



EMPIR Project: '18SIB04 QuantumPascal'

Deliverable D8: 'Report on the circular comparison between a transportable Fabry-Pérot refractometer and the conventional primary standards that are available at different facilities, including conclusions about how the refractometric method and the optical refractometer should be handled outside of well controlled laboratories in order to enable and simplify future adaptations by end users

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Report on the circular comparison between a transportable Fabry-Perot refractometer and the conventional primary standards that are available at different facilities, including conclusions about how the refractometric method and the optical refractometer should be handled outside well controlled laboratories in order to enable and simplify future adaptations by end users

Deliverable D8 in the EMPIR project 18SIB04, "QuantumPascal"

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Abstract This report summarize the circular comparison of pressure balances using a transportable Fabry-Perot refractometer developed within 18SIB04 QuantumPascal. The report shortly describe the transportable system and how it operates. Thereafter, details about each measurement together with the results showing that transportable refractometer is capable of performing a full circle comparison that can demonstrate that the pressure balances agrees well within their uncertainties. Finally, some general conclusion and recommendation on how a transportable refractometer can be improved in terms of both performance and simplicity for future use.





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1 Introduction

This report, which is Deliverable D8 in the EMPIR project 18SIB04 "QuantumPascal", summarizes the 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 and demonstrate the capability of the transportable refractometer 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 significantly and automatically reduces the influence of various type of disturbances (primarily fluctuations and drifts)[2, 3], which makes the system extraordinarily sturdy, and hence ideal as a transportable instrument [4].

Although the refractometer in principle can operate as a primary standard and has, in an earlier work [5], been evaluated in terms of its uncertainty, it has in this work been operated as a transportable standard. The main reason for this is that it significantly reduces the complexity of operation.

The report is structured as follows. First a short description of the GAMOR-based transportable refractometer is presented. Thereafter, the report 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 together with the results of the individual measurements at other institutes. Finally, the report present the combined results of the comparison, and present some future recommendations.

It also worth to point out that due to the Covid-19 pandemic, the circular comparison was delayed roughly 18 months, and the results therefore have not yet been submitted for publication in peerreview, which is the intention. The results presented in this report are hence preliminary as further analysis will be carried out after the completion of the 18SIB04 project. Furthermore, the delay also limited the circular comparison to only include pressure balances as conventional standards.

2 Short description of the Transportable system

The refractometry system, denoted the Transportable Optical Pascal (TOP), has previously been described in detail [5, 6]. In this section, an overview of the system is given, as well as some details regarding the transportable aspects of the system.

2.1 System details

The refractometer is based on a dual Fabry-Pérot cavity (DFPC) made of Invar and is designed to operate between 1 Pa and 100 kPa [7]. By using a metallic cavity paired with the GAMOR methodology, the system offers several advantages that are of particular importance when operated as a transportable system:

- 1. Invar has favourable thermal properties as compared to glass-materials, which provide thermal stability within some tens of seconds [8]; and
- 2. the spacer can easily be customized and machined, allowing it to be repaired or taken apart to be cleaned; and
- 3. the automatic mitigation of drifts and fluctuations due to the GAMOR methodology.

The refractometer system fits on a wheelsequipped 19-inch rack with a 60×60 cm 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 [6]. For operation, the system requires external vacuum pumps.

The system differs from a previously constructed stationary system (SOP) [5, 7, 9] 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 an uncertainty by 25 parts-per-million (ppm), which is on par with the uncertainties of pressure balances used in the circular comparison. To improve on this, the TOP have in this work been complemented by an external temperature calibration device.





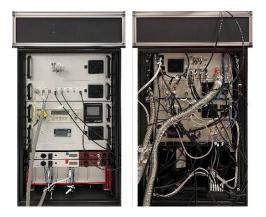


Figure 1. The TOP from a front (left) and rear (right) point of view.

2.2 Design considerations to allow for transportation

The TOP was designed to allow for easy transportation, unpacking, setting up (or initialization), and serviceability 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 weather or not the system should be designed and constructed so it could be operated by 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 to be 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.

Based on this decision, the system was designed and constructed with this in mind. This has several advantages as it allows for significantly relaxed constrains 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 a drawback. (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 [5, 7, 9]. 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 [7]. Although a potential drawback with this design is that it might increase the risk for misalignment due to the transport. 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. Evenvthough this adds some complexity to the system and the setup after each transportation, it eliminates the risk of a major failure of the system that would require time consuming and costly repairs.

Furthermore, 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 fixed point cell (which is utilized in the SOPsystem, and provides an excellent accuracy) the TOP assesses temperature of the cavity (and thereby the gas) using Pt-sensors whose outputs were assessed by the use of a DAQ-system. The reason for this is that it was considered inexpedient 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 wheelequipped-19-inch rack, where the actual cavity sits on top of the rack. See both Figures 1 and 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 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 [6].

2.3 Transportation of the system

As was alluded to above, the relatively small size of the system enables the use of a standard EUR-pallet for the transportation. 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. In this fashion, the system was 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). Finally, it was transported back to RISE in Borås, Sweden.

Additionally, Figure 3 depicts a tilt indicator that shows that the pallet at least once during the trans-

portation from PTB to INRiM, was, at some point, tilted at least 40° or subjected to significant acceleration from both sides due to impact (as was the case for subsequent transportation, INRiM-LNE and LNE-RISE). Despite this and visual damages to the parcel after each transportation, the system was fully functional when arriving at each location, requiring only minor optimization of the mode matching of the light to the cavities.



Figure 3. Indicator showing that the EUR-pallet containing the TOP was tilted 40° during transportation.

3 Measurements at different facilities

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 mPa $+28 \times 10^{-6}$ P] [5]. However, due to limitations of the vacuum system at that time, that characterization was solely carried out in the 10 - 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, should not rely on the previous cavity deformation characterization. The system was instead characterized against a traceable pressure balance at RISE (Ruska 2365A-754). It was therefore operated as a transportable standard (see section 3.1).

At each location, the experimental schematic shown in Figure 4 was used. Here, a N_2 gas canister (99.999% purity) is connected to the TOP, acting as the controller. The TOP coarsely controls the flow of gas and the pressure within both the TOP and the pressure balance, while the pressure balance subsequently regulates to the exact pressure determined by the series of masses placed on the piston. The TOP also controls the valves to the shared turbo pump evacuating the common gas system between each pressurized cycle.

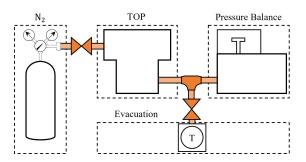


Figure 4. Schematic illustration of the connection between gas supply (N_2) , TOP, pressure balance, and the gas evacuation system.

3.1 Initial calibration at RISE

To perform the initial calibration of the refractometer, it was connected to the pressure balance at RISE in Borås according to the schematic in Figure 4. A picture of this is shown in Figure 5, here, the TOP is shown to the left in the picture, the pressure balance is to the right. The gas canister and the turbo pump are not visible in the picture but are placed to the left of the TOP and under the table on which the pressure balance is placed, respectively.

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 was evaluated using standard expressions for pres-

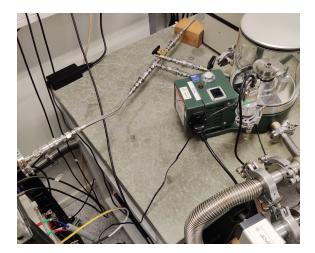


Figure 5. Picture of the TOP (to the left in the picture) connected to the RISE pressure balance(to the right in the picture).

sure, 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) [10], i.e., by use of the Eqs. (1b) and (4) in Zakrisson et al. 2020 [11] with both the relative deformation and the penetration depth set to zero.

M.#	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	

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

Although the response of this calibration was looking ostensibly linear on a pressure-vs-pressure plot, Figure 6(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 = -0.50641 Pa, b = 1.0021, and $c = 1.5148 \cdot 10^{-9}$ [Pa]⁻¹.

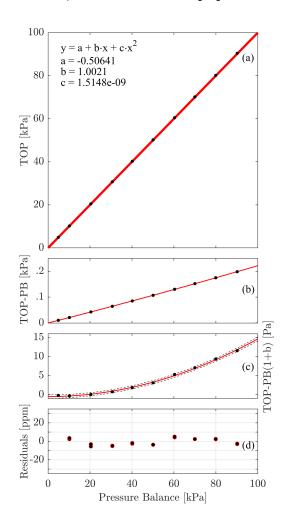


Figure 6. Calibration data: panel (a), black markers - TOP vs. Pressure Balance red line - quadratic fit of the form shown in the top left corner; panel (b), black markers - difference between TOP and pressure balance vs. pressure balance, red line - quadratic fit of the difference between the TOP and pressure balance; panel (c), the non-linear components of panel (b); panel (d), black markers with red outline - residuals of the fit

Figure 6(b) shows the difference in pressure assessed by the TOP and that set by the pressure balance versus the pressure balance.

To visualize the degree of non-linearity, Figure

6(c) displays the deviation of the data in Figure 6(b) from a linear fit together with 99% confidence interval of the fit in Figure 6(b).

Figure 6(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. This is also the data/plot that is to be compared to the measurements from the different sites, as well as the data obtained from the same pressure balance once the TOP is back in Borås.

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 interest to scrutinize the most likely reason for the deviations between the TOP and the pressure balance. The offset of -0.5 Pa can conceivably be attributed to an insufficient evacuation of the cavity of each measurement cycle.

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 zero. 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.

3.2 PTB Measurements

The system was transported to PTB using a standard transportation service. The unpacking, installation, and setup at PTB went very smooth and the system was operational within four hours, despite some visual evidence of rough handling during transportation. Figure 7 depicts the fully packed system directly after arrival at PTB.

After installation, the TOP was left to stabilize for 24 hours after which alignment of lasers was carried out. Furthermore, the high sensitivity of the TOP to molar density changes allows for testing of the gassystem in terms of leakages. By continuously evacuating one of the cavities and connecting the other to the rest of the system by successively opening valves, sources of leaks can efficiently be detected. As such, leaks were identified and removed (to some extent), and it was concluded that the combined leaks in the TOP and pressure balance were sufficiently low to allow for comparative measurements.







Figure 7. The TOP after arrival at PTB-Berlin.

Thereafter, measurements were carried out against the pressure balance 'FLUKE 2465A Gas Piston Gauge' in combination with the piston-cylinder assembly 'FLUKE TL1346' (calibrated in Jul. 2022) assembly denoted 'PTB2'. The results can be seen in Fig. 8.

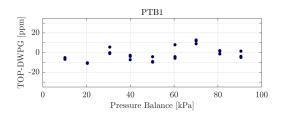


Figure 8. The comparative data from PTB; difference between the TOP and the pressure balance.

3.3 INRiM Measurements

Figure 9 shows the system during unpacking at IN-RiM. The pressure balance used at INRiM was of the model 'DHI-FLUKE PG7601' with piston-cylinder assembly '7100-10' (serial number: 1742). Also here, the installation took roughly 4 hours, after which it was left to stabilize for 24 hours, followed by alignment and leak testing.



Figure 9. The TOP during unpacking at INRiM.

During the measurements, the TOP was operated by RISE/UmU staff, and the height/rotation of the pressure balanced was controlled by INRiM staff. The data is presented in Fig. 10.

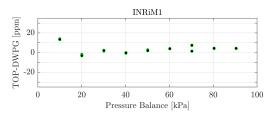


Figure 10. The comparative data from INRiM; difference between the TOP and the pressure balance.

3.4 LNE-CNAM Measurements

At LNE/CNAM, the system was unpacked and installed in the same time frame as at the previous sites. 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 lose 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.

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 RISE/UmU, measurements could be performed without any noticeable issues. Figure 11 shows, to the right, RISE/UmU staff operating the TOP, and to the left, LNE staff operating the pressure balance (model 'DHI PG 7607(sn 114A)' with a '20 cm²' piston-cylinder assembly).

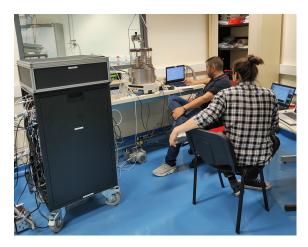


Figure 11. Picture of the TOP, pressure balance and staff operating the two, during the one of the measurements at LNE/CNAM.

The data from the measurements are presented in Fig. 12.

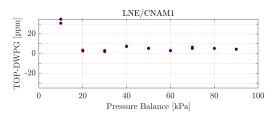


Figure 12. The comparative data from LNE/CNAM; difference between the TOP and the pressure balance. Note specifically that the two measurements at 10 kPa should be interpreted with some caution as the pressure balance did not provide a fully stable pressure during the measurement cycles.

3.5 Second RISE Measurements

After shipment back to RISE in Borås, the system was successfully installed in 2 hours, after which the system was left to stabilize for 24 hours before alignment of the lasers to the cavities. Similar to the issue at LNE/CNAM, the cavity ensemble was not firmly attached to its correct position which consumed one extra working day before the measurements could be carried out on day four.

The fully installed and operating TOP and pressure balance (Ruska 2365A-754, same as during the first visit to RISE) is shown in Figure 13.



Figure 13. Picture of the TOP connected to the pressure balance at RISE. In the foreground, the data evaluation- and control software running laptops can be seen.

The TOP and pressure balance can be seen to the left and right, respectively, in the background, and





the laptops in the foreground are the data evaluation computer and the laptop running the control software to the left and right, respectively. Here, one can see that the TOP-system in its entirety takes up about the same space in the laboratory as the EURpallet it came on.

Finally, the data from the second measurement campaign at RISE is shown in Fig. 14.

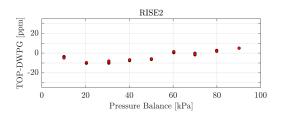


Figure 14. The comparative data from the second measurement campaign at RISE; difference between the TOP and the pressure balance.

3.6 Combined results

The combined results from all sites are shown in Figure 15. Here, the mean of the measurements corresponding to the same pressure balance and pressure level are shown. This does not affect the overall conclusion and is done purely for visual purposes.

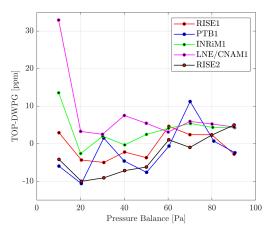


Figure 15. Combined data

As can be seen, all measurement points (except LNE/CNAM1 - 10 kPa, where the deviation is attributed to the fact that the pressure balance did not provide a stable pressure during the measurement cycles for this pressure) are in good agreement. It is worth to note, that the initial and final measurements (RISE1 and RISE2) does not fully agree. However, this can fully attributed to, and explained by, the fact that the resolution in temperature assessment is limited to a mK-level (1 mK corresponds to a 3.3 ppm difference).

4 Conclusions

It has been shown that the TOP can successfully be transported using standard shipping services and that it is capable of working outside well-controlled laboratories. Furthermore, it has been shown that the TOP has not undergone any noticeable changes during this entire measurement campaign. This means that there are no notable changes related to time on the scale of one year, and there are no changes related to the handling during transportation. Finally, the comparison indicates that all pressure balances agrees well within their respective uncertainties.

To conclude, the TOP is suitable to be employed in future comparisons of conventional standards.

4.1 Recommendations

To summarize the recommendations, the following list of priorities is provided. Here the recommendations are given in descending order.

- 1. Implement alternative temperature uncertainty reduction module.
 - The calibrated FLUKE-system, used to correct the pt-100 probes in the TOP, has been proven to work well, however, it is not sustainable. The FLUKE-system was during this comparison handled by care by the RISE/UmU—operator and transported as hand luggage to maintain complete oversight of the instrument. At any time during this campaign, the calibration could have been completely lost in the event of any type of shock, which would have been extremely time consuming and perhaps detrimental to the comparison as a whole.
- 2. Cavity spacer ensemble suspension rework.





- After transportation to LNE/CNAM and RISE (the second time), the suspension screws of the cavity ensemble were loose due to vibrations. Although time consuming to fix, the main issue is the risk this entails. If the screws were to loosen slightly more, this would have caused the cavity ensemble to drop a few centimeters, possibly changing and damaging the ensemble. A worst case scenario would be one or more cracked mirrors, which in theory can be replaced but; without knowledge of the impact on the TOP—calibration.
- 3. More robust laser locking.
 - When looking at the TOP data for each individual cycle, from time to time one of the lasers unlock for a few measurement points (0.25 - 1 s) during a measurement window. Since the beat frequency is still measured during this time frame, it compromise the entire measurement cycle and it can be hard to detect unless each measurement point undergo rigorous scrutiny (which is most often not plausible as time can be limited during measurement campaigns such as these).
- 4. Thermal stabilization rework.
 - Thermal stabilization has been one of the most time consuming activities during this circular comparison. Since the system can be left to stabilize overnight after installation, this is not an immediate issue. However, if the stabilization is disturbed substantially (such as re-fixing the cavity ensemble), it requires yet another day. To resolve this, another mode of operation for the temperature stabilization system can be added, one with higher power but larger instability. This would enable the operator to utilize a fast thermal stabilization during setup (when time is of the essence), and a slow stabilization during measurements (when exceptional stability is needed).
- 5. Control software rework (LabVIEW).

- The control software, in its current state, can be difficult to operate for inexperienced staff. In order to enable dissemination of the technology and the technique, it needs to be simplified and optimized.
- 6. Evaluation software rework (Matlab).
 - Similar to the control software, the evaluation software is difficult to execute without prior experience. It should be made more general if possible.

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