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Original

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CIRCULAR COMPARISON OF CONVENTIONAL PRESSURE STANDARDS USING A TRANSPORTABLE OPTICAL REFRACTOMETER: PREPARATION AND TRANSPORTATION

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Abstract:

Using a transportable Fabry-Pérot cavity refractometer, a circular comparison of existing primary standards at several national metrology institutes is currently underway. This paper provides information about the refractometer, the preparation for the comparison, and the transportation procedure.

Keywords: pressure; circular comparison; transportable refractometer; GAMOR; Fabry-Pérot cavity; pressure balance

1. INTRODUCTION

By measuring the refractivity and the temperature of a known pure gas, its pressure can be assessed with high accuracy from fundamental principles. One such type of instrument is based on a dual Fabry-Pérot cavity (DFPC), where two lasers with similar wavelengths are locked to the longitudinal modes of the two cavities. When gas is filled in one of the evacuated cavities, the frequency of the corresponding laser will change. By combining the light from the two lasers onto a fast photodetector, the relative change can easily be measured. From the frequency change, the refractivity of the gas can be measured, and by use of the Lorentz-Lorenz equation and an equation of state, the pressure can be assessed. Within the EMPIR 18SIB04 QuantumPascal project, such instruments have been further developed and scrutinized, with the result that their performance has been significantly improved.

Among several tasks, the project comprises an experimental circular comparison of pressure balances at four National Metrology Institutes (NMIs) using a transportable refractometer. The

main purpose of the circular comparison is to test the capability of the technology to operate outside well-controlled laboratories as well as to investigate whether it is suitable to be employed in future official comparisons of conventional standards.

The refractometer has been jointly constructed by RISE and Umeå University (UmU), both in Sweden, and utilizes the Gas modulation refractometry (GAMOR) methodology [1]. This methodology reduces significantly the influence of various type of disturbances (primarily fluctuations and drifts), which makes the system extraordinary sturdy, and hence ideal as a transportable instrument [2].

Although the refractometer in principle can operate as a primary standard and has, in an earlier work [3], been evaluated in terms of uncertainty, it was in this work operated as a transportable standard. The main reason is that this could significantly reduce the complexity of operation.

This paper presents how a GAMOR-based transportable refractometer system was designed to maximize robustness and serviceability and to enable easy transportation and setup at the visited sites. It also describes in some detail how the initial setup/calibration preparation was carried out to allow for a successful and efficient measurement campaign at the different NMIs. Since the campaign presently is still undergoing, no performance data will be reported here; those will be presented in a future, upcoming report.

2. THE TRANSPORTABLE REFRACTOMETER

The refractometry system, denoted the Transportable Optical Pascal (TOP), has previously been described in detail [3], [4]. Besides a brief

overall description, the emphasis of this paper is placed on the transportable aspects of the system.

2.1. System details

The refractometer is based on a DFPC made of Invar and is designed to operate between 1 Pa and 100 kPa [5]. By using a metallic cavity paired with the GAMOR drift-and-fluctuation-reducing methodology, the system offers several advantages that are of particular importance when operated as a transportable system, in particular:

- 1) it has favourable thermal properties as compared to glass-materials, which provide thermal stability within some tens of seconds [6]; and
- 2) the spacers can easily be customized and machined, allowing them to be repaired or taken apart to be cleaned.

The refractometer system fits on a wheels-equipped 19-inch rack with a 60×60 cm² footprint and a height of 120 cm (Figure 1). It comprises, in its interior, seven modules that contain, among other things, two lasers, fibre-optics, electronics, and a gas-handling system. The DFPC is placed on top of the rack for ease of realignment [4]. For operation, the system requires external vacuum pumps.

The system differs from a previously constructed stationary system (SOP) [3], [5], [7] primarily by the way the temperature is assessed; instead of assessing the temperature with respect to a Ga fixed point cell, it utilizes calibrated Pt-100 sensors. This contributes to its uncertainty by 20 parts-per-million, which is on par with the uncertainties of pressure balances used in the circular comparison. Its short-term precision has been shown to be significantly better than its uncertainty, in the $1:10^8$ range [4].



Figure 1: The TOP from a front (left) and rear (right) point of view.

2.2. Designed for transportation

The TOP was designed to allow for easy transportation, unpacking, setting up (or initialization), and serviceability so as to be able to perform measurements in a reasonably simple and fast way with a performance on par with existing standards. Hence, it was not designed to reach the highest possible performance (e.g., in par with the SOP system). To achieve this, several aspects of the design were considered.

The first thing to consider was if the system should be designed and constructed so it could be operated by unexperienced staff with only minor training. The advantage of this would be that it could be shipped by “a third party” and solely be operated by local staff. Although this was considered possible in theory, given the overall complexity of the system, it was decided that the scope of the undertaking would benefit from having the system accompanied by RISE/UmU staff to oversee the measurements. This would also allow for identification of features that could be improved to future versions of the instrumentation, which then possibly could be made more autonomous.

Given this decision, the system was designed and constructed with this in mind. This has several advantages as it allows for significant relaxed constraints in terms of installation complexity, setup, and control and data management complexity. This is, for example, manifested in the possibility to ship many of the components in modules, such as vacuum pumps, electronics, and vacuum connections, which makes it possible to fit the full system on a standard EUR-pallet. See Figure 2.



Figure 2: The TOP under packing.

Overall, this allows for a more simplified design, but with the drawback that the system is not a fully “turnkey” system; it is rather a “plug’n’play” system. However, since the installation and setup were carried out by experienced operators during the measurement campaign, this was not considered to be an issue. (See below for details about the setup at each institute).

Given the fact that the system would be accompanied by experts, it was also decided that the TOP could mimic the SOP situated at UmU when it comes to the cavity setup [3], [5], [7]. This implies, in short, that the cavity ensemble, including the free-space optics, was constructed in such a way that, if needed, it is easy to access, open, and realign. This also includes the cavity mirrors, which, instead of being attached with adhesive or optical contacting, are pressed against and into the Invar cavity spacer by mechanical means [5]. Although a potential drawback with this design is that it might increase the risk for miss-alignment due to the transport (which so far has not been addressed), the advantage is that if the transportation adversely affects the alignment of the optical components, it can be addressed by the RISE/UmU staff relatively swiftly. Even though this adds some complexity to the system and the setup after each transportation, it eliminates the risk of a total failure of the system in case the transportation would adversely affect the alignment of the optical components.

To simplify the transportation and initialization, two main design compromises were made with respect to the SOP system.

Firstly, instead of assessing the temperature by use of thermocouples referred to the melting point of gallium by use of a Ga fixed point cell (which provides an excellent accuracy), the TOP assesses the temperature of the cavity (and thereby the gas) using Pt-sensors whose outputs were assessed by the use of a high-performance DAQ-system. The reason for this is that it was considered unsuitable to base the system on a Ga fixed point cell since it is not trivial to operate such a device and it takes time to stabilize it after initialization. To ensure sufficient stability of the temperature measurements (traceability at the 5 mK level), the response of the Pt-100 sensors was, after each transportation, calibrated by a standalone calibrated device (brought separately as hand luggage).

Secondly, the TOP is designed around a wheel-equipped-19-inch rack, where the actual cavity sits on top of the rack. See both Figure 1 and Figure 2. This is non-ideal in terms of stability; it would be preferable to place the cavity ensemble on a firm and stable surface, such as an optical table. However, this overall design has the advantages that it makes it very easy to move around within each laboratory, and minimize the footprint of the system, which otherwise can be an issue at host laboratories. It is worth to emphasize that even though the design is far from ideal in terms of performance, it has been shown in a separate work, that, primarily due to the use of the GAMOR methodology, the TOP has an excellent stability,

which for all purposes for the circular comparison is significantly better than needed [4].

3. CALIBRATION AT RISE

The TOP is in principle capable of operating in “primary” mode. This was demonstrated in 2021 when the TOP was given an estimated uncertainty of $[16 \text{ mPa} + 28 \times 10^{-6} \text{ P}]$ [3]. However, due to limitations of the vacuum system at that time, that characterization was solely carried out in the 10 kPa – 30 kPa pressure range. Furthermore, since the characterization of the pressure induced cavity deformation was carried out over two years ago and given that some adjustments have been done to the cavity since then, it can be assumed that the characterization is no longer valid.

Although it would have been possible to perform a new characterization of the cavity-deformation phenomenon before the circular comparison, such a characterization would have been time-consuming. Given some delay caused by the Covid-19 outbreak, it was decided that the TOP, during this circular comparison, instead should be operated in an “uncharacterized” mode of operation. Hence, to perform the circular comparison, and thereby test the performance of the instrumentation outside well-controlled laboratories, it was not considered necessary to have the system fully characterized with respect to its cavity deformation; it was instead considered appropriate to perform a “characterization” against a traceable pressure balance at RISE (Ruska 2365A-754) and operate the TOP as a transportable standard.

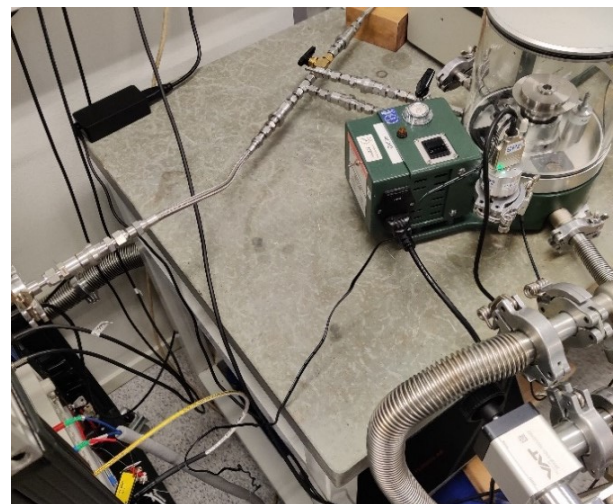


Figure 3: The TOP (left) connected to the pressure balance (right).

To perform this, as is shown in Figure 3, the refractometer was connected to the pressure balance. To reduce the risk for systematic errors, measurements were performed at nine different pressures in a randomized order as shown in

Table 1. The data were evaluated using standard expressions for pressure, molar density, and refractivity although with the latter one in the absence of the cavity deformation (and thereby, for simplicity, also neglecting any possible influence of mirror penetration depth and Gouy phase) [8], i.e., by use of the Eqs. (1b) and (4) in Zakrisson et al. 2020 [9] with both the relative deformation and the penetration depth set to 0.

Table 1: Measurements # - temporal order; Nom. P - Nominal pressure value; Est. P – Estimated pressure from the pressure balance with the weights used.

| Measurement # | Nom. P [kPa] | Est. P [kPa] |
|---------------|--------------|--------------|
| 1 | 30 | 30,637 |
| 2 | 50 | 50,083 |
| 3 | 90 | 90,162 |
| 4 | 20 | 20,398 |
| 5 | 10 | 10,158 |
| 6 | 40 | 40,135 |
| 7 | 70 | 69,976 |
| 8 | 80 | 79,924 |
| 9 | 60 | 60,320 |

Although the response of this characterization was looking ostensibly linear on a pressure-vs-pressure plot, illustrated by Figure 4 (a), which shows the response of the TOP vs. the response of the pressure balance, a closer scrutiny reveals that the response is weakly non-linear. The solid curve shows a second order fit of the form $a + bP + cP^2$, where $a = -0.614$ Pa, $b = 1.0021$, and $c = 1.52 \times 10^{-9} \text{ Pa}^{-1}$.

A simple goodness of fit analysis was performed and the Residual Standard Error (RSE) for the fit, RSE2, was found to be 0.16. The comparable entity for a linear fit, RSE1, was found to be 1.02. This shows that a second order fit is needed to adequately describe the response.

Figure 4 (b) shows the difference in pressure assessed by the TOP and that set by the pressure balance versus the pressure of the latter.¹

To visualize the degree of non-linearity, Figure 4 (c) displays the deviation of the data in Figure 4 (b) from a linear fit together with 99% confidence interval of the fit.

Figure 4 (d) displays the residuals of the fit. These residuals do not show any pressure dependent trend, which vouches for that the assumption of a second order response vs. pressure is appropriate.

While it is not of importance for the circular comparison, in which this fit simply can be seen as a mean to “calibrate” the TOP against the RISE pressure balance, it is of interests to scrutinize the

most likely reason for the deviations between the TOP and the pressure balance.

The offset of -0.6 Pa can mainly be attributed to an insufficient evacuation of the cavity of each measurement cycle.

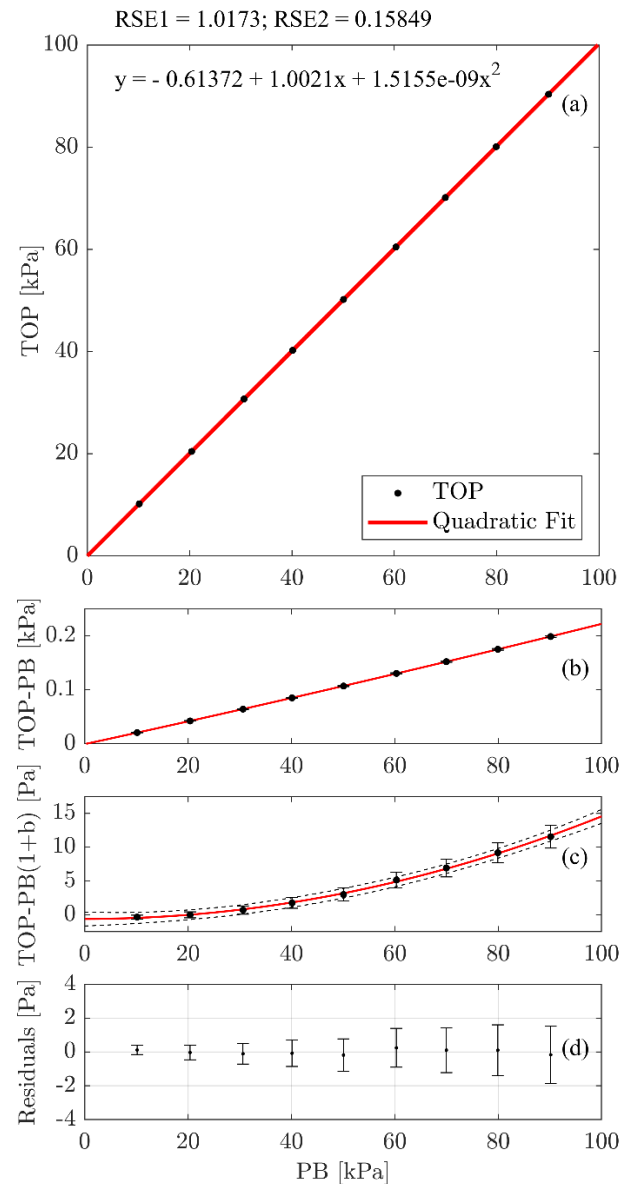


Figure 4: Panel (a): Black markers – TOP as a function of pressure balance (PB); red curve – second order polynomial fit. Panel (b): Black markers – the difference between the TOP and the PB as a function of the PB; red curve – second order polynomial fit. Panel (c): Black markers – the deviation of the data in Panel (b) from a linear fit with $k = 2$ uncertainty; red curve – fit to data without linear term; black dashed curves – 99% confidence interval of the fit. Panel (d): Residuals of fit in previous panels with error bars representing a $k = 2$ uncertainty of the PB. The RSE1 and the RSE2 at the top of the figures are goodness of fit measures for linear and second order polynomial fit in terms of RSE, respectively.

¹ Note that the difference of the fits in panels (a) and (b) of Figure 4 is solely in the linear term that is reduced by 1 unit in panel (b).

The deviation of the b parameter from unity [given by the slope of the fit in panel (b)] can be mainly attributed to the fact that the refractometer was evaluated with the deformation parameter set to 0. Likewise, the non-linearity [given by the fit in panel (c)] can be attributed to a weak second order pressure dependence of the relative deformation, potentially attributed to the removable mounting of the mirrors to the cavity spacer.²

4. TRANSPORTATION

As was alluded to above, the relatively small size of the system enables the use of a standard EUR-pallet. As was shown in Figure 2, the pallet fits both the TOP and auxiliary equipment such as vacuum pumps, oscilloscopes, and spare parts, netting a total weight of around 300 kg. Packing the system in its entirety on a standard pallet makes it easy to ship by standard shipping services. At the time of the finalization of the manuscript the system has so far been successfully transported from RISE in Borås, Sweden, to PTB in Berlin, Germany, after which it was sent to INRiM in Turin, Italy, before it was routed to LNE in Paris, France by using commercially available service (at a cost of roughly 200 Euro per transport). Presently, only the final shipment back to RISE in Borås, and the final measurements, remain to be carried out.



Figure 5: The package containing the TOP just after arrival at PTB, Berlin.

The unpacking, installation and setup at PTB and INRiM went very smooth at both sites and the system was operational within four hours, despite rather rough handling during transportation. Figure 5 depicts the fully packed system directly after arrival at PTB.

Additionally, Figure 6 depicts a tilt indicator that shows that the pallet at least once during the transportation from PTB to INRiM, was, at some point, tilted at least 40° or subjected to significant acceleration from both sides due to impact. Despite this, the system was fully functional when arriving at INRiM, requiring only a small amount of optimization of the mode matching of the light to the cavities.



Figure 6: Tilt indicator of the package after delivery.

Figure 7 shows the system when installed and fully operational at INRiM. Here, the TOP was placed next to a “stable table”, on which the pressure balance was installed (seen to the right on the table, behind the TOP).



Figure 7: System installed at INRiM. The pressure balance can be seen on the table behind the TOP.

² It is worth to mention that this non-linearity has not been clearly seen before when pressure up to only 30 kPa has been considered, as was the case in [3] and [9].

The systems were run on N₂ from a separate gas cylinder, which, in the picture, stands to the left, next to the vacuum pumps used, which, in turn, are on the floor (not in the picture). Besides the workstation with the laptop running the control software (in front of the TOP, partly seen in the lower left corner), one can see that the system in its entirety takes up about the same space in the laboratory as the EUR-pallet it came on.

At LNE and LNE-CNAM, the system was unpacked and installed in the same time frame as at the previous sites, see Figure 8. This time, however, it was not operational straight away; it had obviously been unfavourably affected by the transportation. During installation, some “issues” became apparent which took approximately two full days to solve. These were related to untightened screws; optical fibres were not attached firmly to their connectors, the cavity ensemble was not firmly attached in its correct position, etc., which all were attributed to vibrations during transport.



Figure 8: System installed at LNE and LNE-CNAM. The pressure balance (PG-7607 with automatic weight changing system) can be seen to the right of the TOP.

Most likely this was the culmination of rough handling or transportation. Another potential reason is that, during the transport, the seasons were shifting from spring to summer, and the outside temperature changed significantly; from an average of 10 °C in Turin to 30 °C in Paris. Nevertheless, after the on-site service by accompanying personnel from UmU, measurements could be performed by LNE without any noticeable issues.

5. SUMMARY AND CONCLUSIONS

This paper presents the preparation for, and implementation of, a test circular comparison of pressure balances that includes four NMIs in which a DFPC-based refractometer utilizing the GAMOR methodology has been used as a transportable standard. The paper also presents some details about

how the initial characterization of the system was done, as well as how the system was transported.

The purpose of the comparison is not only to assess the compatibility between conventional pressure standards but also to assess the ability of one of the arising quantum-based methods to serve as a transportable instrumentation for assessment of pressure during a ring comparison.

Results from the comparison between the conventional pressure standards will be presented in a future publication.

Regarding the refractometer, despite rough handling during transportation and its high complexity, it demonstrated robustness since only minor adjustments were required before operation at each institute visited. The experiences gained during this circular comparison will be further assessed and used as a basis for future upgrades of the system.

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