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Evaluation of supplementary comparison EURAMET.M.P-S14 in the range 50 MPa to 1 GPa of hydraulic gauge pressure

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ABSTRACT
In this work, we present a mathematical procedure to evaluate a hydraulic gauge pressure comparison in the range to 1 GPa which was piloted by the Physikalisch-Technische Bundesanstalt. The comparison used a transfer standard consisting of two series of modern high-pressure transducers, i.e. eight pressure transducers in total. This set of parallel arranged transducers should ensure the reliability of the transfer standard at high pressures and provides rich data for testing the performance of modern high-pressure transducers. The analysis of the comparison results was based on the evaluation of the individual measurement deviations of these transducers with respect to the laboratory standards, whereas the corresponding comparison reference values and their uncertainty were determined separately at each pressure point and pressure transducer. All these results were summarized to derive the degree of equivalence for each laboratory at each pressure. The degree of equivalence was found to be consistent for all laboratories at almost all pressures.

Section: RESEARCH PAPER

Keywords: high pressure; comparison; pressure transducers

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1. INTRODUCTION

PTB organized a supplementary comparison between five national metrology institutes (NMIs) from EURAMET – PTB (Germany), SMU (Slovakia), CMI (Czech Republic), LNE (France) and METAS (Switzerland) – in the pressure range up to 1.0 GPa. This comparison was embedded into the joint research project IND03 “High pressure metrology for industrial applications” which was carried out within the European Metrology Research Programme. The objective of this project was to create a primary pressure standard for the range up to 1.6 GPa using pressure multipliers [1], and besides this, to develop a transfer standard (TS) for pressures up to 1.5 GPa on the basis of modern high-pressure transducers. It was aimed to determine the applicability of high-pressure transducers as a TS for pressure comparisons, i.e. testing of typical parameters like drift, hysteresis, sensitivity, repeatability, long-term stability and load cycling effects, see [2]. Due to the structure of a TS consisting of a set of eight different and parallel pressure transducers with individual behaviour, the evaluation of the degree of equivalence of the participants required a special procedure for the analysis of the rich data.

It was agreed that PTB would be the pilot laboratory. The analysis of data on the basis of the results from the participants was performed by the pilot institute, hence providing uniform treatment for all participants.

2. ORGANIZATION OF THE COMPARISON

Information about laboratory standards (LSs) including their piston-cylinder units (PCUs) is given in Table 1.

Table 1. Laboratory standards of the NMIs.

NMI	Type of reference standard	Relative standard uncertainty in ppm at 0.5 GPa and $p_{\max}$
#1	Pressure balance: Harwood, modified, PCU: Desgranges et Huot, controlled clearance	29 at 0.5 GPa 52 at 1.0 GPa
#2	Pressure balance, PCU: Desgranges et Huot, controlled clearance	26 at 0.5 GPa 51 at 1.0 GPa
#3	Pressure balance, PCU: DHI, free deformation	100 at 0.5 GPa
	Pressure multiplier: Desgranges et Huot, controlled clearance	145 at 0.8 GPa [1]
#4	Pressure balance, PCU: DHI/Fluke, free deformation	32 at 0.5 GPa
#5	Pressure balance, PCU: own production, free deformation	200 at 0.5 GPa

[1] NMI #3 could reach only 0.8 GPa due to leakage problems. Its measurements in the 1 GPa range were therefore withdrawn.

The transfer standard was calibrated at PTB at the beginning and end of the comparison. The participants had minor technical problems during the comparison, but the TS did not significantly change its metrological properties, and the comparison was completed successfully.

3. TRANSFER STANDARD AND CALIBRATION

PTB developed and provided the transfer standard for the comparison. It consisted of eight high-pressure transducers of two manufacturers: type A (foil strain gauge, transducers S1 to S4) and type B (thin layer strain gauge, transducers S5 to S8), see Figure 1. Thus, from each manufacturer, there were four pressure transducers with different pressure ranges: one with (0 – 0.5) GPa, one with (0 – 1.0) GPa and two with (0 – 1.5) GPa pressure ranges.

The whole device was enclosed in an aluminium box with a Plexiglas window. Additionally, for the read-out of the type A transducers, a measuring amplifier (range 2.5 mV/V, resolution 10^{-6} mV/V - 1 ppmFS) with several measuring channels was circulated as well, whereas the type B transducers could be read out directly via a USB connection.

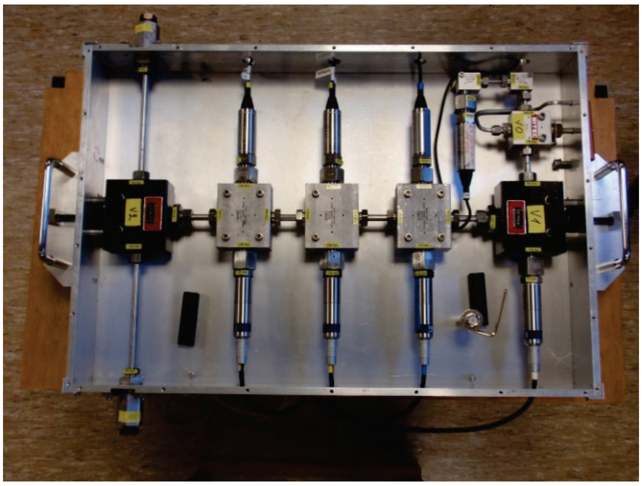

Figure 1. Image of the transfer standard. For more details see [2].

Measurements were carried out in two different pressure ranges, up to 0.5 GPa and up to 1 GPa. Laboratories capable of measuring up to 0.5 GPa, i.e. #4 and #5, performed measurements only in this measurement range. Laboratories with a measurement capability up to 1 GPa, i.e. #1 to #3, performed the measurements two times, in the range of 0.5 GPa and, additionally, in the 1 GPa range. Due to their leakage problems and the associated piston fall rates which were too high at pressures above 500 MPa, NMI #3 decided to withdraw its measurements in the 1 GPa range. Each laboratory performed four measurement cycles in each pressure range.

After preloading, one complete measurement cycle in each pressure range consisted of 22 measurements – 11 for ascending and 11 for descending pressures. The pressures generated by the LSs had to deviate from the nominal pressures by less than 0.1%. At each pressure point, the TS had to be in pressure equilibrium with the LSs. The difference between the TS output (converted to pressure) and the LS pressure was used as a measure for the comparison. Software was provided for readings of all eight transducers. After achieving equilibrium, seven readings from each of the eight transducers were taken. From the seven readings, the average and its standard deviation (σ) were calculated for each transducer at each pressure point by each participant and were reported together with the respective pressure value of each participant's laboratory standard and its standard uncertainty at this pressure point.

4. COMPARISON RESULTS

4.1. Data processing

For each participant each pressure transducer in each measurement cycle, a zero-pressure correction was applied. The result of each participant obtained in cycle c (x_c) was expressed as the difference between the pressures of the TS and the laboratory standard:

$$x_c = p_{\mathrm{TS}} - p_{\mathrm{LS}} \tag{1}$$

The average (x) and its standard deviation ($\sigma(x)$) from the results in four cycles were calculated:

$$x = \sum_{c=1}^{4} x_c / 4, \quad \sigma(x) = \left[\sum_{c=1}^{4} (x_c - x)^2 / 3 \right]^{0.5} . \tag{2}$$

The analysis of the PTB results obtained at the beginning and the end of the comparison revealed that the changes of the eight pressure transducers were rather irregular, so that an assumption of a linear change in properties of the transducers can hardly be justified. Therefore, the difference in the PTB results from 2013 and 2014 was handled as an additional uncertainty contribution due to the TS drift, u_{Drift} :

$$u_{\mathrm{Drift}} = \left(x_{\mathrm{PTB,2014}} - x_{\mathrm{PTB,2013}} \right) / \left(2\sqrt{3} \right) . \tag{3}$$

The uncertainty of the transfer standard, u_{TS}, was determined for each participant, each pressure transducer in each measurement cycle and for each pressure, taking four components into account:

- the standard deviation of the four x_c values, $\sigma(x)$, calculated according to (2),
- uncertainty due to the TS drift, u_{Drift} , as defined above by (3),

- the zero deviation of the TS, u_{ZeroDev} :

$$u_{\text{ZeroDev}} = \max\left\{\text{abs}\left[x_1(p) - x_1(p_{\text{up}} = 0)\right], \ldots, \text{abs}\left[x_4(p) - x_4(p_{\text{up}} = 0)\right]\right\} / \left(2\sqrt{3}\right) \quad (4)$$

- and the instability of the TS, u_{Instab} , taken as the resolution or the fluctuation of readings,

$$u_{\text{Instab}} = \max\left(u_{\text{Resol}}, u_{\text{Fluct}}\right) \quad (5)$$

with the resulting uncertainty contribution due to TS

$$u_{\text{TS}} = \left[\sigma^2(x) + u^2_{\text{ZeroDev}} + u^2_{\text{Instab}} + u^2_{\text{Drift}}\right]^{0.5} . \quad (6)$$

The uncertainty of the TS pressure transducers measured by the laboratories especially in the 1 GPa range was observed to be very different. This was caused, to high extent, by very different fluctuations of readings.

4.2. Deriving the degrees of equivalence

The following considerations were made to account for the special setup of the TS:

- The TS comprises eight pressure transducers. Each of the eight transducers has different properties at increasing and decreasing pressures. The same pressure transducer has different properties when used in 0.5 GPa and 1 GPa measurements. It is thus only possible to directly compare results of laboratories which were obtained for the same pressure transducer, in the same pressure change direction and in the same maximum pressure comparison to determine *one* degree of equivalence at each pressure from the numerous "degrees of equivalence" available at the same pressure.
- At each nominal pressure value, 16 comparison reference values (CRVs), (i.e. 8 pressure transducers, 2 pressure change directions) can be generated for the 0.5 GPa, and 12 CRVs (i.e. 6 pressure transducers, 2 pressure change directions) for the 1 GPa measurements. As some nominal pressures of the 0.5 GPa and 1 GPa measurements coincide, the number of CRVs can be reduced at certain pressures.
- For each of these CRVs, a participant deviation from it and the uncertainty of this deviation can be calculated, which represents the degree of equivalence for this particular CRV.
- Results of the NMIs were considered to be independent. The method by Cox [3] was used (uncertainty weighted mean) to calculate each particular CRV R_j.
- Results of an NMI obtained in n measurements at the same pressure were considered as fully correlated. This is justified by the observation that the performance of all eight transducers in 0.5 GPa and six pressure transducers in 1 GPa measurements, e.g. fluctuations of readings, is strongly correlated.

With the following designations and definitions,

- x_{ij} – result of NMI i for measurement j according to (2),
- $i = 1 \ldots N$ with N being equal to 5 at pressures (50- 500), and 2 at pressures (600-1000) MPa,
- $j = 1 \ldots n$, $n = 16, 28, 16, \ldots, 12$ at pressures of $p = (50, 100, 150, \ldots, 1000)$ MPa, as described above,
- $u(x_{ij})$ – uncertainty of x_{ij} with $u(x_{ij}) = \left[u^2_{\text{TS}} + u^2_{\text{LS}}\right]^{0.5}$, u_{TS} according to (6) with u_{LS} as the uncertainty of LS,
- $u_{ij} = u^2(x_{ij})$,
- R_j – CRV of measurement j, and
- $u(R_j)$ – uncertainty of R_j,

one obtains the expressions below:

$$R_j = \sum_{i=1}^{N}\left(x_{ij}/u_{ij}\right) \Big/ \sum_{i=1}^{N}\left(1/u_{ij}\right), \quad (7)$$

$$V_j = u^2\left(R_j\right) = 1 \Big/ \sum_{i=1}^{N}\left(1/u_{ij}\right). \quad (8)$$

Accordingly, the deviation of the result j of laboratory i from the CRV and its corresponding uncertainty are:

$$d_{ij} = x_{ij} - R_j , \quad (9)$$

$$u\left(d_{ij}\right) = \left(u_{ij} - V_j\right)^{0.5} . \quad (10)$$

Equation 9 (and equation 10 resp.) represents the deviation of each NMI at each pressure from each of the n reference values R_j with its associated uncertainty. A calculation of a mean CRV of n R_j values is not reasonable because each of them is individual in respect to the individual pressure transducers and measurement conditions. However, deviations d_{ij} and their uncertainties $u(d_{ij})$ can be processed to determine a mean deviation $\bar{d}_i$ with its uncertainty $u(\bar{d}_i)$. An analysis of the uncertainty contributions revealed a very high variability in the TS uncertainties not only between the NMIs but also for the same NMI in different measurements. For this reason, to minimize the uncertainty of the mean deviation, it was calculated as a weighted mean:

$$\bar{d}_i = \sum_{j=1}^{N}\left[d_{ij}/\left(u_{ij} - V_j\right)\right] \Big/ \sum_{j=1}^{N}\left[1/\left(u_{ij} - V_j\right)\right], \quad (11)$$

$$u\left(\bar{d}_i\right) = \sum_{j=1}^{N}\left[1/\left(u_{ij} - V_j\right)^{0.5}\right] \Big/ \sum_{j=1}^{N}\left[1/\left(u_{ij} - V_j\right)\right]. \quad (12)$$

From equation 7, one can straightforwardly derive the following condition:

$$\sum_{i=1}^{N}\left[d_i/u^2\left(d_i\right)\right] \Big/ \sum_{i=1}^{N}\left[1/u^2\left(d_i\right)\right] = 0 . \quad (13)$$

However, the situation is different for the actual case of a series of several transducers. It can be shown that the $\bar{d}_i$ values may be shifted by a value Δ. This depends on the results obtained by an NMI in different measurements at the same nominal pressure, in particular when some results lie below and some above R_j and the uncertainties of the single measurements σ_c of the same NMI and/or of different NMIs strongly vary, as was the case in this comparison. The deviations corrected for the shift Δ are then given as

$$\Delta = -\sum_{i=1}^{N}\left[\bar{d}_i/u^2\left(\bar{d}_i\right)\right] \Big/ \sum_{i=1}^{N}\left[1/u^2\left(\bar{d}_i\right)\right] \text{ and} \quad (14)$$

$$d_i = \bar{d}_i + \Delta \quad (15)$$

with $u(d_i) = u(\bar{d}_i)$ and the expanded uncertainties $U(d_i)$ taken as $U(d_i) = 2 \times u(d_i)$.

4.3. Results

From the raw data of the comparison presented in [2], one can derive the degrees of equivalence according to equation 15. As a result, it can be found that most results are in agreement with each other. There is one exception: participant #5 deviates from the CRV at 150 MPa. The degrees of equivalence with respect to the CRV are displayed in Figure 2 for some nominal pressures.

In some cases, one has to note a strong imbalance between the uncertainties of the LS according to the CMCs within the CIPM MRA and the uncertainties estimated in the comparison. That means, in the case of NMI #2, the latter uncertainties were too optimistic, but at the same time they also appeared quite small compared with the corresponding deviations from the CRV. This was presumably related to the rather poor performance of the transfer standard in its measurements mostly due to fluctuations of readings at higher pressures. This might indicate that the stability of the generated pressure at the NMIs was different, which usually cannot be seen when performing cross-float measurements.

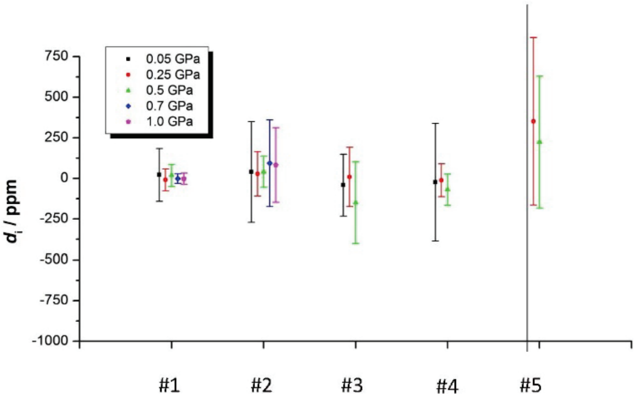

Figure 2. Degrees of equivalence with respect to the CRV of each laboratory averaged over all pressure transducers of appropriate pressure ranges. The error bars show the expanded uncertainty of the degree of equivalence for each calibrated value.

5. CONCLUSIONS

The measurements of this supplementary comparison EURAMET.M.P-S14 for high pressures in the range up to 1 GPa were conducted during 2013 and 2014 with five participating institutes: PTB, LNE, METAS, CMI and SMU. The comparison was piloted by PTB. The transfer standard comprised eight pressure transducers. Some of these, in some of the measurements, had performance as low as $14 \cdot 10^{-6}$, comparable with the uncertainty of the laboratory standards of the participants. But in general, the uncertainty of the transfer standard appeared too high to completely verify the uncertainty of the national standards claimed by the laboratories. These elevated uncertainties of the transfer standard might be influenced by pressure instabilities in the pressure generation systems of the laboratories, as concluded from high fluctuations of the transfer standard readings.

A special procedure was derived to calculate the degree of equivalence on the basis of numerous results obtained with different pressure standards, different pressure ranges and pressure change directions.

To summarize, most of the results of the participants were equivalent. The results show that a comparison of very high pressures by means of high-pressure transducers is possible, but special measures are required have stable generated pressures. However, the comparison also reveals the limits which laboratories have when calibrating high-pressure transducers.

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