Draft

EURAMET Key and Supplementary Comparison of National Pressure Standards in the Range 1 Pa to 15 kPa of Absolute and Gauge Pressure (EURAMET.M.P-K4.2010)

Technical Protocol

Pilot laboratory:

Czech Metrology Institute (CMI), Czech Republic

1. Introduction

This comparison was initially EURAMET Project No. 1047. At the EURAMET TCM meeting held in Malta in March 2009, it was agreed to change it into a key/supplementary comparison in the range from 1 Pa to 15 kPa of absolute and gauge pressure. The comparison results in absolute mode will serve as a EURAMET Key Comparison linked to CCM.P-K4 via PTB. The comparison results in gauge mode will serve as a supplementary comparison. A participant can choose to take part in both gauge and absolute pressure or only in one of these ranges. This comparison is aimed to state the equivalence of the national low pressure standards. The Czech Metrology Institute (CMI) was agreed to be a pilot laboratory in this and provides a transfer standard (TS) for it. The staff of CMI will visit other laboratories for the subsequent bilateral comparisons resulting in a star comparison. This Technical Protocol specifies the procedures to be followed in the comparison and has been prepared in accordance with the Guidelines for CIPM Key Comparisons.

2. Participants

For budgetary and practical reasons, the number of the participants had to be reduced (compares to the number of participants of Project No. 1047) to five, all from European Union plus Schengen states.

The list of the participating laboratories and the names of the contact persons in the participating laboratories with their phone and fax numbers as well as their e-mail addresses is given below.

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3. Time table

The measurements with the transfer standard should be preferably performed in accordance with the schedule given below. However due to the possible problems of transport of a large transfer-standard together with staff, some changes or delays are to be expected.

Measurement time	Institute	Range
IX 2008	MIKES	both
X 2010	INRIM	gauge only
spring? 2011	PTB	both
spring/summer? 2011	LNE	absolute only

4. Transportation

Transportation of TS to and from each participant (except LNE) will be performed by a CMI staff. Transportation of the standard of LNE to and from CMI will be performed by LNE staff. Therefore there are no instructions according to transportation of TS, its unpacking, unpacking etc.

5. Costs

Each participant bears the costs for its measurements. The insurance of TS during travel abroad will be paid by CMI. The insurance of laboratory standard (LS) of LNE during transport to CMI will be paid by LNE.

6. Transfer standard

The transfer standard is a digital non-rotating piston gauge FPG8601 manufactured by DH-Instruments, identified by serial number 107, in combination with a reference vacuum gauge 627B1TDD1B manufactured by MKS Instruments, identified by serial number 000754687.
A capacitance diaphragm gauge (CDG) will serve as a zero indicator and as a separator between TS and LS.

The list of equipment transported together with TS:

1. TS - FPG8601, serial number 107, together with a reference vacuum gauge 627B1TDD1B, serial number 000754687.
2. VLPC control unit of TS plus a notebook with controlling software.
3. A differential CDG serving as a zero indicator with the set of valves that is also provided by the pilot lab. This CDG is capable of reading via PC with installed FPG TOOLS.
4. A trapeze for checking the linearity of mass comparator.

The list of necessary equipment disposable at the visited laboratory:

1. A spinning rotor gauge (SRG) or an ion vacuum gauge for zeroing the reference vacuum gauges.
2. Two turbomolecular pumps.
3. A set of weights for checking the linearity of mass comparator.

7. Measurement procedures

The nominal pressure points p_n will be 1 Pa (optional), 3 Pa, 10 Pa, 30 Pa, 100 Pa, 300 Pa, 1 kPa, 3 kPa, 10 kPa and 15 kPa both absolute and gauge. Measurements will be made in 2 cycles for absolute pressure and 2 cycles for gauge pressure. Each cycle must be performed different day, although one absolute and one gauge cycle may appear the same day. Thus, 40 (36) measurements shall be performed in total. Those laboratories that chose comparison in only either gauge or either absolute mode will do 20 (18) measurements in total. *(Note: With the vacuum pumps of MIKES there cannot be performed the measurement of the pressures 1 Pa and 3 Pa in the same cycle as the higher absolute pressures).*
The pressure transmitting medium shall be dry nitrogen (dry is the gas entering VLPC unit, however the FPG adjusts humidity of the gas to approximately 50% via its internal reservoir of water).

The following measurement procedure is applicable for comparison of two FPGs. If the LS will be based on another principle (as expected for comparison with PTB) it cannot be used without some modifications.

Both TS and LS should have valid calibration of their internal sensors of temperature, pressure and humidity.

The comparison measurements should be performed in a laboratory free of vibrations, with air conditioning able to ensure ambient temperature stable within 0.2 °C during one measurement point and with minimized disturbances due to instabilities of atmospheric pressure (doors etc.). However the last condition is relevant to gauge measurements only.

The preparation of TS for use will be done in assistance of the CMI staff. TS is recommended to be located close to the laboratory's reference standard to keep the pressure line between the two instruments as short as possible.

To minimize uncertainties in pressure measurement, the height difference between the reference levels of TS and LS will be kept as low as possible. The height difference will be measured with an uncertainty of about 1 mm.

Horizontality of TS and LS should be carefully checked with the built-in spirit level.

Both TS and LS should be switched on at least 24 h before the start of the comparison.

Linearity of mass comparator of both TS and LS will be checked before the start of the comparison measurements.

Both TS and LS must have stable zero indication and internal calibration (using internal standard mass).

The comparison measurements will be performed using 1 torr CDG as the zero indicator. This CDG will be provided by the pilot laboratory with an actual calibration valid for both plus and minus indications with emphasis on the range around zero (between ±3 Pa), however during measurement the CDG indication should be kept as near to zero as possible. The zero indication will be corrected by this calibration results. A bypass line with a valve should connect the both sides of the zero pressure indicator to control its zero pressure reading.

The zero indicating CDG will be heated during absolute mode measurements, but not heated (but long term stabilized) during gauge mode measurements.

The CDG must be connected to TS and LS with tubings (bellows) that are as similar to each other as possible concerning their diameters and volumes. The valve closing

by-pass of this CDG should not induce large changes of pressure. The leak checking of the by-pass must be performed.

Before the start of the comparison measurements both TS and LS will be zeroed and then calibrated internally. Check of the internal calibration will be repeated every four hours.

Then both instruments will be zeroed again and the zero will be checked and read. Then the isolation valve between LS and TS is closed (but with open CDG by-pass valve). Only after this, the target nominal pressure will be set by an FPG that is not connected to CDG at the moment. Then the generated target pressure is set by another FPG filling CDG. After stabilization the zero of the CDG will be read by open by-pass valve. Then the by-pass valve will be closed and isolating valve open.

After stabilization of reading, at least 5 points are measured by averaging outputs of FPGs and CDG during at least 1 min. (Measurement should cover at least 5 regulation cycles.)

The pressure points set on TS and LS should be such that the indication of CDG would oscillate near its zero.

After measuring a point a check of CDG zero drift (if stable this checking needs not to be performed after every point) and check of TS and LS zero drifts. The results will be corrected for this drift.

For absolute mode measurements; it is recommended to check (calibrate) the reference vacuum gauges by a vacuum meter (an SRG or another suitable vacuum gauge mounted between reference ports of TS and LS, the same gauge for zero checking of both TS and LS) at real working reference pressure value.

By gauge mode measurements, it is recommended to let both reference ports of TS and LS fully open to atmosphere, i.e. nothing connected to KC16 flanges. (It is not recommended to connect the reference ports of TS and LS by any tubing).

These recommendations are based on the experience and large amount of experimental data acquired during six weeks research of FPGs of CMI and MIKES at CMI in September and October 2008. We recommend using these data to this comparison.

8. Report of results

If p_{TS} denotes the pressure as determined by the TS and p_{CDG} the pressure reading of the CDG, for gauge mode and for absolute mode and nominal pressure p_n = 100 Pa

and higher, where no thermal transpiration effect exists, the predicted pressure in the LS is given by:

$$(p_{LS})_{predicted} = p_{TS} - (p_{CDG} - (p_0 + p'_0)/2) \cdot C_{CDG},$$

where

p_0 zero reading of the CDG before the measurement (about 318 K),

p'_0 zero reading of the CDG after the measurement,

C_{CDG} calibration factor of the CDG.

For nominal pressures lower than 100 Pa in absolute mode, the pressure in the LS can be predicted as:

$$(p_{LS})_{predicted} = \frac{p_{TS}\left[1 + f(p_t)\left(\sqrt{\frac{T_{CDG}}{T_{TS}}} - 1\right)\right] - (p_{CDG} - (p_0 + p'_0)/2) \cdot C_{CDG}}{1 + f(p_t)\left(\sqrt{\frac{T_{CDG}}{T_{LS}}} - 1\right)},$$

where

T_{CDG} absolute temperature of the CDG,

T_{TS} absolute temperature of the TS,

T_{LS} absolute temperature of the LS,

$f(p_t)$ thermal transpiration correction factor (0…1).

For each measurement i (i = 1…5) on day j (j = 1,2) at the defined target pressure the difference d_{ij} between the two systems will be calculated as:

$$d_{ij} = p_{LSij} - (p_{LSij})_{predicted}$$

For each nominal pressure a single value of d will be calculated by taking the mean of all measurements of the two days:

$$d = \frac{1}{10} \cdot \sum_{j=1}^{2} \sum_{i=1}^{5} d_{ij}$$

Each laboratory should provide the pilot laboratory with the information concerning its own pressure standard, including its traceability and the estimation of its uncertainty, as well as any other useful additional information. Uncertainties shall be given as standard uncertainty (k=1) according to GUM.

Reports with the results of the measurements should be sent to the pilot laboratory by e-mail to the address

dprazak@cmi.cz

within six weeks after finishing the measurements.