

EXPERIENCE WITH A NEW 1 GPa STANDARD OF THE CMI

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Abstract:

The expansion of the high technology leads to the increasing demands for the calibration services in the extremal pressure ranges, both vacuum and high gauge pressure in liquid medium. The presentation will deal with the second one, focusing on the solution chosen by the Czech Metrology Institute (CMI) to improve its services in the range of gauge pressure from 0.5 GPa to 1 GPa. A new high-pressure standard will be described, and its first evaluation outlined.

Keywords: high pressure; pressure in liquid medium; pressure balance; free deformation piston-cylinder

1. INTRODUCTION

The ever-evolving industrial needs for the more precise calibrations in the until recently exotic ranges should be met by the expansion of the services of the national metrology institutes (NMIs). This task was highlighted for example in the recent review papers [1], [2]. However, ref. [2] also shows that the range above 0.5 GPa is ensured by only 8 NMIs. Nevertheless, we must take into account that some NMIs cover these without the framework of the Key Comparison DataBase (KCDB) of the BIPM [3] and there are also other research institutes having capabilities to provide some calibrations in this ranges [personal communication of the second author]. Also, when searching the KCDB, we can find only 3 completed comparisons in a range above this limit (EURAMET.M.P-K6, EURAMET.M.P-S14, APMP.M.P-S8) while another has not been finished yet (COOMET.M.P-S3).

The development of the primary metrology of the very high pressures has recently been revived in Europe as well which led to a cooperative research project [4], [5] and a novel interlaboratory comparison in 1 GPa range (EURAMET.M.P-S14) [6], [7].

2. ORIGINAL SITUATION OF THE CMI

The CMI keeps the Czech national standard of gauge pressure in oil medium up to 0.5 GPa which is based on a state-of-the-art pressure balance with

a set of various piston-cylinders [8]. This standard also proved well in several international comparisons [6]-[13]. However, the CMI has had to rely on a very outdated pressure multiplier system from 0.5 GPa to 1 GPa, with an unsatisfactory uncertainty 0.2 % of measured value (for $k = 2$) – by far the worst value according to [2], [3]. It was also the reason why the participation of the CMI in the comparison EURAMET.M.P-S14 had to be limited only up to 0.5 GPa. Such a situation had to be solved.

3. A NEW HIGH-PRESSURE STANDARD

We chose a novel instrument on the market – type BH5-10000B by French company Aréméca, see ref. [14] and Figure 1. It has a tungsten-carbide piston-cylinder of a simple free deformation design which is a breakthrough, because until recently only the re-entrant and the controlled-clearance piston-cylinders have been utilised for the range above 0.5 GPa, see [1].



Figure 1: The new high-pressure standard



Figure 2: The piston with a carrier mounted on it (right) and the cylinder (left)

The piston-cylinder, see Figure 2, has a very tiny nominal effective area equal 0.5 mm^2 . A radius of the piston equals cca 0.4 mm, an outer radius of the

cylinder cca 8.9 mm, a height of the cylinder cca 39.6 mm, a height of the piston cca 193.3 mm and a piston stroke cca 7 mm.

A nominal working range of the instrument is from 0.02 GPa to 1 GPa. The manufacturer states that it can be operated up to 1.09 GPa and remains safe up to 1.5 GPa. The manufacturer stated effective area $A_0 = 5.03002 \cdot 10^{-7} \text{ m}^2$ (for 20 °C) and $\lambda = 9.4 \cdot 10^{-7} \text{ MPa}^{-1}$. The reported accuracy class for the nominal range equals 25 kPa + 0.05 % of measured value.

The instrument is operated using di(2)-ethyl-hexyl-sebacate oil and so it is compatible with the rest of our high-pressure equipment. The weight of the piston with its carrier is approximately 1 kg, the remaining set of 15 mass pieces has a total weight 50.5 kg. The pressure generation and regulation are provided by a pair of hand-operated presses. An automatic rotation of the piston can be utilized but it can be also detached.

4. TRACEABILITY AND UNCERTAINTY

The new instrument was purchased and installed in summer 2021. It was calibrated by the mentioned above Czech national standard of gauge pressure in oil medium in autumn 2021, see Figure 3 and Figure 4.

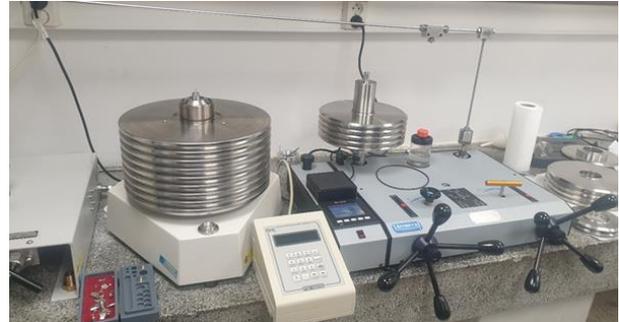


Figure 3: The new high-pressure standard (right) and the national standard of gauge pressure in oil medium (left) during their mutual comparison

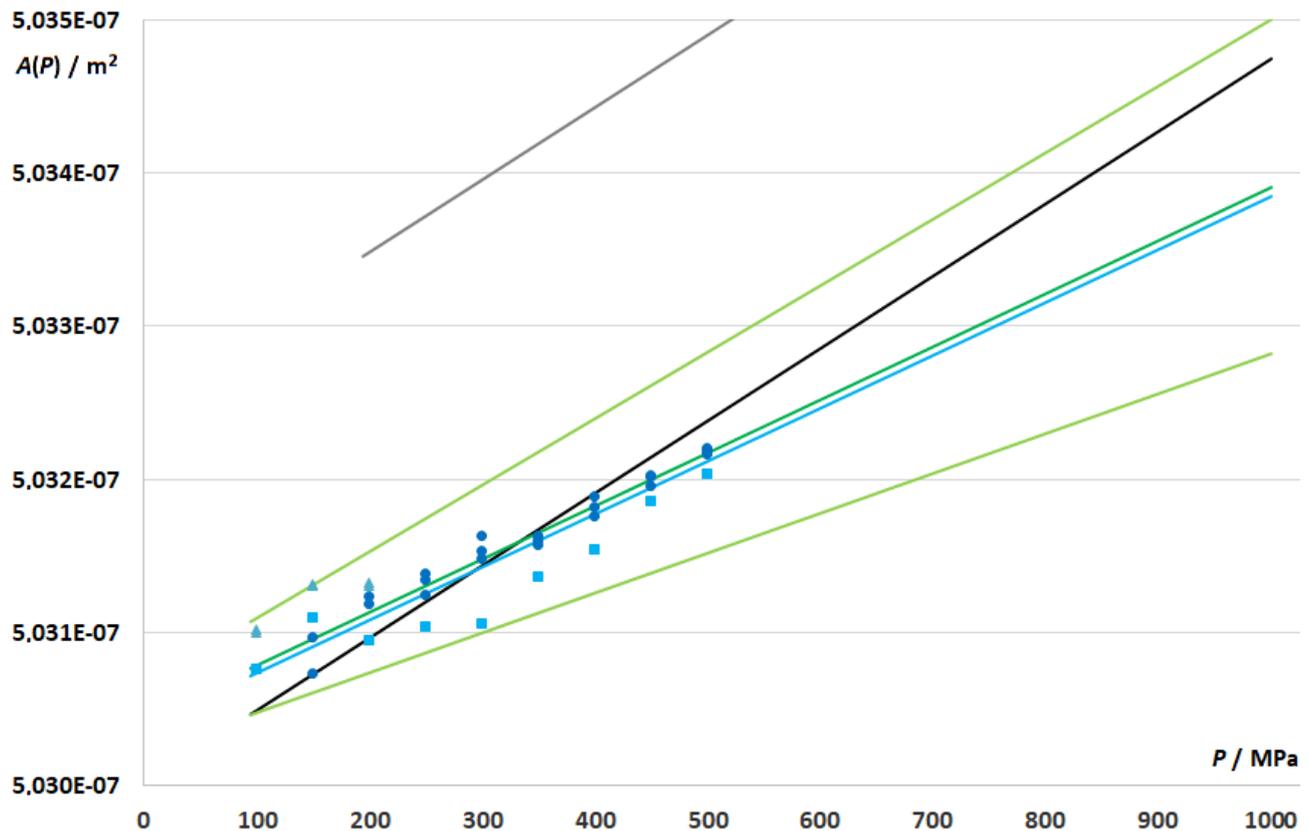


Figure 4: Traceability, uncertainty and manufacturer's specification. Measurements with piston-cylinder no. 1637 - dark blue dots, p.-c. no. 109 - light blue squares, p.-c. no. 119 - teal triangles, 500 MPa data linear regression procedure - blue line, resultant pressure dependence of effective area - dark green line, its uncertainty limits - light green lines, determination of the manufacturer - black line, specification of the manufacturer - grey line.

Firstly, piston-cylinder no. 1637 was used for the range 150 MPa to 500 MPa, with 50 MPa step, in two increasing series and one decreasing, dark blue dots in Figure 4. Secondly, piston-cylinder no. 109 was used for the range 100 MPa to 500 MPa, with 50 MPa step, in one increasing series, light blue

squares. Finally, piston-cylinder no. 119 was used for the range 100 MPa to 200 MPa, with 50 MPa step, in two increasing series, teal triangles. During these measurements we also monitored free rotation time, which remained satisfactory for this range, and piston fall rate which was cca 0.2 mm/min at

20 MPa, steadily increasing up to cca 1.5 mm/min at 200 MPa, remained stable up to 900 MPa, then jumping to cca 5 mm/min at 1 GPa.

We used the results of the measurements with the 500 MPa piston-cylinders for a linear regression procedure (blue line) which resulted in a value of the effective area $A_0 = (5.03039 \pm 0.00014) \cdot 10^{-7} \text{ m}^2$ and the pressure deformation coefficient $\lambda = (6.9 \pm 0.4) \cdot 10^{-7} \text{ MPa}^{-1}$ (for 20 °C, only 95 % coverage type-A uncertainties of linear fit of 32 pairs of values). Then we used this pressure deformation correction for the entire set of all the measured points. From the resulting set of the zero-pressure effective areas we obtained the average $A_0 = (5.03044 \pm 0.00006) \cdot 10^{-7} \text{ m}^2$ (only 95 % coverage type-A uncertainty of average of 38 values).

We increased the uncertainty of A_0 by the uncertainty the national standard of gauge pressure in oil at 100 MPa (42 ppm) which using square root of sum of squares gave 44 ppm and put uncertainty of λ to be 25 % for safe. This lead to the final values of $A_0 = (5.03044 \pm 0.00022) \cdot 10^{-7} \text{ m}^2$ and $\lambda = (6.9 \pm 1.7) \cdot 10^{-7} \text{ MPa}^{-1}$ (for 20 °C, for $k = 2$).

Our determination of A_0 and λ differs from the those of the manufacturer by more than our uncertainty. However, if we depict in a pressure dependence of our effective area (dark green line) together with its uncertainty limits (light green lines), then, in the range from 0.1 to 1 GPa, the dependence of the manufacturer (black line) is totally in those limits and everything lies deeply in the conservative specification of the manufacturer (grey line).

We also used Lame's equation to determine λ . We used the effective area value to get piston radius and cylinder inner radius and a calliper measurement for cylinder outer radius. The tungsten-carbide mechanical properties were taken from [15], where they are stated as the intervals. Hence, the averages were taken and their uncertainty guessed from the limits. Thus, we got $\lambda = (6.6 \pm 0.7) \cdot 10^{-7} \text{ MPa}^{-1}$ (for $k = 2$). Although the method is rather coarse, it leads to a very good agreement with the experimental value.

Our first estimation of the best measurement capability of the new instrument is 0.025 % of measured value (from 0.15 GPa to 0.5 GPa, for $k = 2$). We are also contemplating to use a quadratic pressure dependence of the effective are as suggested in [16]. However, the linear dependence has been also used satisfactorily in the pressure range up to 1 GPa, see [17,18]. One way or another, the extrapolations both for the linear fit and the quadratic fit lead to uncertainties well below manufacturer's specification which would be a considerable improvement for our laboratory. Nevertheless, an interlaboratory comparison is

necessary to find the most suitable way of evaluation of λ and to confirm a correctness of our determination of the uncertainty budget. We continue our preparations for it.

5. SUMMARY

The CMI responded to the European developments in the high-pressure metrology. A novel instrument was purchased, made traceable to the Czech national standard and evaluated. The first results and experience are very promising. However, a correctness of the described above traceability of our new standard must be still confirmed by an international comparison which is a task for the nearest future.

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