The stabilisation time at each pressure point was one minute and the recording time of the transfer standard readings at each pressure level thirty seconds.

The one cycle procedure is described below:

- zeroing of the pressure module by running the “AutoZero” function of the transfer standard after connecting together the TEST(+) port with the TEST(-) port,
- feeding the transfer standard from the reference standard at the successive pressure levels down to –950 hPa, avoiding to come back to zero pressure between the points.
- applying a stabilisation time of five minutes at –950 hPa, then feeding the transfer standard from the reference standard at the successive pressure levels up to zero pressure.
- applying a stabilisation time of five minutes at zero prior to a new zeroing of the pressure module and the beginning of another cycle.

5. TRANSFER STANDARD STABILITY

The transfer standard was calibrated four times at LNE on the following dates (day/month/year): 05/01/2011; 24/02/2011; 01/07/2011 and 23/03/2012. Figure 5 shows the

stability of the transfer standard as observed at LNE during this period. In the Figure 6, the differences are represented at -950 hPa.
No drifts or trends can be identified and all the measurements agree within their standard uncertainties (Fig. 6). The standard uncertainty due to the stability of the transfer standard u_{stab} is then estimated from the maximum difference observed at LNE at –950 hPa between the first and last calibration:

$$u_{stab} = \frac{\max\left|D_{LNEend} - D_{LNEbegin}\right|}{\sqrt{3}} = 0{,}29Pa \tag{6}$$

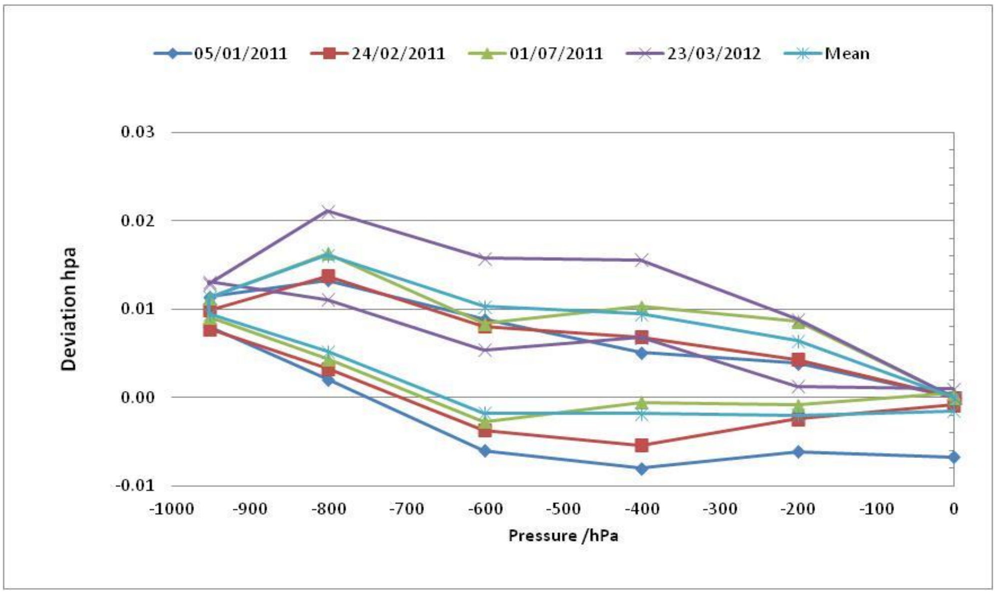

Fig. 5: Stability of the transfer standard as observed at LNE

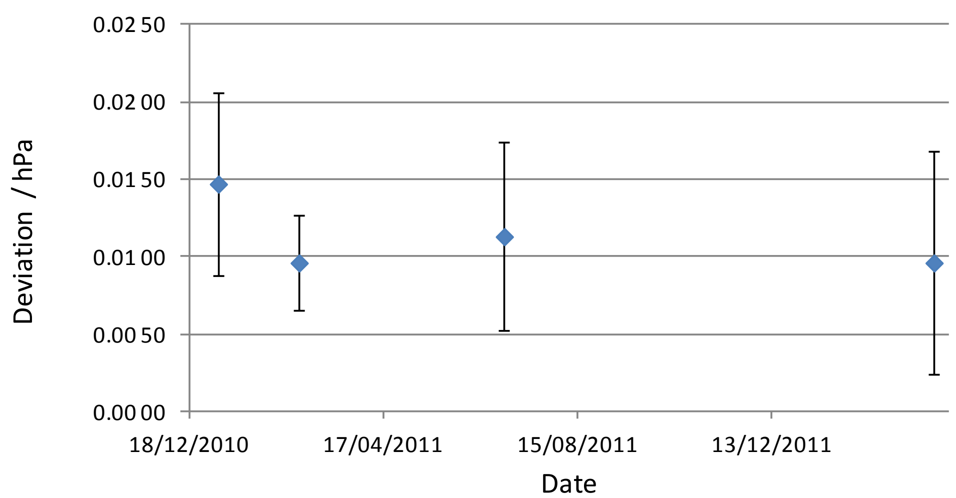

Fig. 6: Deviation of the transfer standard at –950 hPa as observed at LNE. The vertical bars represent the standard uncertainty

6. RESULTS

The mean deviations ($D_{p,i}$) measured by the participants corrected from zero and their standard uncertainties are presented in Table 6. For the pilot laboratory, only its last measurement before the beginning of the comparison was considered in order for one to attribute equal weights to all participants.

Table 6. Mean deviations ($D_{p,i}$) measured by the participants and their standard uncertainties.

	CEM	CMI	PTB	METAS	UME	MIKES	LNE
Nominal pressure / hPa	Mean deviations ($D_{p,i}$) / hPa and their standard uncertainties / hPa						
0	0.00 0 *0.01 5*	0.00 0 *0.00 1*	0.00 0 *0.00 2*	0.00 0 *0.00 7*	0.00 0 *0.02 4*	0.00 0 *0.00 8*	0.00 0 *0.00 3*
-200	0.01 9 *0.01 5*	0.01 3 *0.00 7*	0.00 3 *0.00 5*	0.00 5 *0.00 8*	0.02 1 *0.02 4*	0.00 9 *0.00 9*	0.00 6 *0.00 7*
-400	0.03 4 *0.01 7*	0.02 4 *0.00 8*	0.00 2 *0.00 8*	0.00 3 *0.00 9*	0.02 4 *0.02 4*	0.01 8 *0.01 1*	0.00 9 *0.00 8*
-600	0.04 2 *0.01 7*	0.02 6 *0.00 9*	0.00 4 *0.01 1*	0.00 9 *0.00 9*	0.02 1 *0.02 4*	0.02 4 *0.01 3*	0.01 0 *0.00 7*
-800	0.05 9 *0.01 9*	0.02 4 *0.01 0*	0.00 4 *0.01 5*	0.01 2 *0.00 7*	0.02 0 *0.02 4*	0.02 9 *0.01 4*	0.01 4 *0.00 8*
-950	0.07 6 *0.02 0*	0.02 1 *0.01 1*	0.00 2 *0.01 8*	0.02 2 *0.00 6*	0.01 9 *0.02 4*	0.03 5 *0.01 5*	0.01 0 *0.00 7*
-950	0.07 6 *0.02 0*	0.01 9 *0.01 1*	0.00 4 *0.01 7*	0.01 5 *0.00 5*	0.01 6 *0.02 4*	0.02 8 *0.01 5*	0.00 9 *0.00 8*
-800	0.04 3 *0.01 9*	0.00 2 *0.01 0*	-0.00 7 *0.01 5*	0.01 0 *0.00 9*	0.01 3 *0.02 4*	0.01 1 *0.01 4*	0.00 6 *0.00 7*
-600	0.03 0 *0.01 7*	0.00 2 *0.00 9*	-0.00 9 *0.01 1*	0.00 6 *0.00 4*	0.01 5 *0.02 4*	0.00 1 *0.01 3*	0.00 1 *0.00 7*
-400	0.01 2 *0.01 7*	0.00 3 *0.00 8*	-0.01 0 *0.00 8*	0.00 4 *0.00 6*	0.01 8 *0.02 4*	-0.00 1 *0.01 1*	0.00 0 *0.00 6*
-200	0.00 4 *0.01 5*	0.00 3 *0.00 7*	-0.01 0 *0.00 6*	0.00 0 *0.00 5*	0.01 8 *0.02 4*	-0.00 6 *0.00 9*	-0.00 1 *0.00 6*
0	-0.01 4 *0.01 5*	-0.00 1 *0.00 1*	-0.00 9 *0.00 3*	-0.00 1 *0.00 4*	-0.00 2 *0.02 4*	-0.01 6 *0.00 8*	0.00 0 *0.00 6*

7. REFERENCE VALUES

Three methods have been tested to evaluate the key comparison reference values (KCRVs): the mean, the weighted mean and the median. Table 8 and Figures 7a and 7b present the KCRVs and their associated standard uncertainties calculated at each pressure level from formulas of Table 7.

A chi-square test has been applied to carry out a consistency check of the obtained results. The test consists in comparing the values of χ^2_{obs} calculated by eq. (7) with the value of the chi-square distribution calculated for υ = 7-1= 6 degrees of freedom at probability 0.05.

$$\chi^2_{\text{obs}} = \sum_{i=1}^{7} \frac{\left(D_{p,i} - D_{p,\text{ref}}\right)^2}{u^2\left(D_{p,i}\right)} \tag{7}$$

$$\chi^2_{\text{obs}} < \chi^2(6;0.05) \tag{8}$$

with, $\chi^2(6;0.05) = 12.7$

The results presented in Table 8 show that the KCRVs calculated from the mean fail the consistency check for three pressure levels and pass it at all pressures for the weighted mean and the median with similar chi-square values. The results also indicate that the weighted mean method gives the lowest uncertainties and consequently is the one used to calculate the KCRVs for this comparison.

Table 7. Equations used for the reference values calculations D_{ref} and their standard uncertainties u(D_{ref}).

Method	D_{pref}	$u\ (D_{pref})$
Weighted mean	$D_{p,\mathrm{ref}} = \sum_{i=1}^{N} \frac{D_{p,i}}{u^2(D_{p,i})} \Big/ \sum_{i=1}^{N} \frac{1}{u^2(D_{p,i})}$	$u(D_{p,\mathrm{ref}}) = \left[\sum_{i=1}^{N} \frac{1}{u^2(D_{p,i})}\right]^{-0.5}$
Median	$D_{p,\mathrm{ref}} = \mathrm{med}(D_{p,i})$	$u(D_{\mathrm{pref}}) = \frac{1.858}{\sqrt{N-1}} \mathrm{med}\lvert D_{p,i} - D_{p,\mathrm{ref}}\rvert$

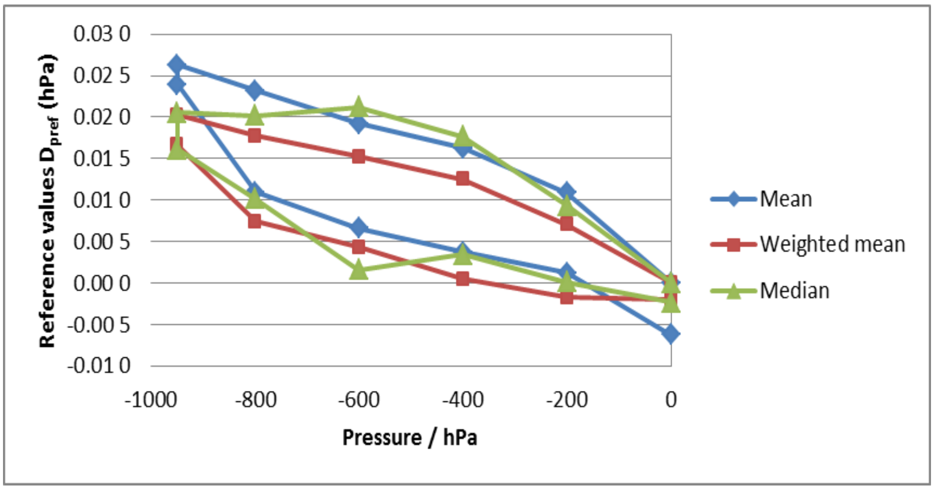

Fig.7a. KCRVs calculated as mean, weighted mean and median

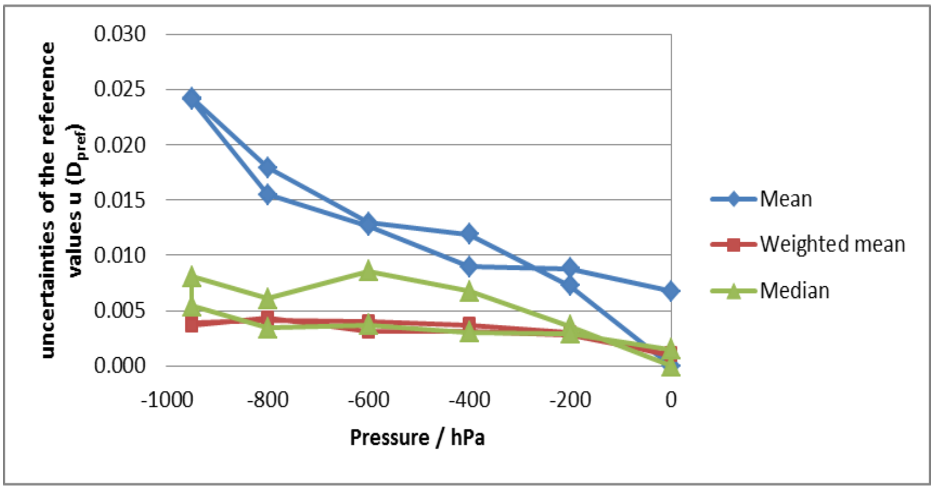

Fig.7b. Standard uncertainties of the KCRVs calculated as mean, weighted mean and median

Table 8. Reference values calculated as mean, weighted mean and median; standard uncertainties and observed chi-squared value (χ2obs)

Nominal pressure (hPa)	Mean			Weighted mean			Median		
	D_{pref} (hPa)	u (D_{pref}) (hPa)	χ^2_{obs}	D_{pref} (hPa)	u (D_{pref}) (hPa)	χ^2_{obs}	D_{pref} (hPa)	u (D_{pref}) (hPa)	χ^2_{obs}
0	0.00 0	0.00 0	0. 0	0.00 0	0.00 1	0	0.00 0	0.00 0	0
-200	0.01 1	0.00 7	4. 2	0.00 7	0.00 3	2.5	0.00 9	0.00 4	3.1
-400	0.01 6	0.01 2	8. 3	0.01 3	0.00 4	7.3	0.01 8	0.00 7	9.2
-600	0.01 9	0.01 3	7. 4	0.01 5	0.00 4	6.4	0.02 1	0.00 9	8.6
-800	0.02 3	0.01 8	9. 2	0.01 8	0.00 4	7.5	0.02 0	0.00 6	7.9
-950	0.02 6	0.02 4	***14. 5***	0.02 0	0.00 4	12	0.02 1	0.00 8	12
-950	0.02 4	0.02 4	***15. 0***	0.01 7	0.00 4	11	0.01 6	0.00 5	11
-800	0.01 1	0.01 6	5. 6	0.00 7	0.00 4	4.9	0.01 0	0.00 3	5.3
-600	0.00 7	0.01 3	5. 2	0.00 4	0.00 3	4.6	0.00 2	0.00 4	5.4
-400	0.00 4	0.00 9	4. 1	0.00 1	0.00 3	3	0.00 3	0.00 3	3.9
-200	0.00 1	0.00 9	4. 7	-0.00 2	0.00 3	3.6	0.00 0	0.00 3	4
0	-0.00 6	0.00 7	***27. 6***	-0.00 2	0.00 1	10.4	-0.00 2	0.00 2	10.6

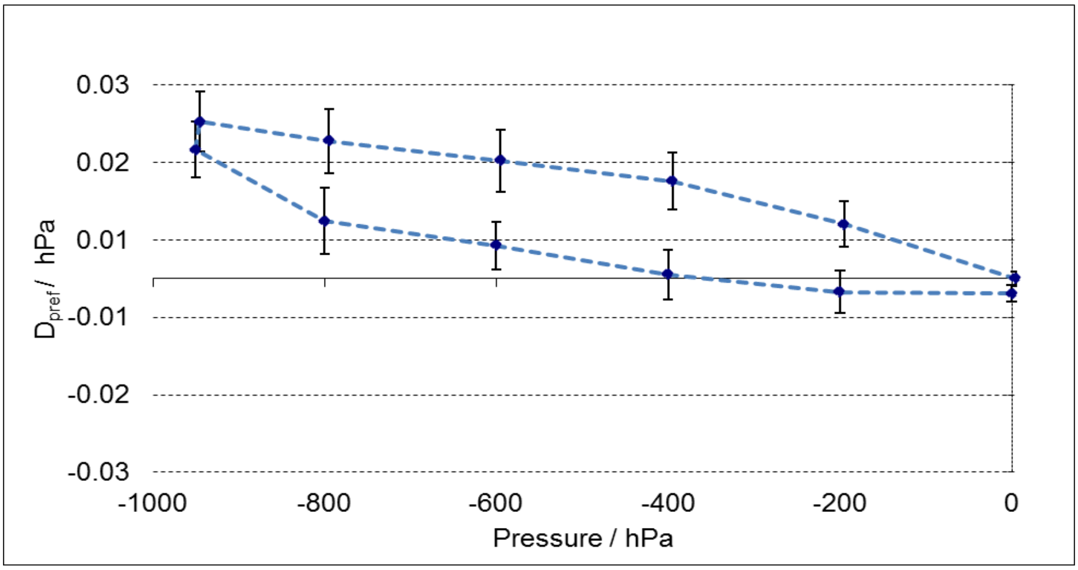

Fig. 8: Reference values D_{pref} calculated as the weighted mean. For visual clarity, the values for the decreasing pressures are slightly offset from the nominal pressure. The vertical bars represent the standard uncertainties of the reference values u (D_{pref}).

8. DEVIATIONS FROM THE REFERENCE VALUES

The differences between the deviation of the laboratories and the reference deviations $\delta_{p,j}$ ($D_{p,j}$ - D_{pref}) for each pressure and their expanded uncertainties $U(\delta_{p,j})$ are given in Table 9 and illustrated in Annex B .

$U(\delta_{p,j})$ is calculated as the combination of the uncertainty of the reference value, the uncertainty of the transfer standard stability, and the uncertainty of laboratory deviation. The formula (9) involves a difference of two variances as a consequence of mutual dependence of $D_{p,i}$ and D_{pref}. This is established by Cox in Appendix C of [4].

$$U(\delta_{p,i}) = 2\left[u^2(D_{p,i}) - u^2\left(D_{pref}\right) + u^2_{stab}\right]^{1/2} \qquad (9)$$

Table 9. Differences of the deviations of the laboratories to the reference values ($D_{p,j}$ - D_{pref}) for each pressure and their expanded uncertainties U (D_p).

Nominal pressure	CEM		CMI		PTB	
	$\delta_{p,i}$	$U(\delta_{p,i})$	$\delta_{p,i}$	$U(\delta_{p,i})$	$\delta_{p,i}$	$U(\delta_{p,i})$
hPa	hPa	*hPa*	hPa	*hPa*	hPa	hPa
0	0.00 0	0.03 0	0.00 0	0.00 6	0.00 0	0.00 7
-200	0.01 2	0.02 9	0.00 6	0.01 5	-0.00 4	0.01 0
-400	0.02 1	0.03 4	0.01 2	0.01 5	-0.01 1	0.01 5
-600	0.02 6	0.03 3	0.01 0	0.01 8	-0.01 1	0.02 2
-800	0.04 2	0.03 8	0.00 6	0.01 9	-0.01 4	0.02 9
-950	0.05 6	0.04 0	0.00 0	0.02 1	-0.01 8	0.03 6
-950	0.05 9	0.04 0	0.00 3	0.02 1	-0.01 2	0.03 4
-800	0.03 5	0.03 8	-0.00 6	0.01 9	-0.01 5	0.02 8
-600	0.02 6	0.03 4	-0.00 3	0.01 7	-0.01 3	0.02 2
-400	0.01 1	0.03 4	0.00 3	0.01 6	-0.01 0	0.01 6
-200	0.00 6	0.02 9	0.00 5	0.01 3	-0.00 8	0.01 2
0	-0.01 2	0.03 0	0.00 1	0.00 6	-0.00 7	0.00 8

Nominal pressure	METAS		UME		MIKES		LNE	
	$\delta_{p,i}$	$U(\delta_{p,i})$	$\delta_{p,i}$	$U(\delta_{p,i})$	$\delta_{p,i}$	$U(\delta_{p,i})$	$\delta_{p,i}$	$U(\delta_{p,i})$
hPa	hPa	hPa	hPa	hPa	hPa	hPa	hPa	hPa
0	0.00 0	0.01 5	0.00 0	0.04 8	0.00 0	0.01 7	0.00 0	0.00 8
-200	-0.00 2	0.01 5	0.01 4	0.04 8	0.00 2	0.01 8	-0.00 1	0.01 5
-400	-0.00 9	0.01 7	0.01 2	0.04 8	0.00 5	0.02 2	-0.00 4	0.01 6
-600	-0.00 7	0.01 6	0.00 6	0.04 8	0.00 8	0.02 5	-0.00 5	0.01 4
-800	-0.00 6	0.01 3	0.00 2	0.04 8	0.01 1	0.02 7	-0.00 4	0.01 6
-950	0.00 2	0.01 1	-0.00 1	0.04 8	0.01 5	0.03 0	-0.01 0	0.01 3
-950	-0.00 1	0.00 9	-0.00 1	0.04 8	0.01 1	0.03 0	-0.00 8	0.01 5
-800	0.00 3	0.01 7	0.00 5	0.04 8	0.00 3	0.02 7	-0.00 2	0.01 3
-600	0.00 2	0.00 8	0.01 1	0.04 8	-0.00 3	0.02 6	-0.00 4	0.01 4
-400	0.00 3	0.01 2	0.01 7	0.04 8	-0.00 1	0.02 2	-0.00 1	0.01 2
-200	0.00 2	0.01 1	0.01 9	0.04 8	-0.00 4	0.01 8	0.00 1	0.01 2
0	0.00 1	0.00 9	0.00 0	0.04 8	-0.01 4	0.01 7	0.00 2	0.01 3

The degrees of equivalence E_n quantified by Eq.(10) are presented in Table 10 for each pressure and laboratory.

$$E_n=\delta_{p,j} / U(\delta_{p,j}) \qquad (10)$$

Table 10. Degrees of equivalence E_n for each pressure and each laboratory

Nominal pressure hPa	CEM	CMI	PTB	METAS	UME	MIKES	LNE
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
-200	0.42	0.41	-0.39	-0.16	0.30	0.13	-0.10
-400	0.61	0.80	-0.69	-0.53	0.24	0.24	-0.23
-600	0.80	0.60	-0.53	-0.40	0.13	0.33	-0.39
-800	1.10	0.31	-0.49	-0.44	0.05	0.41	-0.24
-950	1.41	0.02	-0.52	0.16	-0.03	0.51	-0.76
-950	1.50	0.12	-0.37	-0.13	-0.01	0.37	-0.52
-800	0.93	-0.29	-0.51	0.16	0.11	0.13	-0.13
-600	0.77	-0.15	-0.58	0.26	0.22	-0.13	-0.26
-400	0.33	0.19	-0.66	0.25	0.36	-0.05	-0.07
-200	0.20	0.37	-0.67	0.17	0.41	-0.24	0.10
0	-0.40	0.21	-0.88	0.10	-0.01	-0.85	0.13

9. CONCLUSION

Seventy-four of the seventy-seven values (96%) reported by the laboratories agree with the reference values within the expanded uncertainties with a coverage factor k = 2. The results can be compared directly to the EURAMET M.P-S9 comparison loop 1. The comparison shows the performance of three very different methods in negative gauge. Although the results can be considered satisfactory for this loop, a comparison with a transfer standard with better metrological features in terms of short-term stability and resolution will allow a deeper understanding of this widely used pressure range.

10. REFERENCES

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[3] SAXHOLM, S., HEMMING, B., HEINONEN, M., OTAL, P., RANTANEN, M., ESALA, V.-P., Lassila, A.: Traceability of the pressure balance effective area at MIKES. PTB-Mitteilungen, 121, 2011, Heft 3.

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11. ANNEX A – PROCEDURE TO CARRY OUT THE TRANSFER STANDARD

Data acquisition

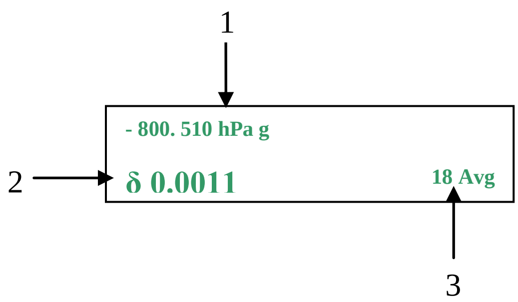
Data acquisition of the transfer standard could be performed automatically via RS232 port or manually. The two procedures are described in the paragraphs below.

Manual acquisition

The **average** DISPLAY should be active with an averaging time period of 30 seconds:
To access the Average DISPLAY, press **[DISPLAY], <1Avg>.**
Edit the averaging time period, 30 s.

Averaging Period: **30 s**

Press **[ENT]** and returns to the main run
The Average DISPLAY is active the main run screen is:



1. Average measured over the last completed averaging period.
2. Standard deviation of the last completed averaging period.
3. Countdown in seconds until completion of ongoing averaging period.

Automatic acquisition

The configuration parameters of the RS232 port are described below

Baud Rate	:	9200 à 19200
Parity	:	EVEN
Data bits	:	7
Stop Bit	:	1

Installation procedure of the transfer standard

1- Ensure that the transfer standard will always be isolated from pressures out of its operating range –950 - +600 hPa.

2- Connect the manometer to the 230 VA sector, switch on the device and wait for the initialisation.

3- Configure the transfer standard in « GAUGE » mode, press the [MODE] function key and select <gauge> mode.

4- Connect together the TEST(+) port with the port TEST(-) and run “AutoZero” function, press [AutoZ]. The value indicated should be zero (± 1 Pa)

5- Leave the TEST(-) port unobstructed or connect it to the reference standard’s port if available. The reference pressure should be within [950hPa – 1050 hPa].

6- Before connecting your pressure standard, please ensure that its pneumatic circuits are at atmospheric pressure.

7- Finally, connect your pressure standard output to the TEST(+) port. The port VENT must be always opened to the atmosphere.

8- If the message «SDS closed» is flashing, please press the button « SDS » and then answer « YES » to the question « Defeat SDS ». The opening of the SDS will limit the damages in case of overpressure. In any case of overpressure, contact the pilot laboratory.

12. ANNEX B. DIFFERENCE BETWEEN THE DEVIATION OF THE LABORATORIES AND THE REFERENCE DEVIATION $\delta_{P,J}$ ($D_{P,J}$-D_{PREF})