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1 Executive summary

This research has enabled the SI traceable measurement of absolute, positive and negative gauge pressures in the intermediate range from approximately (1 to 10^4) Pa with an accuracy level of $3 \cdot 10^{-5} \times p$ + 0.005 Pa in order to increase the efficiency of industrial productions and processes. The work has included the production of primary and transfer standards for dissemination of the pressure scale and developing appropriate calibration methods for high-accuracy state-of-the-art pressure devices in order to establish a calibration service in this pressure range.

SI traceable measurement of absolute, positive and negative gauge pressure in the intermediate range is required for relevant industries such as power plants, cleanroom technologies, petrochemical and pharmaceutical production, storage of nuclear and toxic wastes, in order to support innovation and efficiency in industrial production and processes. Reliable, accurate, traceable pressure measurements are needed for such industries as they are subject to strict international requirements with respect to safety, precision, sterility and performance. Therefore, to ensure traceability of measurements with sufficient accuracy for the demands of industry, high-accuracy primary standards for disseminating the pressure scale in the intermediate range (from approximately 1 Pa to 10^4 Pa) needed to be developed. Low absolute, differential, positive and negative gauge pressure measurements play a vital role in numerous industrial processes that demand high accuracy of positive and negative gauge pressure measurements at all stages of the traceability chain. Conventional calibration procedures (applied to instruments for low differential pressures) are also extremely dependent on weather conditions, especially the stability of atmospheric pressure; and often the target uncertainty level cannot be achieved. Therefore, there was a need for alternative calibration approaches and techniques to ensure a low level of uncertainty, independent of ambient conditions, and for a high-accuracy calibration service. Further to this, the EU mercury strategy includes a comprehensive plan addressing mercury pollution both in the EU and globally. Therefore, support is required for the replacement of primary mercury manometers which are still in use in many research institutions and reference laboratories.

Primary and transfer pressure standards for a consistent dissemination of the pressure scale in the intermediate range (1 to 10^4) Pa were developed based on liquid column manometry and force-balanced piston gauges. A dual Fabry-Perot cavity to measure gas densities by measuring the refractive index was designed and investigated. Calibration methods for positive and negative gauge pressure standards in the range from approximately -100 kPa to 10 kPa were developed. An automated set-up for the calibration of low negative and positive gauge pressure instruments was created. New sensors for differential 100 Torr capacitance diaphragm gauges and associated electronics were designed and tested under different environmental conditions. Alternatives for mercury-containing pressure-measuring devices (barometers, sphygmomanometers) were provided. Calibration service in the range of (-100 to 10) kPa of gauge pressure and (1 to 10^4) Pa of absolute pressure was established.

The project impacts many industries. The reliability and accuracy of low gauge, differential and absolute pressure measurements was improved at National Metrology Institutes, accredited commercial laboratories, in industry and with end users. It has a direct and indirect positive influence on the European economy, environment and society. The project established new primary standards and supports the dissemination of the pressure scale in the intermediate pressure range (1 to 10^4) Pa. This improves the reliability and accuracy of low gauge, differential and absolute pressure measurements at many levels from NMIs, to accredited commercial laboratories, to the end users. The execution of the Commission Regulation which restricts the use of mercury in barometers and sphygmomanometers is facilitated by providing equivalent alternative pressure standards to mercury manometers. It supports the reduction in the number of mercury-containing pressure-measuring devices in Europe. Calibration guidelines for positive and negative gauge pressure standards were drafted to be used by hundreds of calibrating and industrial laboratories. Improved traceability provides basis for more accurate pressure measurement in the cleanroom technologies and processes and will allow realisation of tighter tolerances of non-equilibrium conditions, reduce energy expense and costs. The costs of operation with toxic and nuclear materials as well as of the storage of environmentally dangerous toxic and nuclear wastes can be reduced and the safety of these processes increased. The project established a calibration service that gives access to end-users to calibrations in the range (0 to 15) kPa of absolute pressure with low uncertainties. New measurement capabilities of barometric pressure will be beneficial for more efficient and safe use of airspace by aircraft. Transportable middle vacuum range calibration equipment was created to provide a calibration service at an end user site.

2 Need for the project

Low absolute, differential, positive and negative gauge pressure measurements play a vital role in numerous industrial processes, e.g. in pharmaceutical, petrochemical or energy production industries. The pharmaceutical industry operates in an environment with strict requirements on safety, sterility and precision in accordance with the guidelines on Good Automated Manufacturing Practice (GAMP) of the International Society of Pharmaceutical Engineers (ISPE). To realise cleanroom conditions in semiconductor fabrication, micro- and nanotechnologies or critical product filling systems, differential pressures must be maintained between different rooms or zones in order to prevent contaminated air from entering the critical zone. In hospitals and research laboratories, the pressure difference (negative pressure) prevents the spread of germs, contaminants and dust.

Positive pressure in the filling room maintains the hygiene conditions during filling operations in the food and pharmaceuticals industry [ISO 14644 and ISO 14698]. The pressures in the cleanroom technologies typically range from 100 Pa to 1000 Pa in the makeup and re-circulation units, from 250 Pa to 500 Pa drops through HEPA and 50 Pa to 80 Pa through pre-filters, with an overpressure of approximately 7 Pa to 12 Pa in the cleanroom and 5 Pa between adjacent rooms. Cleanrooms are highly energy intensive to operate – a pressure increase in 1 Pa in a medium size cleanroom requires 3000 kWh additional energy per annum. All the pressures must be monitored continuously and with high accuracy – 1 Pa and lower is the desirable uncertainty target. Maintenance of the required conditions demands high accuracy of positive and negative gauge pressure measurements at all stages of the traceability chain from primary standards through to working standards operated by end users. Conventional calibration procedures when applied to instruments for low differential pressures lead to results which are extremely dependent on particular weather conditions, especially the stability of the atmospheric pressure and as a consequence often the target uncertainty level cannot be achieved. Therefore, alternative calibration approaches and techniques to be applied by NMIs and industrial calibration laboratories needed to be developed, in order to ensure that they would be independent of the ambient conditions.

Chemical and petrochemical industries are subject to strict international requirements like the PED and ATEX [Directives 97/23/EC and 94/9/EC]. Therefore, to meet the safety and performance requirements, reliable, accurate, traceable pressure measurements had to be provided to these industries. Low positive and negative pressure measuring devices are used in fire protection systems as described in international and European legal standards and regulations [FM Approvals LLC, Class No. 2311, April 2008; Commission Regulation (EC) No 1497/2007].

In power plants, pressure measurement, as well as indirect pressure measurement such as flow, plays an important role in safety, efficiency and cost savings. Examples of pressure measurement for safety include avoiding pipe and vessel overpressure situations, inputting measurements for safety instrumented functions such as coolant system leakage detection, actuation of pumps, valves, breakers etc., and preventing release of harmful products, e.g. air pollutants or radioactive substances. In order to support efficiency accurate measurements are required that help to narrow the operating range and keep processes from operating at unnecessary pressures or vacuums.

Differential pressure measurement can be used to indirectly measure flow and level. However, absolute, gauge and differential pressures are measured over a wide range from 0 to approximately 20 MPa in the areas of process control, flow measurement and emissions management. Currently, nuclear power plants are required to calibrate almost all the important pressure transmitters in their primary and secondary systems, including safety-related and non-safety-related transmitters and this task can only be accomplished using high-precision pressure reference standards that are traceable to the national primary pressure standards [EC Nuclear Regulators' Working Group, Final Report – August 2004].

The EU mercury strategy includes a comprehensive plan addressing mercury pollution both in the EU and globally. It contains 20 measures to reduce mercury emissions including a ban on exports of mercury from the EU (that came into force in 2011), and new rules on the safe storage of mercury. A further amendment by the Commission Regulation (EU) No 847/2012 on 19/9/2012 restricts the use of mercury in barometers and sphygmomanometers for industrial and professional use from 10 April 2014 onwards. This regulation poses a serious problem for research institutions and reference laboratories in weather monitoring and forecast services where mercury manometers are still widely used as reference pressure standards, as well as the aviation industry.

The increasing number of aircraft made it necessary to reduce the standard vertical separation in airspace across Europe from 600 m to 300 m (RVSM) between 1997 and 2005. Now only aircraft with specially certified

altimeters and autopilots may fly in RVSM airspace. If the number of aircraft continues to increase, airspace use in the future will require a further refinement of the standard vertical separation and, consequently, a measurement of the barometric pressure that is accurate to 15 Pa, particularly below 30 kPa.

The demands of end users in Europe for traceable pressure measurements are predominantly covered by hundreds of calibrating laboratories accredited by national accreditation bodies. Over the last decade the advanced instrumentation available in accredited laboratories has produced a need for new, more accurate primary pressure standards in the range from approximately (1 to 10^4) Pa to provide traceability of the pressure measurements to the SI units with a required level of accuracy of $3 \cdot 10^{-5} \times p$ + 5 mPa.

3 Objectives

The overall objective of this project was to enable the SI traceable measurement of absolute, positive and negative gauge pressure in the intermediate range from approximately 1 Pa to 10^4 Pa. This had to be done for relevant industries such as power plants, cleanroom technologies, petrochemical and pharmaceutical production, storage of nuclear and toxic wastes, in order to support innovation and efficiency in industrial production and processes.

The specific objectives of the project were:

- To develop and characterise primary and transfer pressure standards - for realisation and dissemination of the pressure scale in the intermediate range 1 Pa to 10^4 Pa. This will enable comparisons with both primary high pressure standards, e.g. dead-weight pressure balances and liquid column manometers, and primary vacuum standards, usually static and continuous expansion systems.
- To develop calibration methods for positive and negative gauge pressure standards in the range from approximately -10^5 to 10^4 Pa - in order to reduce the uncertainty of the pressure calibration down to $3\times10^{-5} p$ + 1 Pa independent of variable ambient conditions, and in industrial conditions to better than $2\times10^{-4} p$ + 3 Pa. This will enable accurate calibrations with a high level of accuracy that is independent of variable ambient conditions.
- To meet the EU restrictions of mercury use in measuring devices (barometers) - replacement of primary mercury manometers with alternative pressure standards.
- To establish a calibration service in the range of approximately -10^5 Pa to 10^4 Pa of gauge pressure and approximately 1 Pa to 10^4 Pa of absolute pressure – with an accuracy level sufficient for accredited calibrating laboratories and industrial companies. This had to be achieved by the development of state-of-the-art pressure measurement instrumentation such as force-controlled piston gauges with a resolution of 1 mPa.
- To engage with industries that utilise pressure in the intermediate range 1 Pa to 10^4 Pa - facilitating the uptake of the technology and the measurement infrastructure developed by the project.

4 Results

4.1 Development and characterisation of primary and transfer pressure standards

Three different techniques were applied to develop and characterise primary pressure standards for low pressure below 100 kPa: liquid column manometry, force-balanced piston gauges and refractometry.

A new interferometric liquid column micromanometer for 2 kPa range of absolute and gauge pressure was developed. Force-balanced piston gauges of FRS and FPG type, operated in the range 15 KPa of absolute and gauge pressure, were characterised as primary pressure standards by applying dimensional measurements on their piston-cylinders and state of the art flow modelling. A dual Fabry-Perot cavity (DFPC) to measure gas densities by measuring the refractive index was designed and investigated, which, after further studies, can serve for a primary realisation of the pressure unit in the range of about 100 kPa of absolute pressure.

4.1.1 Development of an oil-based interferometric micromanometer as a primary absolute and gauge pressure standard

The scope of this work was to develop a new oil-based U-tube interferometric micromanometer, assembled at PTB, with *in-situ* density measurement as a primary standard of absolute and gauge pressure for the pressure range from 1 Pa to 2 kPa.

Design of the interferometric oil-based micromanometer

The over-all design is visualized in Fig. 4.1.1.1. Here, the key components are clearly visible: the limbs of the manometer U-tube are filled with vacuum oil as the manometric liquid.

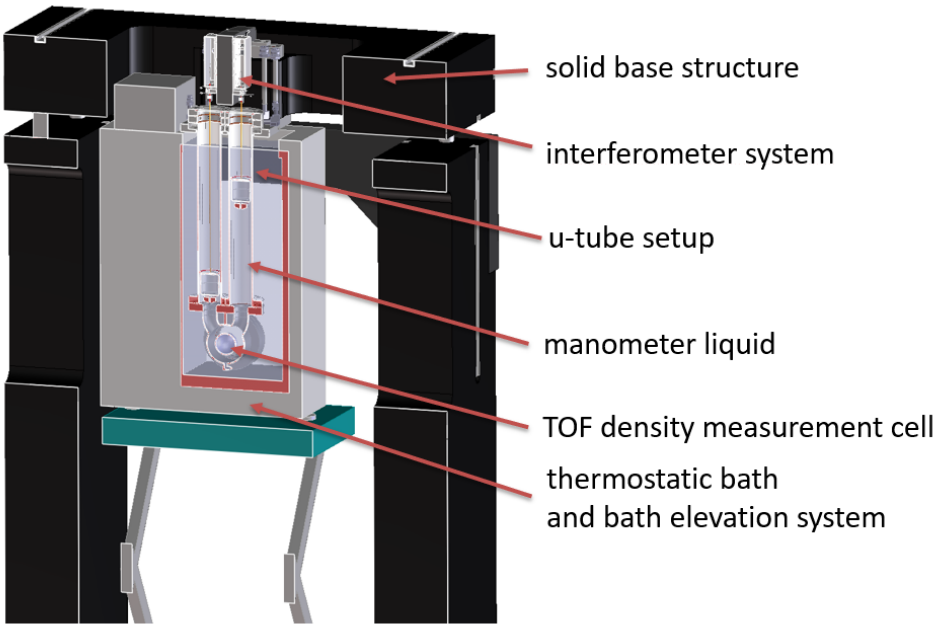

Figure 4.1.1.1: Key components of the micromanometer

The mechanical parts are attached to a solid-base structure on top made of granite which contains a levelling system and the interferometer retainer to fix a twin-differential interferometer system. The U-tube body consists of deep-drilled main tubes with contains welded vacuum flanges and an oleophobic inner coating. The temperature-of-flotation density measurement cell is integrated directly into the U-tube as a link. This cell contains a Ti hollow sphere as a density standard which acts as a float whose position depends on the thermodynamic equilibrium situation inside the cell directly coupled to the U-tube. The U-tube is soaked into the tank of a commercial temperature bath. An additional cooling bath and improved non-commercial temperature control shall ensure a temperature homogeneity and stability of better than 3 mK at the reference temperature. The temperature bath is elevated by a non-hydraulic spindle lift table. In the following, there is a list of components which have a significant influence on the performance of the device, and hence, on its measurement uncertainty.

The design of the micromanometer, see Fig. 4.1.1.1, is based on the classical layout of a Torricelli manometer. The pressure in the reference column 1 is denoted p_1. In absolute mode, column 1 is pumped continuously, hence p_1 indicates the residual pressure in this column which is strongly related to the vapour pressure of the manometric oil. In the gauge mode, column 1 is open to atmosphere, hence p_1 is equal to the atmospheric pressure. The pressure applied to column 2 induces a vertical displacement of the surface positions h_1 and h_2 in the two columns, see Fig. 4.1.1.2. The related equation for the hydrostatic pressure in such a U-tube manometer is as follows:

$$p_2 = \rho_{FI} \times g \times h + p_1, \qquad (4.1.1)$$

where g denotes the well-known (from direct measurement) gravitational acceleration, h the displacement of the oil surface levels of the two columns of the U-tube and p_1 and p_2 being pressures within columns 1 and 2, respectively.

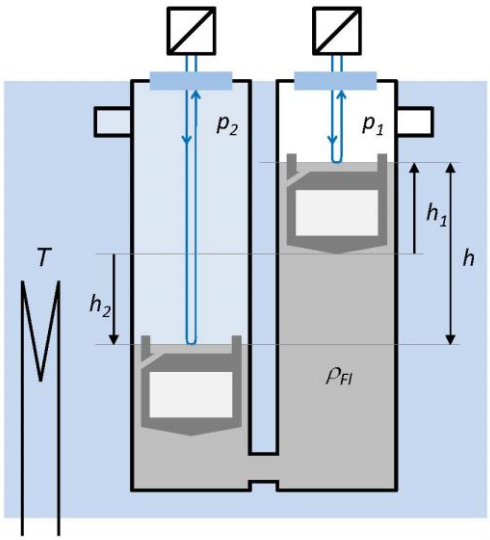

Figure 4.1.1.2: Block diagram of interferometric length determination for a U-tube manometer

Manometric liquid

As can be seen from equation (4.1.1), the manometric liquid properties enter twice: first via the density of the oil and, for the absolute mode, via the residual pressure which strongly depends on the vapour pressure.
A broad market analysis of different oils suitable as manometric liquids was performed by PTB and TUBITAK. Vapour pressure, oil density and viscosity as important materials properties were identified. The request for low vapour pressure set the focus on vacuum oils, of which, in a first step, seven oils were selected [1].
In a second step, three vacuum oils were chosen for detailed characterization. Criteria for selection were the combination of low vapour pressure and viscosity. In the following, relevant material properties are discussed and the main results of characterization presented which will have a significant impact on the measurement uncertainty.

(i) Vapour pressure
When operating the liquid-column manometer in absolute mode, the vapour pressure of the manometric fluid should enable a sufficiently low residual pressure, to avoid non-equilibrium conditions and to minimize local pressure gradients in the reference column. To have no significant effect on the target uncertainty, the vapour pressure should be lower than the expected uncertainty of the lowest measurable absolute pressure by one to two orders of magnitude. In our particular case, it should be less than 0.1 mPa.

(ii) Density of manometer liquid
The coefficient dh/dp being inversely proportional to the product of density and gravity acceleration defines the density range for the manometric liquids. A liquid with a lower density is advantageous for achieving a higher sensitivity of displacement with respect to pressure changes. However, the idea of using fluids with a very low density to maximize the sensitivity to pressure chances is limited, in particular, due to geometric and thermal restrictions. For a pressure range up to 2 kPa, a tolerated density interval between 700 kg/m³ and 1500 kg/m³ would allow a column height difference of 14 cm to 30 cm. The hydrostatic pressure as well as the pressure to be measured have to be considered for the manometric liquid density. Its compressibility known from literature is typically for vacuum oils around 1 ppm/kPa (1 ppm = 10^{-6}). Besides, the thermal expansion coefficient of vacuum oils has to be taken into account with approx. 1 ppm/mK. With this relatively large value, spatial and temporal temperature gradients have a noticeable effect on the fluid density. Hence, thorough temperature stabilization and homogenization of the apparatus within 3 mK has to be fulfilled.
The density and the thermal expansion of the candidate oils were determined at IPQ with a DMA 5000 oscillation-type densimeter made by Anton Paar. To determine the thermal expansion of each fluid, three cycles of density measurement were performed in the temperature interval from 15 °C to 25 °C in steps of 0.5 °C. The thermal expansion coefficient γ, at 20 °C, was given by the quotient of the first derivate of the curve $\rho(T)$, in kg/m³/K, to the density ρ, at 20 °C, in kg/m³.
To obtain the correct density value, the resonance frequency measured by the densimeter needed to be corrected for the oscillation damping effect arising inside the oscillating U-tube, which depended on the liquids'

viscosity. All the densities were corrected for this effect by applying the calibration equation of the density error against the viscosity obtained with reference mineral oils, which is specific to the densimeter used. For this reason, it was necessary to determine the viscosity/temperature dependence of the liquids in the temperature interval from 15 ºC to 25 ºC, which was done using a Stabinger viscometer made by Anton Paar. All kinematic viscosities presented in Table 2 were measured with Ubbelohde-type capillary viscometers at 20 ºC and at atmospheric pressure.
A hydrostatic weighing density meter was used at PTB to obtain more accurate density values with lower uncertainty (Table 4.1.1.3). Here, a silicon sphere of well-known density is suspended under a mass balance inside the candidate oil measuring the buoyancy force. Compressibility was measured using an oscillating U-tube densimeter for higher pressure (DMA HP made by Anton Paar) at pressures between 1×10^5 Pa and 20×10^5 Pa. It is assumed that the measured compressibility values can be extrapolated down to a pressure of order 10 Pa.

Table 4.1.1.3: Fluid density (ρ) and compressibility (β) with standard uncertainties at 20 °C and standard pressure.

Fluid	ρ / (kg/m³)	β / (10^{-6} kPa^{-1})
F02: 1,1,3,5,5-Pentaphenyl-1,3,5-trimethyltrisiloxane	1095.264 ± 0.003	0.637 ± 0.016
F06: Multi-ring polyphenyl ether	1178.453 ± 0.003	0.553 ± 0.014
F07: Mixture of synthetic hydrocarbons	829.227 ± 0.003	0.836 ± 0.021

For liquids F02 and F06, densities obtained with the oscillating-type densimeters and hydrostatic weighing technique are in a very good agreement within their uncertainties. For liquid F07, the difference is 6 times the uncertainty of the oscillating-type measurement, which requires further clarification. With the accuracy of the hydrostatic weighing results, a pressure measurement would be possible with an uncertainty of nearly 2 ppm if the density was stable and all other parameters were measured with sufficient accuracy.
Since the free surfaces of the manometric fluid are in direct contact with the pressure-transmitting gas medium, gas is expected to be absorbed by the fluid and may also desorb into vacuum in the reference column, as well in the measurement column when the pressure later decreases. Therefore, it was necessary to have accurate data on gas absorption and desorption kinetics for our candidate vacuum oils. To quantify the influence on the density, a first density measurement was performed with fluids F02, F06 and F07 in the as-delivered state by using an oscillation-type densimeter DMA 5000 made by Anton Paar. It was expected that the vacuum oil was saturated with typical air components like nitrogen, oxygen, argon and water vapor, but in addition, a certain number of by-products of the production process was assumed in the liquid. Because of the non-standard application of the liquids this information is not specified by the manufacturer. As a second step, the oil was degassed by heating to 110 °C for 5 h/day and simultaneously evacuated to 0.2 kPa in a vacuum oven over a period of one, two and three days, see results in Fig. 4.1.1.4.

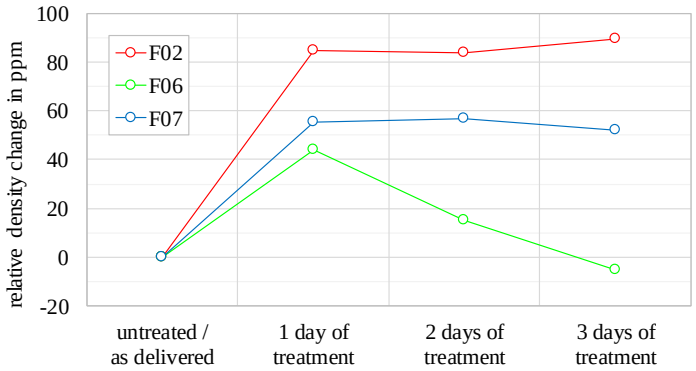

Figure 4.1.1.4: Relative change in density when applying a degassing treatment over one to three days.

It was found that the treatment procedure increased the density by up to 90 ppm. Especially during the start of the evacuation step, in all samples small bubbles were released, which disintegrated with time. Even one day of degassing under the conditions used leads to nearly complete removal of the gas from the liquid, corresponding to the thermodynamic equilibrium; an increase of the degassing time to two or three days does not produce any significant effect on density. F06 behaves differently which may be caused by impurities as part of the production process of the liquid. Or, the assumption of having gas saturated conditions during the as-delivered state did not apply to that liquid. The process of gas saturation at atmospheric conditions takes a

long time; even more than three days after exposure to the atmosphere, a full gas saturation was not reached and the density was by 20 ppm higher than in the as-delivered state. Within the same time, F06 changed its density in contact with air by -100 ppm, which supported the thesis of partly saturated start conditions or the existence of other volatile components.
To gain knowledge about the absorption and desorption dynamics of particular gases in the candidate liquids, measurements were performed at IMT using a quadrupole mass spectrometer (QMS) integrated in a high vacuum setup with a turbomolecular pump. As a first result, it could be shown that, even under high vacuum conditions, for all candidate fluids taken in their as-delivered state it was not possible to reach the level of vapour pressure declared by the manufacturer. The mass spectrometer showed an eminent proportion of higher mass molecules, i.e. hydrocarbons. These QMS signals decreased with time, indicating that a thermal treatment with 40 h of pumping of the oils would remove the hydrocarbons. To investigate the gas absorption and desorption dynamics, the oils were brought in contact with nitrogen, argon, oxygen, helium or water vapor at a pressure of 100 kPa for three hours, and after heavy pumping the quadrupole mass spectrometer was connected to the vessel to detect gases desorbing from the fluid.
From the ion current of the QMS, the emerging gas flow was estimated applying a purely diffusional transport process model of gas in liquid. By applying a numerical model using the finite element method (FEM), gas back dissolution into the fluid sample was taken into account as well. As a result, the diffusion constant D, gas permeability K and gas solubility S were determined for each gas at 25 °C. With this data, the calculation of the gas distribution and, consequently, of the density distribution along the columns of the micromanometer in dependence on time after a pressure change became possible.

(iii) Viscosity and capillary properties

When the micromanometer is subjected to a pressure load, the liquid columns inside the U-tube begin to move until equilibrium is achieved between the hydrostatic pressure and the new pressure difference.
This involves the whole mass of the liquid in the U-tube; the time to reach the required level of equilibrium (a perfect equilibrium would require an infinitely long time) depends, in addition to the U-tube geometry and the liquid density, on the liquid viscosity. Hence, the dynamic viscosity should lie between 830 mPa·s and 1790 mPa·s for oil densities ranging from 700 kg/m^3 to 1500 kg/m^3 and a column diameter of 6 cm. Flow restrictions like valves or flanges could easily shift the values because of the quadratic contribution of the column diameter d. Nevertheless, for the defined conditions within the density range from 700 kg/m^3 to 1500 kg/m^3 and the dynamic viscosity range from 50 mPa·s to 2000 mPa·s, the time to reach conditions where $\delta p < 0.001$ Pa would never be longer than two minutes.
Despite of the viscosity, one has to take the influence of capillary effects into account. When pressure changes, the oil wets the material of the tube and some amount remains on the tube wall in the form of a thin layer, which needs time to flow down. Due to a delay in the liquid exchange between the columns, the oil creeping in one of columns causes a drift of the interferometer signal and produces a deviation in the measured pressure. The volume flow rate in the wetting layer can be derived from the equality of viscous and gravitational forces. Besides, one has to consider the influence of wetting in more detail: if wettability of the tube material is not perfect, and if the oil builds up a non-zero angle with the tube wall equal to φ, a meniscus with a curvature radius equal to $a/(1-\cos\varphi)$ takes place at the upper end of the liquid layer inducing a capillary pressure which, in addition to the gravitational force, will cause the liquid to flow down.
Our estimations of this effect assuming a surface tension value of $\sigma = 30$ mN/m and $\varphi = 56$ showed that, for liquids with viscosity ranging from 24 mPa·s to 3000 mPa·s, a deviation of $\delta p = 0.1$ Pa is achieved in reasonable times of 1 minute to 4 minutes. This calculation demonstrated that liquid capillary properties are very important for a fast stabilization of oil surfaces, and materials with a low wettability by the candidate oil are preferable. In addition, this knowledge is used to design the floats' walls such that the oil surface remains nearly flat. Hence, for optimizing the stabilization dynamics of the surfaces of our candidate oils it was necessary to investigate experimentally their surface tension and wetting behaviour in contact with polytetrafluoroethylene (PTFE), as PTFE is considered as a material of the floating pools for stabilization of the liquid-free surfaces and also as a material to coat the inner surfaces of the micromanometer's U-tube to reduce wettability. A Lauda TE3 tensiometer was used, which realizes the static and dynamic Wilhelmy plate method for the measurement of surface tension, contact angle and contact angle hysteresis. It could be seen that, when comparing the angles of the static and the dynamic method, the angles of the dynamic method were always smaller. This might be caused by remaining liquid on the PTFE surface after the first immersion, which improves the wettability in the following cyclic measurements. The measured hysteresis of the wettability was remarkable. This strong effect might have to do with the surface roughness and surface material inhomogeneity, which can be relevant especially in the case of poorly wetted materials. It is known that such surface inhomogeneity can result in reducing dynamic contact angles.

Interferometer system

The interferometric length determination also enters directly via the displacement *h* in equation (4.1.1). The position of the oil surface in each limb is measured individually by a differential interferometer with plane mirror reflector. The interferometer is attached to a retainer inside a granite solid-base structure. To this solid-base, the U-tube construction is fixed as well. This construction has a large stiffness and mass/inertia, both to prevent mechanical tilts or coupling to mechanical vibrations and also to have an identical thermal behaviour as the stainless steel, from which the U-tube and the interferometer casing are made. The interferometer beams cross a glass plate, which has a mirror for the reference beam on the back side. This reference beam is used for compensating environmental influences in air. The reference mirror is located close to the reflector of measurement, the oil surface. Hence, measurement and reference beam suffer from the same environmental influences, and, therefore, the effect of the dead path length is minimized. Moreover, the symmetrical layout serves for a good long-term stability. Due to the strong coupling of the interferometer to the U-tube construction, a very good thermal stabilization is guaranteed, since the U-tube is immersed into a temperature-controlled bath.
The interferometer detects the surface of the manometric liquid, i.e. the oil, directly, and, for this reason, the oil surface is prepared in a float creating a low-depth swimming pool in order to damp surface waves. A rough, non-stable oil surface would disturb back-reflection of the measuring interferometer beam. The inner part forms the pool filled with oil of 2 mm thickness. To adjust the thickness of the oil layer within the pool to a desired value, a tare weight is attached to the buoyancy body. The design of the walls of the pool floats incorporates the contact angle of the oil to the cap material in order to form a horizontal oil surface at the contact line and, thus, to build a flat surface. The commercial interferometer system applied has a resolution of better than 1 nm equivalent to 0.01 mPa and a relative frequency stability of better than 10^{-9}. Preliminary tests of the fitness-for-use could be done by testing simultaneously the interferometer configuration in a testing installation using float cups filled with oil in a glass vessel. A more accurate test of the performance of the interferometer system has been carried out, which even allowed the precise measurement of the position of the oil surface in normal laboratory conditions without special thermalization of the interferometer or housing of the set-up. It was possible to detect a stable signal over 12 hours with an interferometer drift of less than 30 nm meaning a pressure stability of better than 0.25 mPa. With a better insulation from the surrounding and improved alignment of the interferometer beams, the spikes on the interferometric signals could be reduced to lower than 1 nm and the drift within 20 minutes (as a typical time scale for a measurement) was less than 5 nm.

In-situ density-measurement

The temperature-of flotation method used here is based on the buoyancy of a float body (sinker) with respect to the surrounding liquid medium, i.e the manometric oil inside the measurement cell which is in thermodynamic equilibrium with the oil in the U-tube. The sinker is lighted by an LED, and its position is detected by a photodiode. A PC with a PID controller regulates the temperature of the liquid to reach a flotation state of the sinker. At this state and at constant temperature and pressure, the liquid in the density-measurement cell has the same density as the sinker, whose density is known with high accuracy.

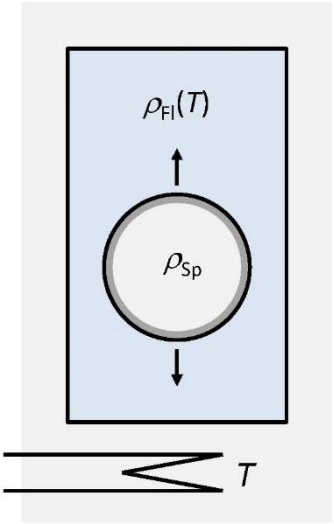

Figure 4.1.1.5: Sketch of the temperature-of flotation method.

The sinker used in the micromanometer is a density standard, which is a hollow sphere made of titanium having appropriate density and geometry, i.e. a diameter of 58 mm and a volume of approx. 100 cm^3. In order to adjust the density of the oil to that of the Ti-sphere in the cell, the temperature is varied untill equilibrium is realized and the sphere floats, i.e. doesn't move up- or downwards. For the intended uncertainty of density measurement of better than 1 ppm by the temperature-of-flotation method, temperature variations within ±0.1 K around the reference temperature and stability and homogeneity of the temperature of better than 3 mK are required.

Measurement uncertainty

(i) Following equation (4.1.1), the interferometric length determination has a major impact on the measurement uncertainty. The interferometric length depends on several input quantities. First of all, the refractive index of air, which is calculated using the Edlen equation, effects the measurement of the distance between the interferometer and the reference mirror on top the U-tube columns. The uncertainty contribution due to the refractive index in air is 6.7 nm. As mentioned earlier, the relative frequency stability $u(f)/f$ of the He-Ne laser used in the interferometer is better than 10^{-9}. Hence, for a measurement length of 0.12 m, the contributing measurement uncertainty to the length measurement is 0.12 nm. Other sources of uncertainty are due to demodulation and rounding deviations. They are in total in the order of $u(l_{dem})$~0.5 nm. Moreover, uncertainty sources arising from geometrical effects are due to tilt of the whole setup. This contributes 5.7 nm to the measurement uncertainty. The resulting standard measurement uncertainty from interferometry is therefore 8.8 nm.

(ii) Another important contribution to the uncertainty arises from the density measurement. Quantitative estimations of the uncertainty in the density measurement realised within the special concept of the new micromanometer are as follows.
A key uncertainty source in the measurement of the oil density is characterisation of the density standard. The volume of the sinker can be determined by hydrostatic weighing in comparison with silicon density standards (uncertainty about 0.1 ppm). Standard uncertainty of comparison by hydrostatic weighing is below 1 ppm for the density of a 100 cm^3 sinker. A second issue is the high thermal expansion coefficient of the vacuum oil (about 1000 ppm/K, thus, temperature should be constant in time and space within a few mK). The thermal expansion of the sinker must be taken in consideration too: within a temperature range of ±0.1 K, an uncertainty of a few (approx. 5) ppm/K in the thermal expansion coefficient is expected. The compressibility of the sinker in the pressure range of 100 kPa shall contribute an uncertainty of about 0.05 ppm/kPa. The compressibility can be determined with the manometer itself. Therefore, one ends up with the uncertainty of 5 ppm.

(iii) In the uncertainty analysis, also the uncertainty of the gravity acceleration should be taken into account, which is smaller than 1 ppm. Besides, the contribution of vapour pressure should be considered. Since all the investigated candidate oils have a vapour pressure in the order of 10^{-2} mPa or lower, this contribution is negligible. Combining it with the estimations of the uncertainty of the height difference and density measurements, the final, combined uncertainty of the pressure measurement is 1 mPa + $2\times10^{-5}\times p$.

Conclusion

The concept, design and construction of the new PTB oil-based interferometric micromanometer serving as a primary standard are presented. Main uncertainty sources were identified and prerequisites for operation of the micromanometer at the required uncertainty level derived. The key impact factors on the uncertainty, namely interferometric determination of the position of the floats, temperature homogeneity, stability of the density and residual pressure of the manometric oil, were identified and their uncertainty contributions estimated.
Requirements for fluids suitable as manometric liquids were set and appropriate oils were selected. Their characterization was performed, their properties with associated uncertainties were determined and their contributions to the pressure measurement uncertainty estimated. This involved such material properties of the candidate oils as viscosity, compressibility and thermal expansion. The gas saturation behaviour of the density of the oils was characterized, as well as gas diffusion kinetics and gas solubility.
The requirements for the interferometric length determinations were formulated and the interferometer performance evaluated. The float design was developed and floats fabricated. Furthermore, an estimation of the performance of the floats for the determination of the interferometric measurement of the surfaces of the manometric liquids was carried out. Although an over-all design of the layout of the interferometric micromanometer has been finished, the solid-base structure is still being manufactured and the mechanical parts are being delivered.

A comprehensive analysis of the uncertainty sources was performed. The main uncertainties to be taken into account are:

- the vapour pressure of the oil used as a manometric liquid,
- density, compressibility and thermal expansion coefficient of the oil, as well as saturation capacity and kinetics of gases containing in the pressure transmitting medium and air,
- viscosity and capillary properties of the oil,
- *in-situ* measurement of the oil density,
- interferometric measurement of the liquid columns' lengths,
- positional instability of the U-tube and the interferometric system.

In summary, the residual gas pressure dealt with the oil vapour pressure has an uncertainty below 0.1 mPa. The density uncertainty related to the oil properties and realisation of the temperature-of-floatation method contributes less than 10 ppm. The uncertainty of the difference of the liquid columns' heights is below 10 nm, corresponding to 0.1 mPa uncertainty in pressure.
Herewith, the combined uncertainty of absolute and gauge pressure measured with the new micromanometer is smaller than 1 mPa + $2\times10^{-5}\times p$.

4.1.2 Development of FPG and FRS piston gauges as primary and secondary absolute and gauge pressure standards

The aim of this research was to establish Force-balanced Piston Gauges (FPG) and Furness Rosenberg Standard (FRS) piston gauges as primary and secondary absolute and gauge pressure standards in the pressure range from approximately 1 Pa to 15 kPa with a target uncertainty below 0.01 Pa + 1.4 10^{-5} p. These instruments are widely used by NMIs and accredited calibration laboratories, and their low uncertainty capabilities have been demonstrated in a EURAMET comparison. However, their SI traceability in Europe was based on a calibration against primary pressure standards above approximately 5 kPa and an extrapolation at lower pressures.
In contrast, classical dead-weight pressure balances have a well-established method of primary evaluation by the Dadson theory, and they are evaluated only based on empirical models. The problem with FPGs is their specific design, which makes modelling of the gas flow in the piston-cylinder gap extremely difficult. The lack of appropriate piston-in-cylinder adjustment techniques in the case of FRS piston gauges and of suitable fluid flow models for the piston-cylinder geometry as well as boundary conditions in the case of FPGs makes their primary characterisation very challenging, which prevented their full utilisation at pressures below 5 kPa.
A key challenge in this research was the gas flow modelling taking into account the molecular properties of the gas as this is particularly important for absolute operation mode when a gas becomes rarefied. The work included:

- Development of methods for the reliable and reproducible alignment of the piston in the cylinder of a FRS piston gauge.
- Dimensional measurements of the piston-cylinder geometries of FPG and FRS piston gauges.
- Modelling of the gas flow in the piston-cylinder gap of FPG and FRS piston gauges with various pressure modes and variable pressure ranges conditions, in order to provide a pressure distribution along the piston-cylinder gap. From the results the effective area of the piston-cylinder assemblies (PCAs) are calculated as a function of pressure and from this the primary characterisation of the FPG and FRS piston gauges comparable to that of the classical dead-weight pressure balances will be determined.
- Validation of the FPG and FRS piston gauges as primary and secondary absolute and gauge pressure standards.

The results of this study are published in [2, 3].

CMI tested the manufacturer's method of centring using the feeler gauges and developed and tested two new methods – a capacitance method and a lower illuminance method. The reached reproducibility of effective area is at least twice better than the anticipated value (80 ppm). A detailed description of the whole method with an instructional movie was distributed among relevant partners. PTB applied the capacitance centring method to its FRS.

Dimensional measurements on FPG piston-cylinders were carried out at PTB, INRIM, RISE and CMI. Dimensional measurements on FRS piston-cylinders were performed at CMI and PTB. All dimensional data of FPGs and FRSs were sent to UTH for gas flow modelling.

Numerical tools to simulate the gas flow in the gap of piston-cylinder assemblies (PCA) of FPG and FRS gauges and to calculate their effective area were developed and established. More specifically, based on flow modelling, the pressure distribution along the gap of both configurations as a function of the operational mode, the operational pressure and the dimensional data of the PCA was determined and the effective area was

computed. The implemented numerical codes and tools are based on a) typical hydrodynamics theory, including the Navier-Stokes formulation and Dadson approximation and b) kinetic modelling as described by the Boltzmann equation and reliable kinetic model equations.

Kinetic and viscous flow's software for the simulations of the piston-cylinder gap flow was developed independently by UTH and PTB. UTH performed several benchmarking tests of the two developed codes to verify their accuracy. In addition, typical CFD software based on the Navier-Stokes equations was used for validation of the developed software in the hydrodynamic regime.

Then, the developed software was extended and enhanced to introduce the real dimensional data and any set of inlet-outlet pressure values. The FPGs owned by PTB, RISE and INRIM were simulated, and simulations were performed for the FRS4 owned by CMI and the FRS5 owned by PTB. The gas flow inside the piston-cylinder gap was simulated in both gauge and absolute mode, for 11 pressure values between 10 Pa and 15 kPa for the FPG and another 11 pressure values between 10 Pa and 11 kPa for the FRS4/FRS5. Effective area was determined using kinetic theory, Dadson theory and CFD [3]. Dadson and CFD results showed an excellent agreement for all investigated PCAs in the whole operating pressure range, thus validating the high accuracy of the fully-developed and one-dimensional flow assumption used (Fig. 4.1.2.1). Comparison between the different approaches showed good agreement between the kinetic (BGK) and viscous (Dadson/CFD) results in the continuum regime. However, as the operating pressure is reduced, the viscous approaches fail and cannot provide reliable results, thus clearly indicating the need of kinetic modelling.

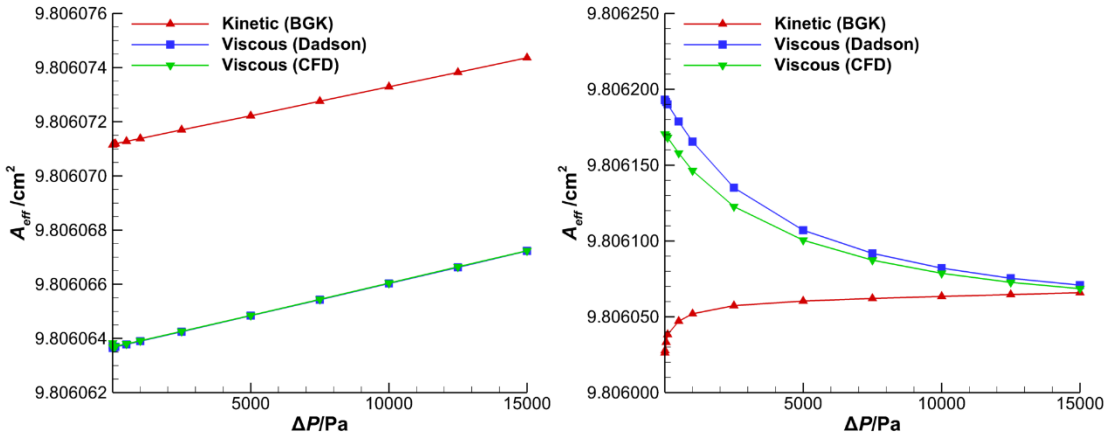

Figure 4.1.2.1: Effective area of FPG8601 of PTB for gauge (left) and absolute (right) mode.

The effective areas of all investigated FPG and FRS PCAs were exchanged between UTH and PTB. The results of UTH and PTB based on kinetic and Dadson theory for the FPG and FRS5 owned by PTB were compared with success in the whole operational pressure range. The kinetic results of all PCAs were examined and the dependence of the effective area on various parameters was investigated, including the dimensional measurements of the device, the accommodation coefficient of the piston and the cylinder surfaces, the temperature, the gas humidity content, as well as numerical parameters such as the collision model and the numerical scheme. In all cases, the variation of the effective area was proved to be negligible and only the uncertainties related to the dimensional measurements and the accommodation coefficient had some effect. In all cases, however, this variation was found to be less than 3 ppm and only for one FPG (RISE) and one FRS (CMI) device it reached 9 and 7 ppm, respectively, which is mostly due to the uncertainty of dimensional measurements. Finally, the possibility of extrapolating the effective area results above 5 kPa to pressures between 5 kPa and 10 Pa was investigated. Using a second order polynomial, it was concluded that for all devices the maximum deviation in the gauge mode is less than 0.1 ppm while in the absolute mode, it is less than 9 and 8 ppm for the FPG and FRS devices, respectively. Therewith, three FPG and two FRS devices were examined, and their uncertainties were determined as given in Table 4.1.2.2.

Table 4.1.2.2: Theoretical standard uncertainty (in ppm) for all PCAs at maximum operation pressure in the gauge (g) and absolute (a) mode.

PCA	FPG8601 (PTB)		FPG8601 (RISE)		FPG8601 (INRiM)		FRS4 (CMI)	FRS5 (PTB)	
Mode	g	a	g	a	g	a	g	g	a
Dimensional uncertainty	1.07	1.07	8.00	8.00	1.04	1.04	6.26	0.57	0.57
Acc. coef. (α = 0.9)	0.11	0.35	0.47	1.20	0.38	1.13	0.00	0.00	0.17
Acc. coef. (α = 0.8)	0.27	0.75	1.02	2.44	0.81	2.31	0.01	0.00	0.30
Temperature change (±1 K)	0.00	0.01	0.01	0.05	0.01	0.05	0.00	0.00	0.01
Temperature change (±2 K)	0.01	0.03	0.02	0.11	0.02	0.10	0.00	0.00	0.02
Model equation (BGK vs BE)	0.03	0.05	0.11	0.25	0.09	0.19	0.00	0.00	0.03
Relative humidity (50 %)	0.00	0.01	0.01	0.04	0.01	0.04	0.00	0.00	0.01
Total uncertainty	1.11	1.31	8.07	8.38	1.32	2.55	6.26	0.57	0.65

Concerning the theoretical estimation of the effective area and the reduction of the uncertainty, the most critical parameter is the uncertainty of dimensional measurements. In the whole operational pressure range, including the maximum operational pressure, the uncertainty was much smaller than the target uncertainty of 15 ppm reaching a maximum value of 9 ppm in the worst case.

For the experimental study of FPG and FRS piston gauges against classical dead-weight pressure balances, liquid column manometers and primary vacuum standards based on static expansion and continuous flow techniques, a calibration protocol was produced. CMI, TUBITAK, CEM, PTB, LNE, INRIM and RISE each calibrated their FPG against their dead-weight pressure balances in both gauge and absolute mode above 2 kPa up to 15 kPa. LNE calibrated their FPG against their national reference APX and PG 7607 comprising a piston-cylinder assembly of 20 cm² cross-sectional area using absolute, gauge and absolute differential operation modes. CMI and PTB each calibrated their FRS against their dead-weight pressure balances in both gauge and absolute mode above 2 kPa. TUBITAK characterised their FPG by calibrating a 100 Torr capacitance diaphragm gauge (CDG) and a digital pressure gauge in gauge mode up to 15 kPa by the FPG and a dead-weight pressure balance. INRIM calibrated their FPG against INRIM dead-weight pressure balances in both gauge and absolute mode up to 15 kPa.

CEM calibrated their FPG against their mercury manometer in the range 1 kPa to 15 kPa in gauge mode. PTB calibrated their FPG and FRS piston gauge against their mercury manometer above 100 Pa up to their maximum operation pressure (15 kPa and 11 kPa) in both gauge and absolute pressure mode.

In the low-pressure range, CMI calibrated their FPG against a continuous flow apparatuses in absolute pressure mode below 10 Pa. PTB calibrated their FPG against PTB's static expansion system in absolute pressure mode below 100 Pa. LNE compared their FPG with a CDG and a spinning rotor gauge (SRG) to validate the FPG behaviour at pressures below 1000 Pa. CEM calibrated their FPG against CEM's static expansion system in absolute mode in the pressure range 10 Pa to 5 kPa. TUBITAK calibrated a 10 Torr absolute CDG and SRG by their FPG and static expansion system. CEM studied the influence of the lubrication pressure on the initial conditions of the FPG.
An example of comparison of the theoretical and experimental effective areas for the FPG8601 of PTB [2] is presented in Fig. 4.1.2.3. It shows all effective areas to agree well with each other on the level of the experimental uncertainties.

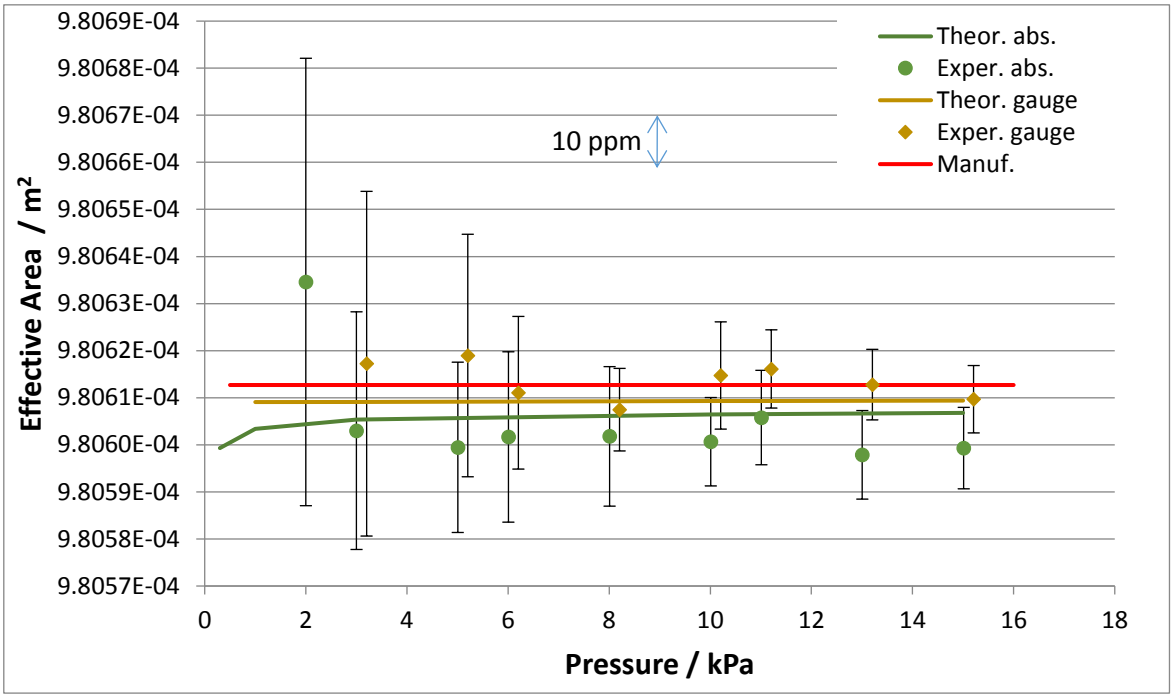

Figure 4.1.2.3: Effective area of FPG8601 of PTB for gauge (left) and absolute (right) mode.

Conclusion

The effective area of three FPG8601, one FRS4 and one FRS5 in the whole operational pressure range of each device varying between 1 Pa and 15 kPa was computed. Since the flow is under rarefied conditions, modelling has been based on the BGK model equation, while the typical Dadson and CFD approaches have been complimentary applied only in the viscous regime.

It was found that the effective area is strongly affected by the geometry of the device and the flow conditions, and its estimated value, as well as its dependency on pressure, may be different even for devices of the same type. Furthermore, an uncertainty analysis was performed. The main source of uncertainty are the dimensional measurements on piston and cylinder, followed by the accommodation coefficient characterizing the gas-surface interaction, while the effect of other flow and modelling parameters is negligible. In all cases, the total uncertainty of the effective area was found to be less than 10 ppm indicating that pressure measurements of high accuracy can be ensured. Since the effective area is computed based solely on simulations, the FPG and the FRS piston gauges characterized in this way can serve as primary pressure standards.

These results are in full agreement with the experimental effective areas obtained by cross-float and with the areas reported by the manufacturer. As a result of this characterization, it was possible to reduce the relative standard uncertainty of the effective area to below 10 ppm.

With the theoretical and experimental characterisation of the force-balanced piston gauges as primary and secondary standards, the pressure uncertainty below the target uncertainty of 0.01 Pa + 1.4 10^{-5} p, was achieved.

4.1.3 Development and characterisation of a transfer pressure standard for the dissemination of the pressure scale in the intermediate range between high pressure and vacuum

The objective of this work was to develop, build and evaluate a pressure transfer standard (TS) in the range 1 Pa to 10 kPa. It is expected to be used by either national institutes of metrology or calibration services. Its targeted uncertainty was $1\times10^{-4}\times p$. LNE has developed and built the transfer standard which is composed of a commercially available resonant silicon gauge (RSG) of 130 kPa full scale in absolute pressure, and three capacitance diaphragm gauges (CDGs) of 130 Pa, 1.3 kPa and 13 kPa full-scale, respectively [4]. The results of measurements with the transfer standard performed and the comparison protocol prepared by LNE were evaluated by the comparison's participants: FCT-UNL, CEM, IMT, TUBITAK and LNE.

Description and principle of the transfer standard

Capacitance diaphragm gauges have been used as low absolute pressure transfer standard (typically below 1 kPa), because they combine a high resolution with a repeatability of a few $10^{-5}\times p$. However, these gauges suffer from poor mean-term reproducibility, especially when transported, between about $5\times10^{-4}\times p$ and $2\text{-}3\times10^{-3}\times p$. This issue can be overcome by rescaling the CDGs with a very stable resonant silicon gauge, as it was done in the comparison CCM.P-K4.2012. Rescaling means correcting the drift of the slope of the CDG by the ratio k_{CDG} of the output pressure signals of the RSG and that of the CDG. Unfortunately, such accurate RSGs are hard to find. LNE has applied a similar method but instead of determining k_{CDG} from one measurement point (usually the CDG's full scale), it was determined from four pressure points so as to disseminate the slope of the RSG's calibration curve over the CDG 10 kPa full-scale. The latter can be then used in turn to rescale the CDG 1.3 kPa full-scale which is finally used to rescale the CDG 130 Pa full-scale. As the slope of the RSG's calibration curve was found to be highly stable from LNE's calibrations over about ten years in the range 10 to 130 kPa (drift less than 10 ppm), the uncertainty contribution of the transfer standard should meet the objective. The set-up of the transfer standard and its drawing with dimensions are shown in Fig. 4.1.3.1. Differential CDGs were used instead of absolute ones because they were found to be in general more stable on the mean-term from LNE's experience.

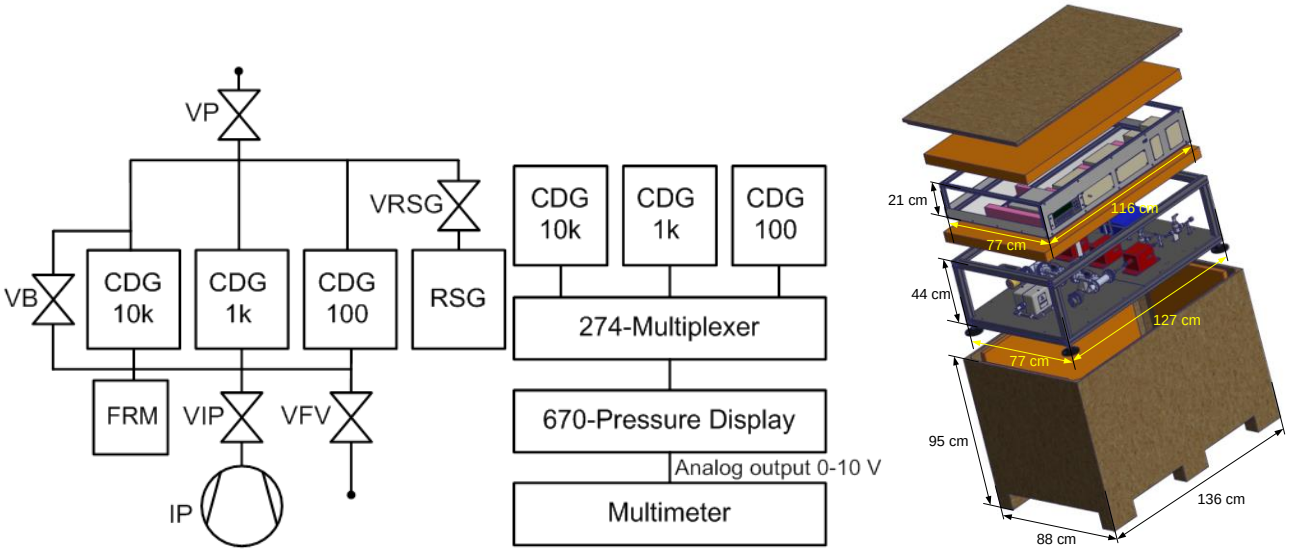

Figure 4.1.3.1: Set-up (pneumatic scheme on the left and electrical connections in the middle) and general view of the transfer standard with its transportation box (on the right).

The development and the initial characterization are described in [5].

A comparison using the TS was carried out with a participation of FCT-UNL, CEM, IMT, TUBITAK and LNE. LNE checked both the stability of the RSG and the stability in shape of the CDGs' calibration curves.

At FCT-UNL, a 1 Torr and a 1000 Torr MKS Baratron CDGs were used as reference standards. CEM calibrated the transfer standard by a direct comparison with its force-balanced piston gauge FPG (Fluke). IMT used its new static expansion facility. TUBITAK used its FPG for the measurements. Also LNE used its force-balanced piston gauge (Fluke FPG 8601) to perform the calibration of the transfer standard.

The basement of the transfer standard is the RSG. From its final calibration, the uncertainty due to its stability was estimated to be 2.6×10^{-5} in relative value (k = 1), which is higher than was first estimated [5]. The key rescaling coefficient is k_{10k} determined from four pressure points performed at 5, 7, 9 and 10 kPa with the RSG and CDG 10k. In the protocol, it was also initially required to perform four pressure points to determine the other rescaling coefficients k_{1k} and k_{100} with CDG10k and CDG1k then with CDG1k and CDG100, respectively. The analysis of the rescaling coefficients determined from the measurements of the participants is shown in Fig. 4.1.3.2.

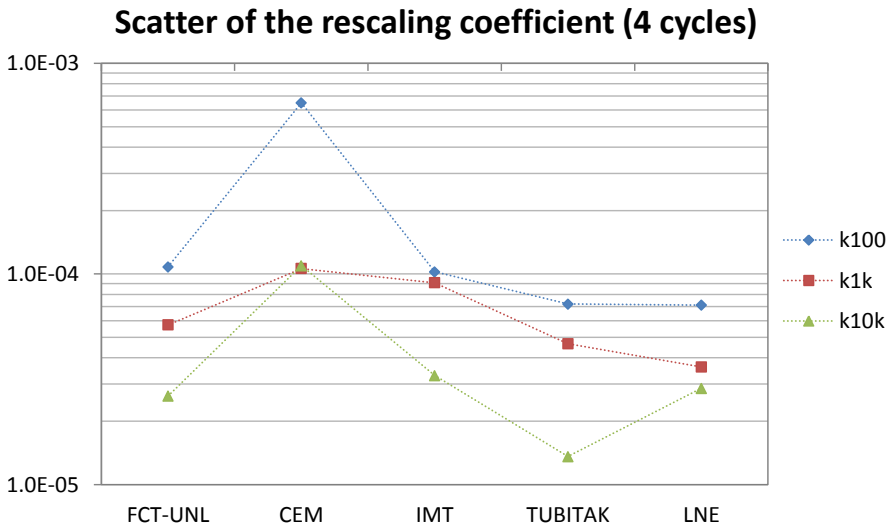

Figure 4.1.3.2: Scatter of the rescaling coefficients of CDGs over the four calibration cycles.

The measurements made by the participants during the comparison highlighted that the CDGs kept a repeatability and a drift conform to their initial performance. Thus, the transfer standard did not suffer from the successive transportations from one European laboratory to another.

The uncertainty of the transfer standard is estimated from the range 1 to 10 kPa moving down to the range 0.1 to 1 kPa and finally 1 to 100 Pa. CDG10k is rescaled with the RSG. Let us denote $u_{resc10k}$, the rescaling uncertainty for CDG10k. It is calculated as the combination of the uncertainty in stability of the RSG (2.6×10^{-5}) and the mean stated repeatability of the set of CDGs u_{repCDG}, which is about 3.0×10^{-5} in relative value (Figure 13). CDG10k is the base to rescale CDG1k. The rescaling uncertainty u_{resc1k} for CDG1k is the combination of $u_{resc10k}$ and of u_{repCDG}. The same way, the rescaling uncertainty $u_{resc100}$ for CDG100 is the combination of u_{resc1k} and of u_{repCDG}. For each range, the uncertainty due to the shape drift of the considered CDG is taken into account as a non-applied correction.

Each participant has calibrated the transfer standard for its own laboratory temperature T_{lab} which has an influence on the signal of a thermostated CDG, due to the thermal tranpiration effect. Then the reference pressure is corrected to the nominal ambient temperature T_0 of 20°C using an empiric thermal transpiration correction. The standard uncertainty for the ambient temperature T_{LAB} is estimated to be 0.5 K. The calculation of $p_{LAB}(T_0)$ with the thermal transpiration function f_{TT} of Takaishi Sensui leads to a standard uncertainty contribution in pressure of 0.0020 Pa, which can be neglected for pressures above 100 Pa. The temperature coefficient of the RSG was studied in the range 15 °C to 25 °C [5]. It is less than 1 ppm·K^{-1} and can consequently be neglected. Table 4.1.3.3. sums up the uncertainty budget of the transfer standard in each range corresponding to each CDG, represented graphically in Figure 4.1.3.4.

Table 4.1.3.3: Uncertainty budget of the transfer standard.

Uncertainty component	**CDG10k (1 - 10) kPa**		**CDG1k (0.1 – 1) kPa**		**CDG100 (1 – 100) Pa**	
	× p	Pa	× p	Pa	× p	Pa
RSG stability (CDG10k)	2.6×10^{-5}	-	-	-	-	-
CDG repeatability (for rescaling operation)	3.0×10^{-5}	-	3.0×10^{-5}	-	-	-
Ambient temperature	-	-	-	-	-	0.0020
Rescaling uncertainty u_{resc}	4.0×10^{-5}	-	5.0×10^{-5}	-	5.8×10^{-5}	-
Shape drift	2.2×10^{-5}	0.24	2.8×10^{-5}	0	7.6×10^{-6}	0.0018
TS standard uncertainty	**5.1×10^{-5}**	**0.12**	**6.4×10^{-5}**	**0**	**4.6×10^{-5}**	**0.0020**

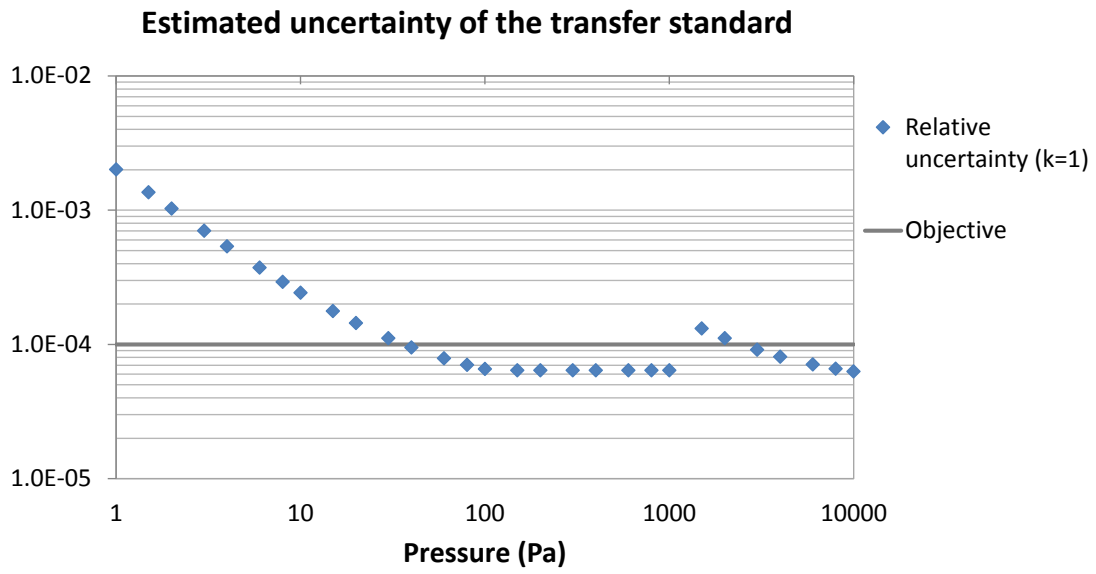

Figure 4.1.3.4: Uncertainty contribution of the transfer standard.

Conclusion

The contribution in uncertainty of the transfer standard for absolute pressures *p* between 1 Pa and 10 kPa developed in the frame of the project is compatible with the target of $1\times10^{-4}\times p$, except for pressures lower than 30 Pa. For the latter, the resolution of the standard and the contribution of the thermal transpiration effect are getting preponderant. This relatively low contribution, compared with the usual stability performances of capacitance diaphragm gauges, is provided by the stability of the slope correction of a commercially available resonant silicon gauge, with which the CDG of 10 kPa full range is rescaled. As the slope correction of the RSG is stable, but an offset drift can occur, several rescaling pressure points are required.
This transfer standard is suitable to demonstrate metrological capabilities of calibration services which are rarely lower than a few $10^{-4}\times p$. It could also be suitable for comparisons between National metrology institutes that need to validate uncertainties as low as a few times $10^{-5}\times p$, knowing that the transfer standard can be improved. In fact, in the protocol used here, the rescaling procedure for CDG1k and CDG100 was not optimum while the protocol to determine the zero of the CDGs was unsuitable for FPG owners. Moreover, the drift of the transfer standard was not estimated in the best way.
This work has also shown that instruments composing the transfer standard with the associated rescaling procedure can be used as a working standard in the range 5-130 kPa [4]. By extension, the standard developed within the project is suitable to be either a working or a transfer standard in the absolute pressure range from 1 Pa to 130 kPa.

4.1.4 Absolute pressure transfer standards for traceability of industrial vacuum gauges below 10 Pa

The aim of this study was to create high-performance transfer standards of absolute pressure to provide traceability of industrial vacuum gauges below 10 Pa. Manufacturers of vacuum measuring instruments need such transfer standards to obtain traceability to primary vacuum standards with the lowest possible uncertainty in the pressure range from 10^{-4} Pa to 10^{5} Pa. However, new low scale (≈1 Pa) Capacitance Diaphragm Gauges (CDGs) have recently been developed which have the potential to be used as transfer standards and to extend the traceable pressure range down to 10^{-2} Pa. With these new CDGs, the uncertainty of traceable low pressure measurements could be reduced to a level comparable with that of spinning rotor gauges (SRGs) in order to create the capability for vacuum measurements in industrial dynamic conditions. Below 10^{-1} Pa, the existing response time of SRGs is usually longer than 10 s, but the new CDGs should enable shorter response times by several orders of magnitude. To characterise and calibrate such new-generation CDGs coming off the manufacturing line, manufacturers need the transfer standards having the same or even lower uncertainty than the CDGs to be calibrated, i.e. ≤0.5 % of reading in the first measurement decade.
RISE evaluated a set of three Stripe CDG045DHS Capacitance Diaphragm Gauges (CDGs) supplied by INFICON LI in the pressure range from 10^{-4} Pa to 1.3 Pa as a potential transfer standard for the pressure range below 1 Pa. RISE has performed experiments to assess these new low scale CDGs (1.3 Pa full scale) with

respect to their output signal stability, repeatability and the influence of ambient temperature on their indicated pressure. During experiments, all pressure levels were realized by RISE ultra-high vacuum system SEA5 (Series Expansion Apparatus 5). A spinning rotor gauge, SRG2 from MKS, was used as reference pressure gauge. An environmental test chamber, Thermotron S1.2, was used to control ambient temperature in the range from +15 °C to +30 °C (Fig. 4.1.4.1).

Figure 4.1.4.1: Three INFICON LI CDGs inside a Thermotron 2800 for temperature control.

Fig. 4.1.4.2 shows a graph of the influence of ambient temperature on the CDG outputs expressed as a temperature coefficient of the CDG reading versus pressure. All gauges show equal results. The graph also shows the modelled contribution from real changes in local pressure at gauge due to the thermal transpiration effect. Thermal transpiration has substantial influence on the temperature dependency in the high-pressure regime. In the low-pressure regime, thermal transpiration is expected to have very little influence on the temperature dependency. In this regime, the gauges still show a dependency of $-1 \cdot 10^{-4}$ Pa/K.

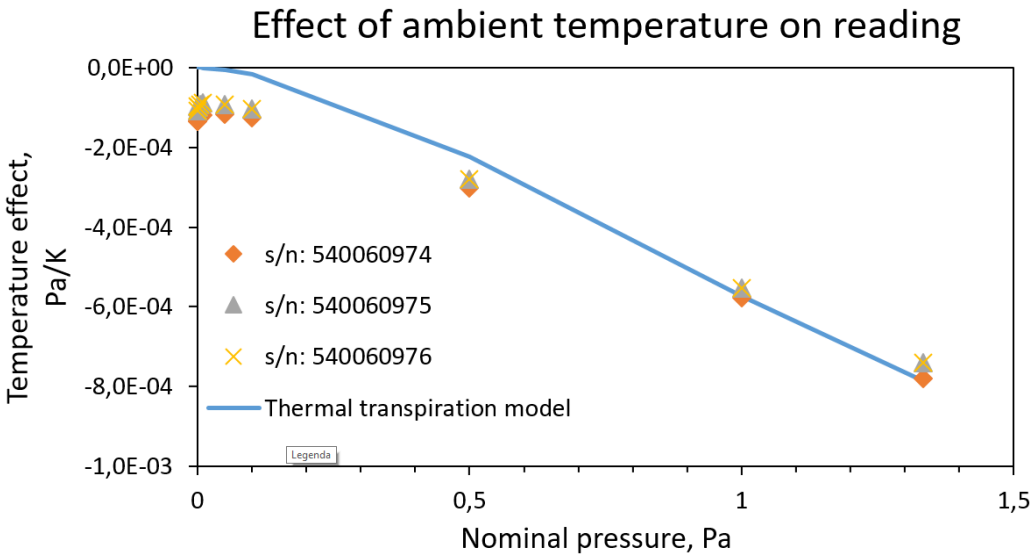

Figure 4.1.4.2: Temperature dependency of CDG reading as a function of nominal pressure. Solid line corresponds to the modelled contribution from real changes in local pressure at gauge due to the thermal transpiration effect.

Looking at the specifications of the manufacturer for the tested CDGs, there is a temperature effect on zero reading up to 0.01% FS /°C, which is equal to $1.3 \cdot 10^{-4}$ Pa/K. So, the obtained results are within manufacturer specification. Manufacturer also specifies a temperature effect on the span of 0.01% of reading /°C, which is equal to $1 \cdot 10^{-4}$ Pa/K at pressure 1 Pa. RISE results at 1 Pa indicate that manufacturer specification regarding temperature effect on the span is also met.

Output from the gauges show a dependency upon ambient temperature. At pressures above 0.5 Pa the dependency is negligible compared to the real effect from thermal transpiration. At pressures below 0.1 Pa the

thermal transpiration approaches zero and the dependency upon ambient temperature is clearly caused by variations of the zero reading, which is not completely compensated by temperature stabilization of the measuring cell at 45°C. The magnitude of the dependency is on such a scale that the ambient temperature fluctuations during measurements should be below 0.5 °C. General reproducibility of the gauges under realistic laboratory conditions are constant over the entire pressure range. Ambient temperature fluctuations may contribute to broadening of the distribution of readings.
Reference stability may also influence the observed repeatability of the CDGs. At pressures below 10 mPa the reference is about 100 times more stable than the CDGs. Any influence at those levels is deemed negligible. At higher pressures the reference pressure indication has reduced repeatability. This reduced repeatability most likely originates from the pressure regulator and not the reference gauge. As the repeatability of the CDGs is measured with respect to the deviation from the reference gauge, any fluctuation in realized pressure level should be cancelled. This is clearly the case since at higher pressures the standard deviation of the reference pressure is larger than the standard deviation of the difference between reference and CDGs.
The performance of the gauges is limited by low-frequency noise that results in a repeatability $2 \cdot 10^{-4}$ Pa in the same laboratory over 4 weeks. The gauges also show a dependency upon ambient temperature, which produces an uncertainty comparable with the repeatability if the ambient temperature varies by more than 1 °C (cca $-1 \cdot 10^{-4}$ Pa/K at pressures below 0.1 Pa).

CMI and IMT investigated another set of three pieces of 10 mTorr INFICON LI CDG 045Dhs PN 3CC9-S53-2380 with serial numbers 540060971, 540060972 and 540060973 denominated as A, B, C, respectively. CMI used their spinning rotor gauge SRG2CE serial number 94157G60SRG2 from MKS manufacturer for the long-term stability study.
CMI utilized its new primary transition regime dynamic expansion system (Fig. 4.1.4.3) in the range 10 Pa down to 10^{-2} Pa and the classical dynamic expansion system down to 10^{-4} Pa. Also, a primary digital piston manometer FPG was used for the measurements above 0.8 Pa. The expanded uncertainty of the FPG system is 0.02 Pa + $2.8 \cdot 10^{-5}$ p. For transition expansion system, the uncertainty is $3 \cdot 10^{-6}$ Pa + $0.015 \cdot p$ and for dynamic expansion system, the uncertainty is equal $5 \cdot 10^{-8}$ Pa + $0.013 \cdot p$. The ambient temperature was stabilized in the range (20.0 ± 0.5) °C.

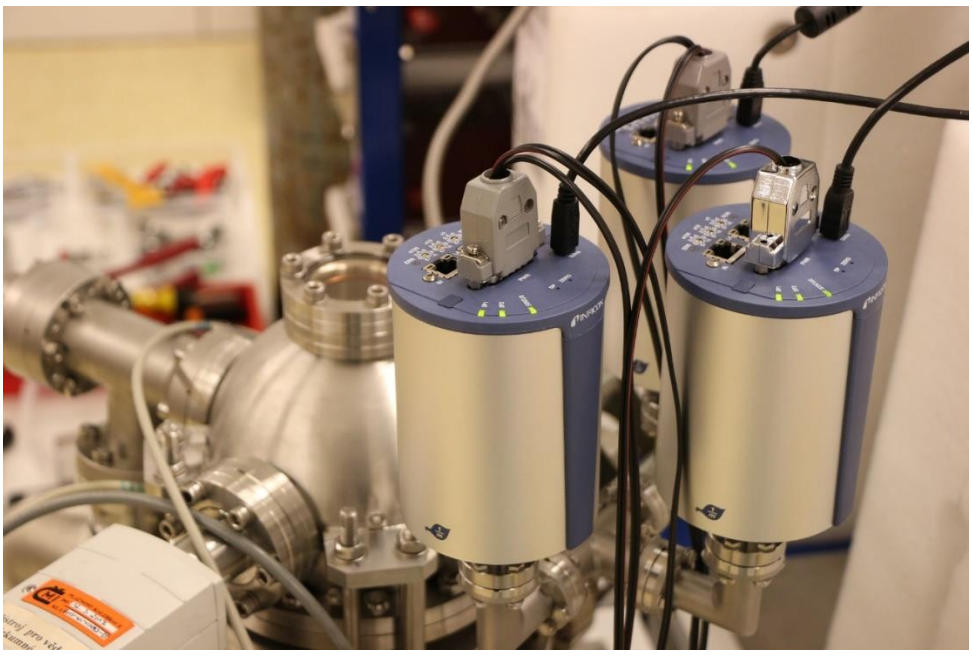

Figure 4.1.4.3: Set of CDG045Dhs 0.01 Torr gauges connected to transition expansion system.

IMT utilized its new static expansion system for the gauges' calibration with the uncertainty $2.4 \cdot 10^{-6}$ Pa + $0.8 \cdot 10^{-3}$ p. A digital communication interface was used to get the output from the INFICON LI CDG gauges. Mini USB Type B Connector (Diagnostic Port) was connected via a standard USB cable to the USB port of a personal computer. The driver created a virtual COM port in this computer. TGaugeExpress software was used for monitoring and logging the values. The output signals were logged every 0.5 s. Before each measurement, the gauges were zeroed, typically at pressures below $2 \cdot 10^{-5}$ Pa (manufacturer recommendation: $p < 6.6 \cdot 10^{-5}$).

The digital output signal was used to analyse the gauges' performance. The gauges were left in the system with pressure kept below $1 \cdot 10^{-5}$ Pa. The gauges A and C showed signal with the typical standard deviation $7 \cdot 10^{-5}$ Pa. The gauge B showed an additional noise of amplitude $1 \cdot 10^{-3}$ Pa. The additional noise disappears for a long time but occasionally reappears at different pressures with a similar or a lower amplitude. From the point of the mid-term stability, the gauges show a drift of the output signal up with the slop e up to 10^{-4} Pa/h. For the long-term stability study, the pressures from (0.1 to 1.3) Pa were investigated.

The zero stability of the gauges was checked. Before each measurement, the gauges were zeroed (or zero reading was averaged and subtracted from next measurements). So, the difference of the gauge indication is now the span slope and/or deviation from the linearity. The gauges are independent of each other. Therefore, if there is an instability in one gauge, the difference of its indication and indication of other gauges will be noticeable. Thus, there is a possibility to compare differences between indication of individual gauge and the reference value, while the reference value may be another gauge indication or the average value of the indication of all gauges. The difference of the indication may be then plotted as a function of pressure for each measurement, see Fig. 4.1.4.4.

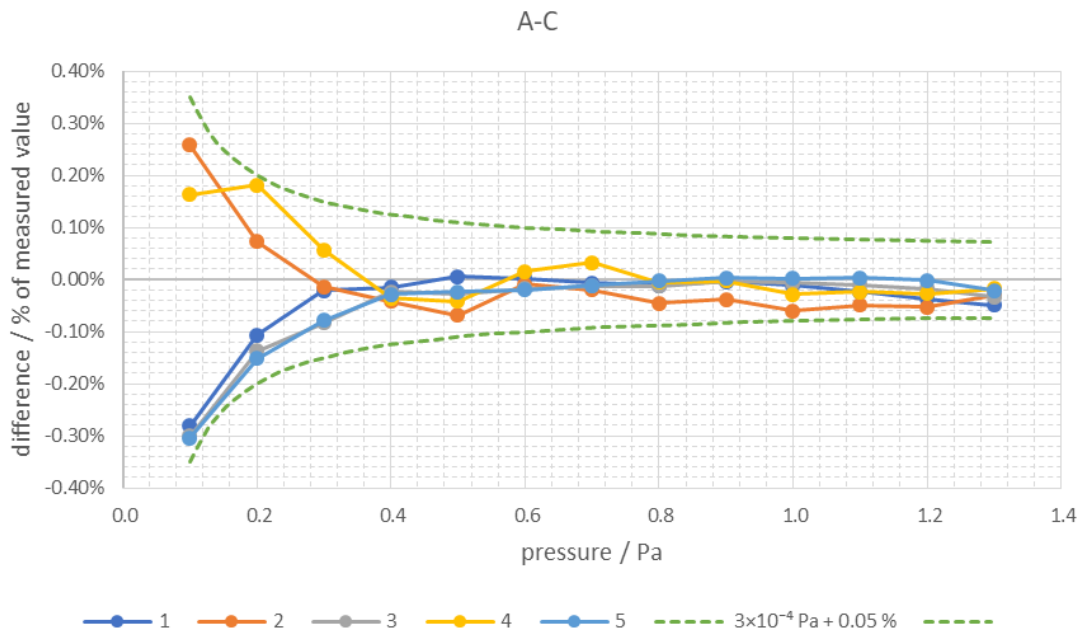

Figure 4.1.4.4: Difference of indications.

During the series of measurements performed at CMI and IMT, a real resolution of 3×10^{-4} Pa was obtained (caused by digital output standard deviation and short term stability). It is similar to the results of RISE. It agrees with the resolution 0.003 % FS stated by the manufacturer. As for the long-term stability, it consists of absolute value 3×10^{-4} Pa and the relative contribution $5 \cdot 10^{-4} \times p$. This value is very acceptable and surpassing other currently available CDGs by nearly one order. As for the question of the linearity (calibration curve), a correction to the calibration curve must be applied to reach the demanded accuracy of 0.5 %.

An important contribution to the uncertainty of pressure measurements and calibrations with CDGs below 10 Pa is the thermal transpiration effect which is gas dependent. Currently, there is a limited amount of data available for the thermal transpiration effect in CDGs, and this is further limited to CDGs heated at 45 ºC. However, numerous industries involve heating CDGs to much higher temperatures.

For the study, INFICON LI provided a set of seven CDGs. IMT performed a study of the thermal transpiration effect of different gases in CDGs which were heated at 45 °C to 200 °C in a pressure range from 10^{-1} Pa to 130 Pa. Following gases were used in the study: H_2, He, CH_4, Ne, N_2, CO, O_2, Ar, CO_2, Kr, water vapour and a hydrocarbon dodecane $C_{12}H_{26}$ (M = 170.33). For the study, IMT assembled a measurement/calibration system based on comparison method. The reference gauge was a CDG MKS model 690A 01TRA, which was used with the heater switched off during the measurements. This enabled operation near ambient temperature, so the difference of response of the reference gauge to different gases due to thermal transpiration effect was negligible. The measurement system for thermal transpiration study was attached to the IMT static expansion primary calibration system, which enabled in-situ calibration of the reference CDG by static expansion method

with nitrogen gas. Effect of different gauge temperature on the thermal transpiration ratio TTR= (P_{gauge}/P_{ref}) for nitrogen can be seen from Fig. 4.1.4.5 (P_{gauge} is displayed pressure of CDG and P_{ref} is reference pressure).

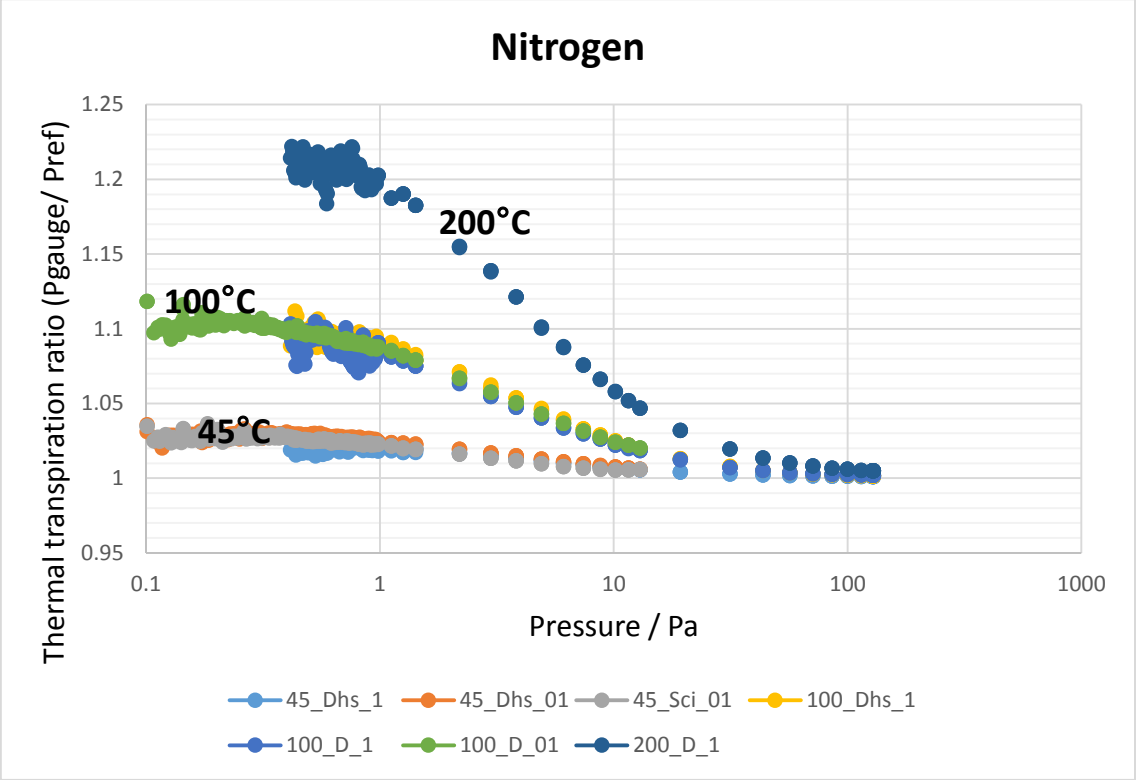

Figure 4.1.4.5: Measured thermal transpiration ratio TTR = (P_{gauge}/P_{ref}) for nitrogen (gas temperature in calibration chamber was 25 °C).

It is clearly seen from Fig. 4.1.4.5 that below 1% of the gauge full scale range the scatter of measurement signal increased. This means that for calibration work where required uncertainty is below 0.5% the usable range of tested CDGs is from 2% to 100% of the gauge measurement span.

Below 1 % of the CDG full scale range, the scatter of measurement signal increased. For calibration work where required uncertainty is below 0.5 %, the usable range of tested CDGs is two decades, from 2 % to 100 % of the gauge measurement span. The thermal transpiration correction for gases other than N_2 can be calculated from thermal transpiration curve obtained from calibration in N_2, and after normalization of pressure scale: $P^* = p/(\langle c \rangle \times \eta)$, where p is measured pressure, $\langle c \rangle$ is mean velocity of gas molecules and η is gas viscosity. Uncertainty of thermal transpiration correction for different gases, calculated from normalized TTR curve, for CDGs operated at 45 °C is better than 0.5 % in the pressure range from 1 Pa to 130 Pa and above. Below 0.1 Pa, uncertainty is increased to approximately 0.8 %. For CDGs operated at 200 °C, the uncertainty of thermal transpiration correction from normalized TTR curve better than 1 % can be achieved in the pressure range from 2 Pa to 130 Pa and above. Below 2 Pa the uncertainty increases to several %.

Conclusion

The results show the advance of the CDG technology. Although the investigated 10 mTorr gauges from INFICON LI, model STRIPE CDG45Dhs, which were tested by RISE and CMI, are not the highest accuracy class (metrology) CDGs, the resolution 3×10^{-4} Pa obtained with the digital output represents a similar characteristic as the common CDGs widely used for traceability in the metrological laboratories. Also, the long-term stability of the CDG sensitivity coefficient at level of $5\times10^{-4}\times p$ was proved achievable. This is a very good result, reached for the laboratory conditions and the same gas. For different gases and wider ranges of the temperature, corrections have to be made and an additional uncertainty should be considered.

The idea of using 1 Pa FS range CDGs as a reference instrument for calibrations in the range from 1×10^{-4} Pa was proved to be challenging. When the correction factor due to thermal transpiration is determined by the calibration of the individual gauge and, in addition, appropriate corrections are made due to the difference of ambient (gas) temperature at the time of gauge calibration and at the time of gauge use, the uncertainty equal to 5×10^{-4} Pa is achievable. This means that, for reaching the demanded accuracy of 0.5 %, the measured

pressure has to be 5×10^{-4} / 0.5 % = 1×10^{-1} Pa or higher. For lower absolute pressures, the stability and reproducibility of existing spinning rotor gauges remain still better than of the studied CDGs.

However, the study shows that, in some special cases in metrology laboratory praxis, for pressures below 1×10^{-1} Pa, CDGs can perform better than SRGs when demanding a quick response or when measuring gas mixtures of roughly known components ratio (mixture composition strongly affects SRGs, but for CDGs, it only affects the thermal transpiration correction). The ambient temperature study and thermal transpiration coefficient examination provide the information for the limits of using CDGs in this special cases.

If we would build a package with INFICON LI gauges which were investigated (Stripe CDG45Dhs), one having full scale range 1.3 Pa and another 13 Pa, we can get an efficient transfer standard for the range from 0.1 Pa to 13 Pa which would be more accurate than any existing transfer standard in this range. Such transfer standard package would be relatively cheap and easy to transport from NMI or other calibration laboratory to the users in industry, compared to a much more sophisticated transfer standard package described in [5].

Based on the results of the present investigation, we can set up uncertainty budget of the proposed transfer standard package, which is given in Tab. 4.1.4.6. It contains only contributions from the transfer standard itself, without uncertainty of calibration. The reason for not including calibration uncertainty in the budget is because the uncertainties of calibration of such transfer standard package can vary considerably depending on the uncertainty of a higher level or primary standard. Therefore, we can name the uncertainty budget given in Tab. 4.1.4.6 as stability uncertainty.

Table 4.1.4.6: Budget of stability uncertainty for a transfer standard package composed from INFICON LI gauges Stripe CDG45Dhs 1.3 Pa FS and 13 Pa FS. For comparison, the estimated uncertainty of transfer standard package developed by LNE (Section 4.1.3) in the range from 0.1 Pa to 13 Pa is also given.

Uncertainty component	Stripe CDG45Dhs (1.3 Pa FS + 13 Pa FS)	LNE transfer standard package
Long term stability	$2.5\times10^{-4}\times p$	
Thermal transpiration correction (assuming 0.2°C uncertainty of gas temperature)	$3.4\times10^{-4}\times p$	
Resolution (noise and zero stability)	2.5×10^{-4} Pa	
Combined uncertainty	$4.3\times10^{-4}\times p + 2.5\times10^{-4}$ Pa	
Expanded uncertainty (k=2) *Note: excluding uncertainty of calibration	$8.6\times10^{-4}\times p + 5\times10^{-4}$ Pa	$1.9\times10^{-4}\times p + 5.9\times10^{-3}$ Pa (See *Note)

Fig. 4.1.4.7 shows comparison of stability uncertainties of the transfer standard composed of INFICON LI Stripe CDG45Dhs gauges and transfer standard package developed by LNE in the range from 0.1 Pa to 13 Pa. Proposed transfer standard package composed of INFICON LI Stripe CDG45Dhs gauges has significantly reduced stability uncertainty in the range from 0.1 Pa up to few Pa.

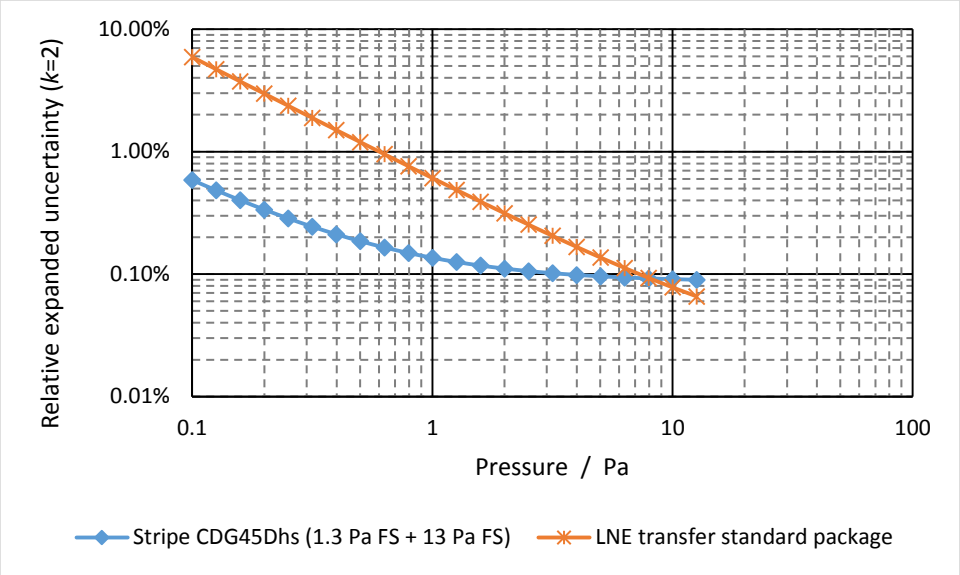

Figure 4.1.4.7: Stability uncertainties of the transfer standard composed of INFICON LI Stripe CDG45Dhs gauges and transfer standard package developed by LNE.

For precise traceability, the absolute pressure range below 1×10^{-1} Pa remains dominated by SRGs. For last years, intensive development of optical methods has been performed [6-9]. Herewith, the optical technology may have an ability to break through under the 1×10^{-1} Pa limit with sufficiently low uncertainties and introduce new measuring techniques for industrial applications in this range of absolute pressure.

4.2 Development of calibration methods for positive and negative gauge pressure standards in the range from approximately -10^5 Pa to 10^4 Pa

Over the past decade, the number of calibrations in the intermediate pressure-to-vacuum range has been increasing whilst at the same time their uncertainties have been reduced. As a result, NMIs and secondary laboratories have had to develop new methods for meeting this increasing demand. The last comparisons in the intermediate pressure-to-vacuum range highlighted the challenge of comparing and thereby calibrating instruments. Moreover, there are few CMCs in this widely-used pressure range, especially the negative pressure range. The aim of this work was to develop and validate calibration and measurement techniques and instruments for the calibration of pressure measuring devices for gauge pressure, including negative gauge pressure, in the range -100 kPa to +15 kPa. Different techniques were to be considered for the different modes of operation of the instruments: absolute, gauge and negative gauge pressure.

4.2.1 Development of methods for accurate, weather-independent calibration of low gauge pressure instruments

The objective of this research was to provide the calibration methods for accurate, weather-independent calibrations of low gauge pressure instruments with an uncertainty in industrial conditions better than 3 Pa + $2\cdot10^{-4}\cdot p$ and to create the general recommendations for the best laboratory practice. To circumvent the constraint of the atmospheric pressure fluctuations, it is often advantageous to replace the real atmospheric pressure by a pressure close to it which is generated and stabilised in an auxiliary volume. Two ways to ensure that were studied and are presented in this report. The first one is utilisation of an additional volume connected to the reference ports of the LS and the DUT. The second method is based on the use of a hermetic chamber large enough to enclose both the LS and the DUT. By adding a volume, whose temperature is regulated, one can control a nearly atmospheric pressure in this volume with the help of a pressure or flow regulator.

Additional volume method

The principle of this method is usage of an extra volume connected to the reference ports of the LS and the DUT. The studies of pressure stability have been carried out using volumes with the capacities of 1.2 litres and 2.7 litres. Results have shown that variations of pressure inside these volumes are below 1 hPa when the laboratory temperature is regulated to within ±1 °C. Fig. 4.2.1.1 shows one of possible calibration set-ups using an external reference stabilisation volume (RSV).

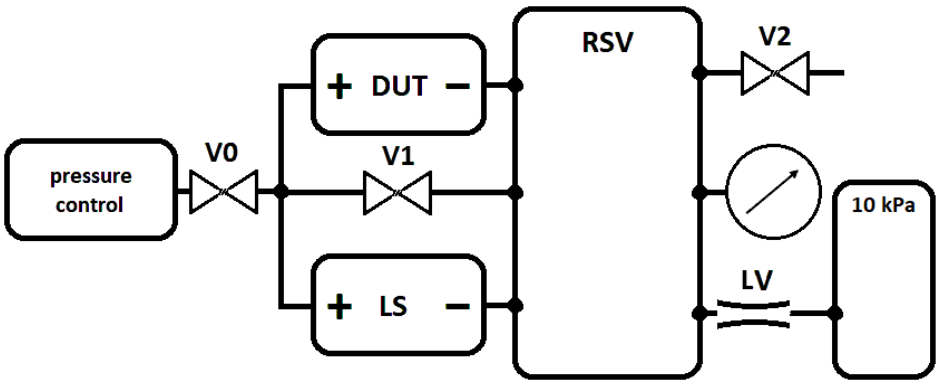

Figure 4.2.1.1: Course vacuum damped reference stabilisation volume set-up.

Here, the leak leads to a stable course vacuum reference of value approximately 10 kPa by an absolute pressure controller. Fine leak valve LV is adjusted to maintain the reference pressure in the volume RSV. A barometer must be connected to the RSV in this case to ensure that this reference pressure is stable and close to the atmospheric pressure. The zeroing is performed in the same way as in the previous case. Then V2 is closed and it can be checked if the pressure is stable. Before starting a calibration, the valve V1 is closed, V0 opened and the leak valve adjusted to set and to maintain the reference pressure near to the desired atmospheric value.

At PTB, tests with an additional reference volume were carried out to test performance of low gauge pressure calibrations at pressures of 100 Pa, 5 kPa and 10 kPa. An FRS5 digital piston gauge with a measurement range of 11 kPa was used as a reference standard (LS), and a Baratron differential pressure gauge by MKS Instruments with a measurement range of 13 kPa was used as a device under test (DUT). The measurement and reference pressure connections of the LS and the DUT are shown in Fig. 4.2.1.2.

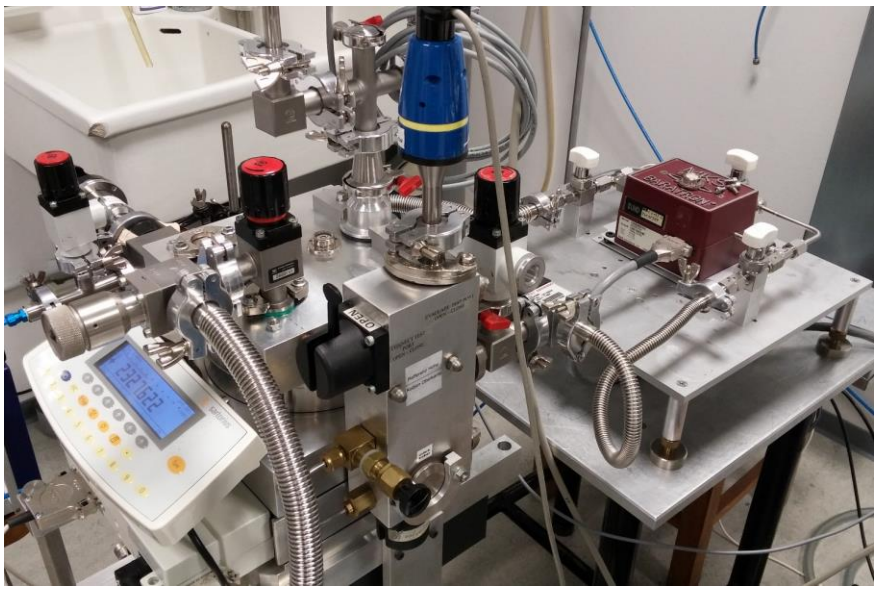

Figure 4.2.1.2: Connections of FRS5 as LS with MKS Baratron as DUT.

Measurements were performed without and with additional volumes of 1.2 l and 2.7 l. One example of measurements without any stabilisation and with an additional volume of 1.2 l is shown in Fig. 4.2.1.3.

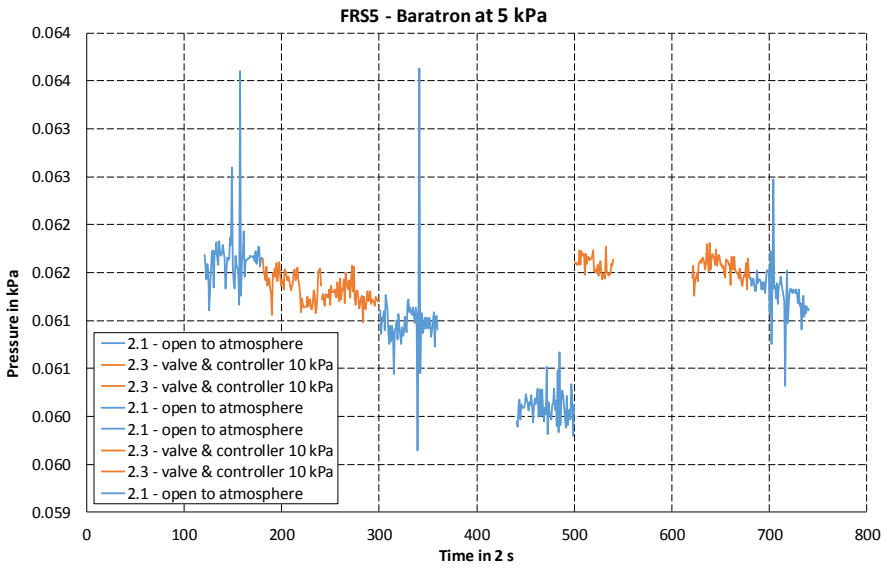

Figure 4.2.1.3: Difference of FRS5 and Baratron readings at about 5 kPa taken without and with an additional volume of 1.2 l.

Without reference pressure stabilisation, the max-min difference and the standard deviation are equal to 4.0 Pa and 0.29 Pa, respectively. With the pressure stabilisation, the max-min difference and the standard deviation are equal to 2.8 Pa and 0.19 Pa, respectively, which is an essential improvement.

Hermetic chamber method

The principle of this method is usage of a hermetic chamber capable to enclose both the LS and the DUT. This method can be used when one of the devices has not got a reference port and, therefore, cannot be connected to a reference volume. Fig. 4.2.1.4 shows one of possible calibration set-ups for using a hermetic chamber serving as RSV.

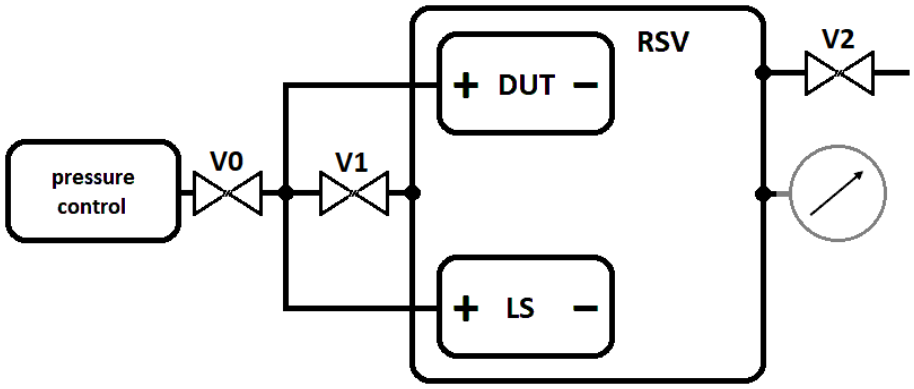

Figure 4.2.1.4: Hermetic chamber set-up.

The RSV can be completely isolated from the external atmosphere by valve V2 in the first set-up or it can always involve a connection between the RSV and the external atmosphere by a small leak damping the atmospheric fluctuations in the second set-up. A measurement of the pressure inside the RSV by a barometer is non-mandatory, however stability of this pressure must be ensured. Both the LS and the DUT must enable an electronic communication, and relevant electrical feedthroughs in the RSV must be ensured, or a window in the RSV to read the indications of the LS and DUT must be present. One of the following devices can serve as a laboratory standard: a) classical pressure balance with a remotely operated mass handling system, b) digital pressure balance, c) digital manometer.

CMI built a chamber capable to house both a low gauge pressure laboratory standard and a device under test at atmospheric pressure while being hermetically isolated from the usual atmospheric pressure fluctuations, Fig. 4.2.1.5.

Figure 4.2.1.5: Hermetic chamber capable to contain both an FPG and a DUT and allowing pressure control.

As an FPG8601 by DHI/Fluke serves as laboratory standard of the CMI (and many other laboratories) for this range, it was decided to adjust its internal dimensions to this instrument, plus include some extra space for a device under test (DUT). An example of pressure measurements at 5 Pa without and with the hermetic chamber's pressure stabilisation is shown in Fig. 4.2.1.6 a) and b), respectively.

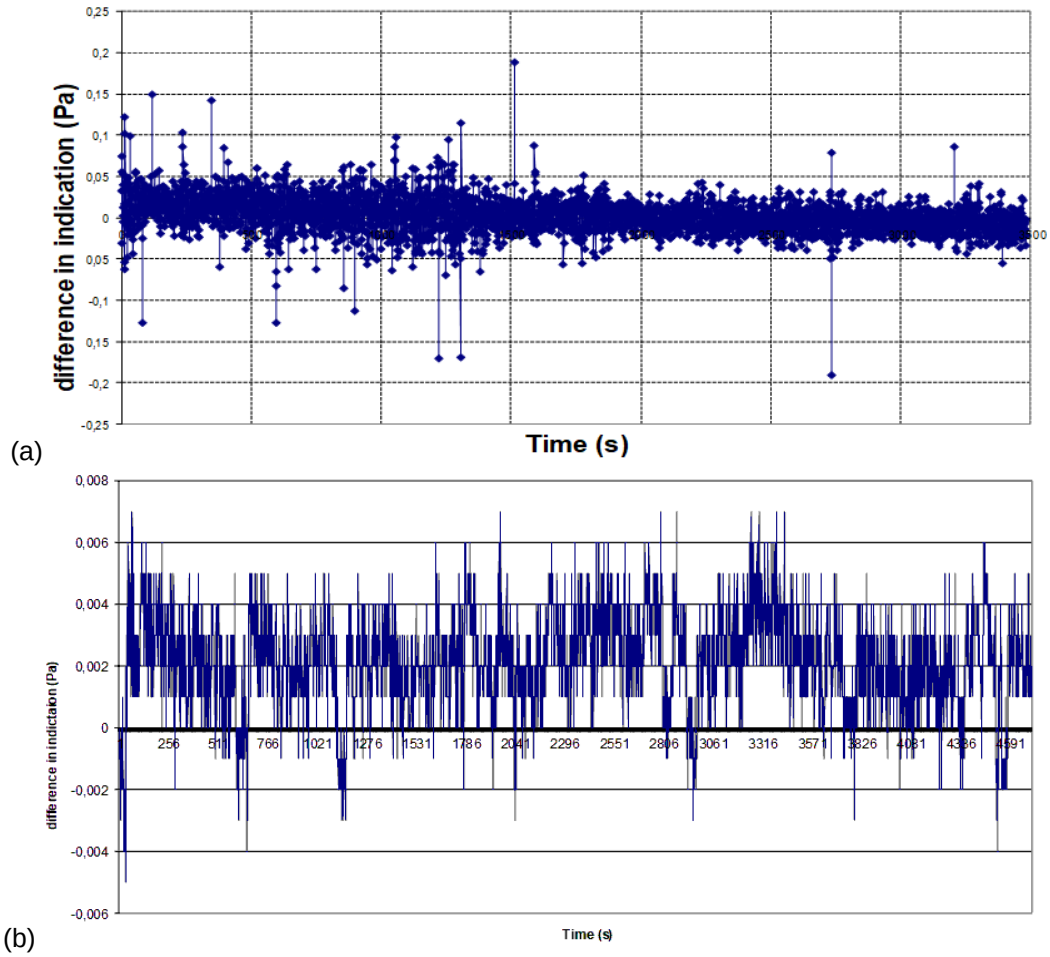

Figure 4.2.1.6: Pressure stability without (a) and with (b) using hermetic chamber.

It shows an improvement of the calibration results by about factor 20.

A summary of the measurements with additional volumes at PTB and a hermetic chamber at CMI is presented in Table 4.2.1.7.

Table 4.2.1.7: Standard uncertainties of calibrations.

Laboratory	P / Pa	$u(P)$ / Pa (k=1)	StD / Pa (without control of the ambient pressure)	StD / Pa (with control of the ambient pressure)
CMI	5	0.020	0.15	0.002
PTB	100	0.047	0.14	0.06
	5000	0.10		0.19
	10000	0.16	0.16	0.13

Conclusion

As the experiments have demonstrated, the new methods allow one to reach the calibration uncertainties below 3 Pa + $2 \cdot 10^{-4} \cdot p$ even at unstable weather conditions. It has also been shown that there was a possibility to carry out calibrations, despite of the severe ambient conditions, by circumventing the constraints related to the instability of the ambient atmospheric pressure by means of either a closed reference volume or a hermetic chamber. The methods are applicable to calibrate mechanical and electronic low gauge pressure measuring instruments.

4.2.2 Development of methods for negative gauge pressure calibration based on absolute and gauge pressure balances, FPGs and liquid column manometers

The aim of this work was to develop calibration methods for positive and negative gauge pressure standards in the range from approximately -10^5 Pa to 10^4 Pa in order to reduce the uncertainty of pressure calibration down to $3 \times 10^{-5}\ p$ + 1 Pa independent of variable ambient conditions, and in industrial conditions to better than $2 \times 10^{-4}\ p$ + 3 Pa. Five methods for positive and negative gauge pressure calibration were studied, three based on dead-weight balances, a fourth using a mercury column manometer and a fifth with a force balanced piston gauge FPG.

Pressure balance based methods: Calibration by using two absolute pressure instruments

The principle of this method is to perform a gauge pressure calibration by using a pressure balance in absolute mode together with a barometer [2]. The standard pressure is calculated by the difference between the pressure balance P_{abs} and the indication of the barometer which is corrected from accuracy error E_0:

$$P_{\mathrm{neg}} = P_{\mathrm{abs}} - P_{\mathrm{atm}} \tag{4.2.1}$$

where $P_{\mathrm{atm}} = P_{\mathrm{baro}} + E_0$. Atmospheric pressure P_{atm} is given by the indication of the barometer P_{baro} corrected for the accuracy error E_0. Figure 4.2.2.1 shows the set-up necessary to calibrate a manometer in negative pressure mode.

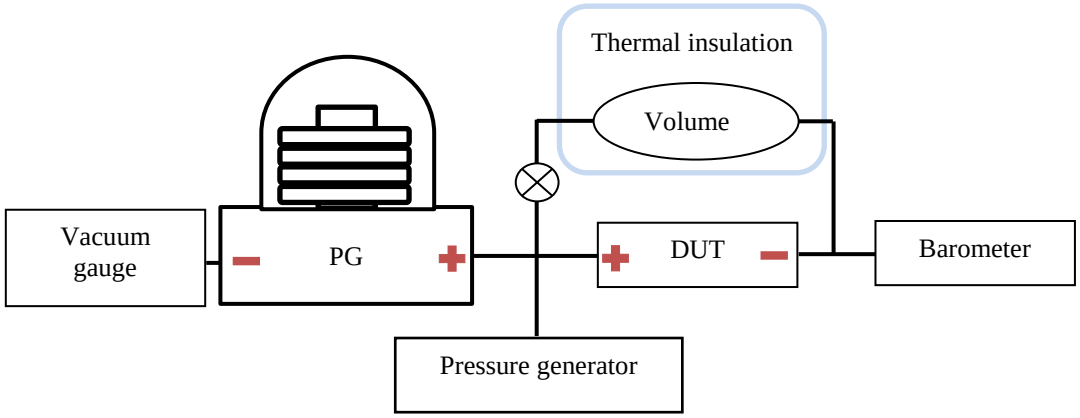

Figure 4.2.2.1: Set-up for negative gauge pressure calibration using two absolute pressure instruments.

The <<Test +>> ports and pressure controller are connected together, while a vacuum manometer is connected to the << Test - >> port of the pressure balance. To read the ambient pressure, a barometer is connected to the reference port of the manometer to be calibrated. This barometer must to be at the same level as the manometer to avoid the need to apply a supplementary head level correction. By adding a volume, which can be regulated in temperature, one can control the atmospheric pressure. This is particularly advantageous in the case of low atmospheric pressure (below 950 hPa) encountered in laboratories at altitude.

When using a model PG 7601 that has a piston of 10 cm² area, one has to apply a pressure of 1050 hPa to perform the measurement point at -950 hPa because the minimum absolute pressure point is 100 hPa. This volume has to be connected directly to barometer and reference port of the manometer and to the pressure controller using a valve.

Pressure balance based methods: Calibration by applying negative pressure to the bell jar of the balance

This method is based on the use of a pressure balance in gauge mode. Unlike the usual mode of operation, here the manometer under calibration is connected to the << Test ->> port while the negative pressure is applied to the bell jar of the balance. As shown in Figure 4.2.2.2, the <<Test +>> port of the balance and the << Reference>> port of the manometer are connected together to reference atmospheric pressure. If desired, a thermally insulated volume can be used in order to provide a better stability of the ambient pressure than that of the bare laboratory.

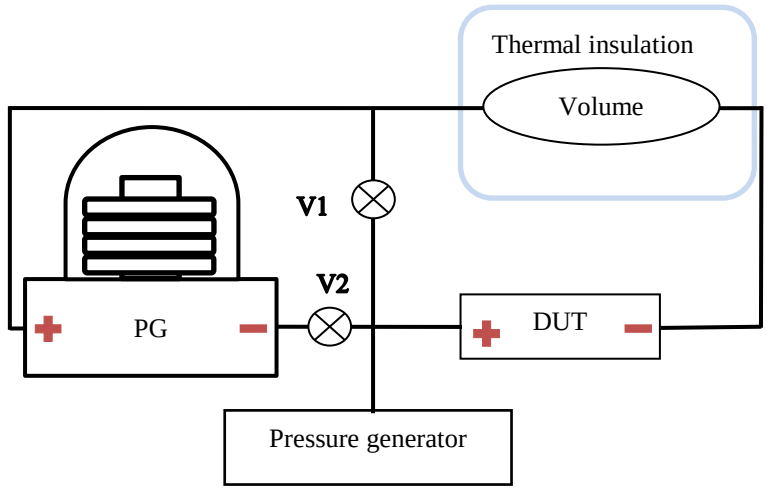

Figure 4.2.2.2: Set-up for negative gauge pressure calibration by applying sub-atmospheric pressure to the bell jar.

The pressure of the pressure balance is that at the <<Test +>> port which is connected to reference atmospheric pressure. In gauge mode, this pressure is by definition equal to 0 hPa. Thus, the negative pressure measured in the bell jar is given as piston load divided by the PCA area.

Pressure balance based methods: Calibration by using piston-cylinder mounted upside-down

The so-called “hanging piston” method is used in some laboratories. As in the previous example, the pressure balance is used in gauge mode, s. Figure 4.2.2.3.

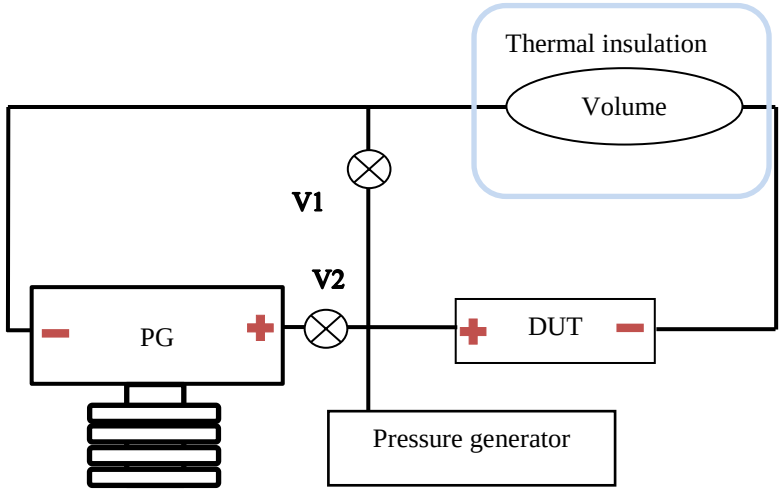

Figure 4.2.2.3: Set-up for calibration using a piston-cylinder assembly mounted upside-down.

In this setup, the <<Test +>> ports of the manometer and of the pressure balance are connected to the pressure controller, while the <<Test –>> ports are at the reference atmospheric pressure. A volume can be used for the same reasons as above. When equilibrium of the pressure balance is achieved, the negative pressure is given as piston load divided by the PCA area.

Mercury column manometer based method

Calibration of a pressure balance to measure negative gauge pressure (hanging piston method) using a mercury manometer is shown in Figure 4.2.2.4.

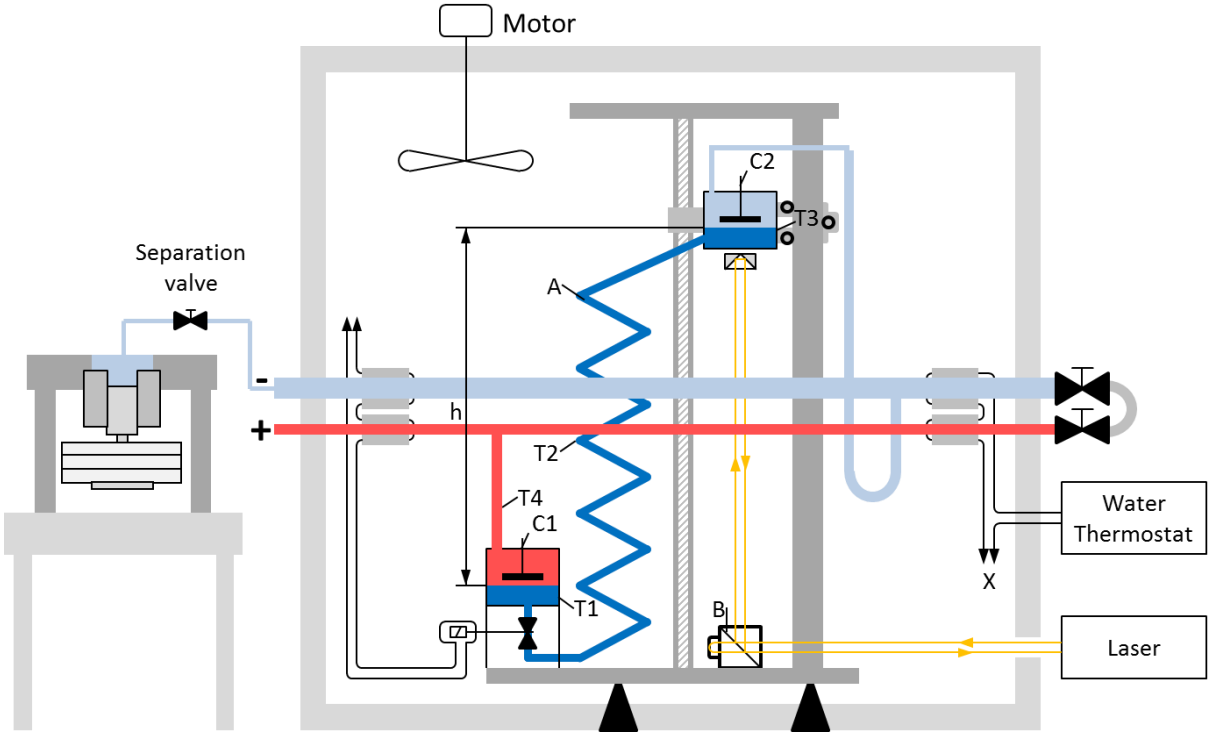

Figure 4.2.2.4: PTB's mercury manometer (right) and the DUC (left). T1 to T4 are thermometers, C1 and C2 are capacitor sensing plates, A is mercury tube, B is laser interferometer consisting of the laser head, beam splitter, corner cube reflector and photodetector.

The pressure port of the gauge pressure balance is connected to the '-' port of the mercury column manometer. The masses applied on the piston of the pressure balance are surrounded by ambient air and the '+' port of the mercury manometer is open to atmosphere. The negative pressure is calculated as the product of the gravity acceleration, mercury density and the height of the mercury column.

Method based on force balanced piston gauge FPG

If the reference port P_r of FPG is connected to atmosphere, the FPG defines a gauge pressure. During a negative gauge pressure measurement, the upper port P_h is connected to atmosphere. This connection is either direct or via an appropriate damping volume connected to the atmosphere via a narrow capillary with a low conductance (to ensure damping of the atmospheric pressure fluctuations). The lower port P_r is connected to a negative gauge pressure regulator and a device under test, see Figure 4.2.2.5. The direction of the force acting on the piston in the negative gauge pressure mode is the same as in the other modes. In negative gauge pressure mode, the calibration weight operates under the same conditions as in positive gauge mode. One needs to disconnect the hoses Test L and Test H from the VLPC. Thereafter, we connect the hoses from the FPG pressure ports (above the by-pass) in the following way: Test L from FPG to a minus port of the regulator and a minus port of the device under test; Test H to atmosphere and a plus port of the regulator and a plus port of the device under test. The vacuum ports are inactive and sealed. The most stable results are obtained by connecting the device under test to the lower port of the FPG and to the Test L simultaneously. To indicate the measured negative gauge pressure, either a terminal of the FPG or software FPG Tools installed on a PC are used. When working in negative gauge mode the software FPG Tools works as in the positive gauge mode and the values of the measured negative gauge pressure are displayed as positive

pressure values. Software FPG Tools is fully active in the negative gauge mode, ensures a full control of the FPG, including all the corrections, calibrations of the internal mass comparator, zeroing and control of the lubrication pressure.

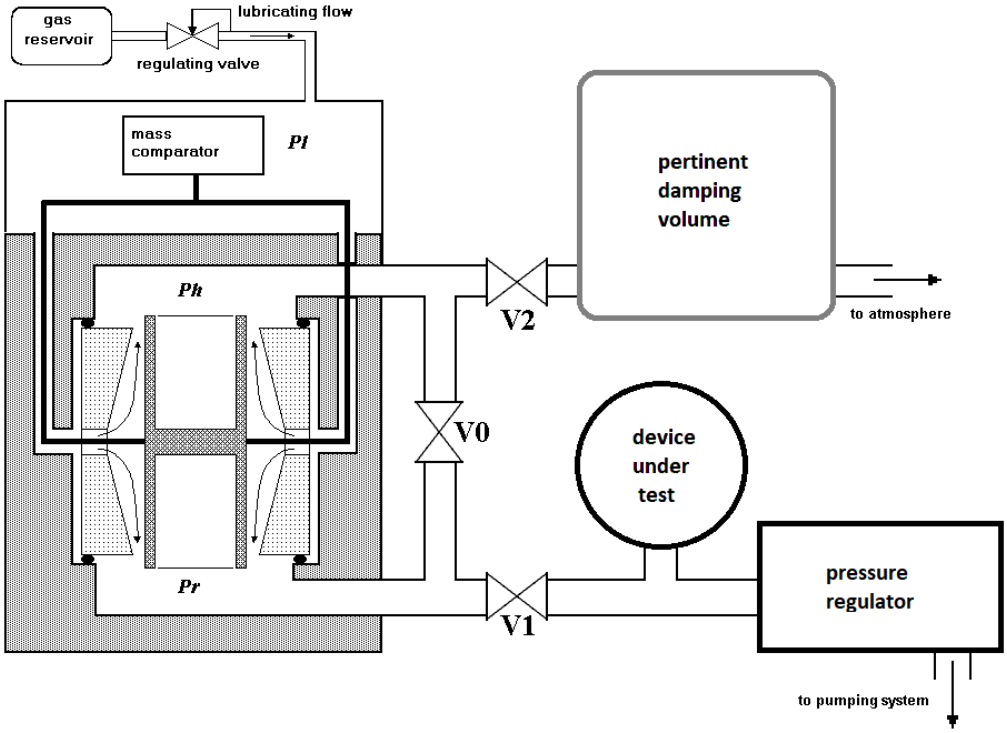

Figure 4.2.2.5: Principle of FPG's negative gauge pressure set-up.

Conclusion

Provided instruments capable of measuring absolute pressure are available, negative gauge pressures can be measured as accurately with balances as with mercury column manometers. This alleviates the needs for the latter, allowing such instruments to be phased out in line with EU policy. The uncertainties obtained for negative pressure measurements are 0.1 Pa + $1.0\times10^{-5}\times$|p| and equal those obtained for positive gauge pressure. They are better than those defined in the project protocol (0.1 Pa + $3.0\times10^{-5}\times$|p|). Moreover, they allow industrial needs for pressure measurements to be met [6].

4.3 Meeting EU restrictions of mercury use in measuring devices

This work covers the study of different methods, devices and technologies with the goal of developing new primary standards for the intermediate pressure range, to establish a mercury-free pressure metrology scale in the pressure ranges traditionally covered by mercury manometers and develop or improve pressure measuring instruments that allow more accurate and efficient traceable pressure measurements in industrial processes.

4.3.1 Alternative systems for pressure measurement using optical methods in the range 1 Pa to 10^4 Pa for absolute, positive and negative pressure

Several alternative optical methods with potential to measure pressure with high accuracy exist and are currently under investigation. The most promising optical technique so far conceived and demonstrated for assessment of pressure is based on refractometry performed in Fabry-Perot (FP) cavities. In this, the frequency of laser light, which is locked to a mode of the cavity in which gas is let in, is monitored. Changes in the density of the gas give rise to alterations in the refractivity, which, in turn, provide a shift of the beat frequency between the laser light and a stable reference light field, which is the measured entity. Since the assessment of gas by refractometry is often based upon a measurement of frequency, refractometric techniques have several unique properties, which give them several advantages as compared to conventional methods for assessments of the presence of gas.

The most sensitive optical techniques for assessment of refractivity rely on interferometry. The two main types of realization of refractometry are based on the Michelson interferometer (MI) or FP cavities. Since the latter allows for interferences created by a significantly larger number of passages in the device than the former (the number of passages of light in a FP cavity is approximately given by the so called finesse of the cavity, which can take a variety of values, in practice up to around 10^5, while the MI creates the interference solely by double passages of light in two arms), refractometry based on FP cavities can provide a significantly higher resolution than realizations based on the MI. Although a few early instrumentations have been realized around the latter, today virtually all are based upon FP cavities.

Fabry-Perot Refractometry

The first (and the simplest) type of FP cavity based refractometry systems utilized a single measurement cavity drilled in a low thermal expansion material. In these, the reference laser was often stabilized with respect to an external frequency reference, e.g. an iodine transition or a frequency comb. However, the drifts in these systems were significant and were often the limiting factor for their performance. To improve on this, systems based on two FP cavities were developed [7]. In these, one cavity is used as the measurement cavity while the other serves as a reference cavity. The justification of this is to eliminate common-mode effects of thermal drifts and relaxations of the spacer on the assessments. Besides reducing these, the introduction of a reference cavity also provides a well-defined reference light field to which the measured change in frequency of the measurement cavity can be assessed, measured as a radio-frequency (RF) beat frequency. An example of a FP refractometry for assessment of pressure is given in Figure 4.3.1.1.

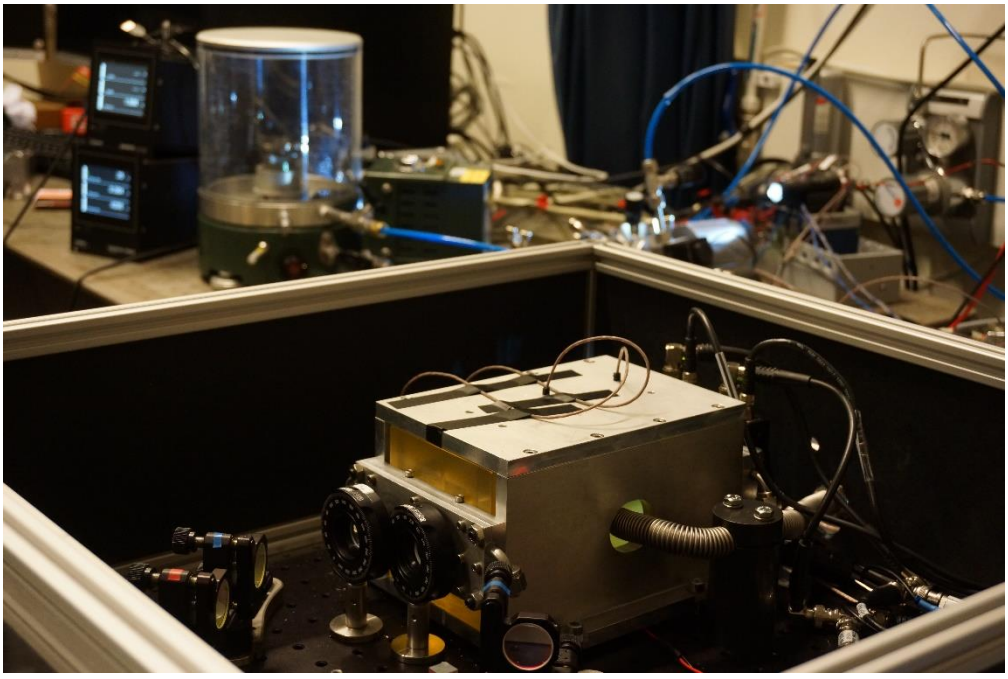

Figure 4.3.1.1: An example of a dual Fabry-Perot cavity refractometry for assessment of pressure.

Conventional FP based refractometry is often limited by drifts of the length of the cavity. Although there are means to control the measurement environment, primarily the temperature, and the material of the cavity spacer can be chosen from a low thermal expansion point of view and be subjected to treatments, this puts severe restrictions on the use or refractometry for everyday assessments. Work was therefore devoted to means to reduce the influence of drifts on the assessments of pressure. With respect to this, since modulation techniques previously have demonstrated an excellent ability to reduce drifts and noise in various types of measurement systems, it was considered whether it was possible to introduce a modulation methodology also in refractometry. It was found that suitable means to do this was to modulate the amount of gas in the cavity. This methodology was analysed in-depth and has been described in [7-9].

To benefit from the scrutiny of the gas modulation concept in these series of papers [7-9], the remaining part focused on a feasibility study of gas modulated refractometry by the realization of a second-generation refractometer system based on modulation of the gas in the cavity, hereafter termed GAs MOdulation

Refractometry (GAMOR). In this, the measurement cavity is repeatedly filled and evacuated with the gas whose properties are to be assessed. This modulation, which repeatedly provides a zero-pressure assessment, significantly (by orders of magnitude) reduces the effects of cavity drifts, so as to realize a “cavity-drift-corrected” methodology for assessment of pressure. This shows that the GAMOR realization does not have the same strong requirements on well-stabilized temperature conditions as ordinary refractometry. Since it is less subjected to environmental restrictions, it provides less constrains on material properties and relaxes the requirement of stabilization of the temperature of the cavity.

To allow a broader implementation of the technique (e.g. for the benefit of potential stakeholders), in the realization of the instrumentation, the project prioritized the use of off-the-shelf optical and electrical components. To demonstrate the possibility of constructing an easily-operated instrumentation, work was performed to realize a fully-computer controlled system [10]. When referenced to a dead weight pressure scale the instrumentation demonstrated assessment of pressures in the kPa range (4303 and 7226 Pa) limited by white noise with standard deviations in the $3.2N^{-1/2}$ - $3.5N^{-1/2}$ mPa range, where N is the number of measurement cycles (each being 100 s long). For short measurement times (up to around 10^3 s) the system showed a (1σ) total relative precision of 0.7 (0.5) ppm for assessment of pressures in the 4 kPa region and 0.5 (0.4) ppm for pressures around 7 kPa, where the numbers in parentheses represent the part of the total noise that was attributed to the refractometer. This showed that if the measurement procedure is performed over short time scales, the inherent properties of the GAMOR methodology allow high precision assessments by using an instrumentation that is not actively temperature stabilized based on a cavity and gas-system that potentially is affected by outgassing and leaks. It was concluded that this opens up for a variety of applications within metrology; e.g. transfer of calibration and characterization of pressure gauges, including piston gauges.

Conclusion

FP based refractometry, when realized correctly, indeed is a very promising technique for high precision assessment of refractivity, density, and pressure (the latter under the condition that also the gas temperature can be accurately assessed). It was shown that when realized with GAs MOdulation Refractometry (GAMOR) sub-ppm precision of pressure in the kPa range has been achieved [10]. Furthermore, the upcoming new SI-system in which the Boltzmann constant, k_B, will be fixed, provides a possibility to realize a primary pressure standard based upon refractometry and the ideal gas law (instead of the ratio of a force and an area). It can be concluded that optical methods in general, and refractometric techniques in particular, will have a large impact on pressure metrology in the future.

4.3.2 Investigation of the use of pressure balances as reference standards for barometric pressure as an alternative to mercury manometers

In primary liquid column manometers, mercury is usually used as a manometric liquid because of its well-known, stable density. Mercury manometers are operated by very few European NMIs, but are still used by numerous calibration, industrial and research laboratories. However, the Commission Regulation (EU) No 847/2012 of 19.9.2012 has restricted the use of mercury in barometers and sphygmomanometers for industrial and professional use from 10 April 2014 onwards. The objective of this study was to demonstrate that pressure balances can replace mercury manometers as standards for absolute barometric pressure.

A pressure balance consists of a vertical piston freely rotating within a cylinder. The two elements of well-machined quality define a surface called the 'effective area'. The pressure to be measured is applied to the base of the piston, creating an upward vertical force. This force is equilibrated by the gravitational downward force due to masses submitted to the local gravity and placed on the top of the piston. The piston is a part of the load. Sometimes, for practical reasons, and essentially at low pressure, the cylinder rotates instead of the piston. The principle and the test methods are exactly the same in this case. The pressure is transmitted to the movable element by a fluid which might be a gas (usually dry nitrogen or air). As the balance measures an absolute pressure, the masses are submitted to vacuum. The residual pressure in the bell jar around the masses, see Figure 4.3.2.1, creates a force in opposition to the measured pressure. The residual pressure has to be measured and added to the pressure created by the masses. The general definition of the pressure measured by the balance is obtained by analysing the different components of the forces applied to the system.

In order to determine the measurement capabilities of pressure balances, their lowest possible uncertainty was analysed. The following uncertainty components should be considered: repeatability of the balance; uncertainty of the effective area of the piston-cylinder assembly; uncertainty of the masses; uncertainty of the local gravity acceleration; uncertainty due to the temperature of the balance; uncertainty due to the thermal expansion coefficient of the piston-cylinder assembly; uncertainty due to the head correction; uncertainty due to tilt; uncertainty due to spin rate and/or direction, if applicable; uncertainty of the residual pressure.

Figure 4.3.2.1: Examples of pressure balances for absolute pressure measurement.

Herewith, the typical expanded uncertainty of the pressure measurement is $U(p) = 1.5\times10^{-5}\times|p| + 0.5$ Pa, which meets the target uncertainty. The mostly limiting factor is the effective area, whose uncertainty can be reduced.

In the range from 1 Pa to 15 kPa, the force-balanced piston gauges described in section 4.1.2 provide an additional alternative to mercury manometers.

An analysis of the results of CIPM and EURAMET comparisons in which Hg-manometers and pressure balances were used as reference standards has been performed. This analysis focused on the uncertainties achieved for the transfer standards by the two different types of the reference pressure standards.

Uncertainties declared by different institutes in the CIPM and EURAMET comparisons versus target pressure, in the range from 1 Pa to 100 kPa, where Hg manometers or pressure balances (excluding digital piston gauges) have been used as reference standards, are presented in Figure 4.3.2.2.

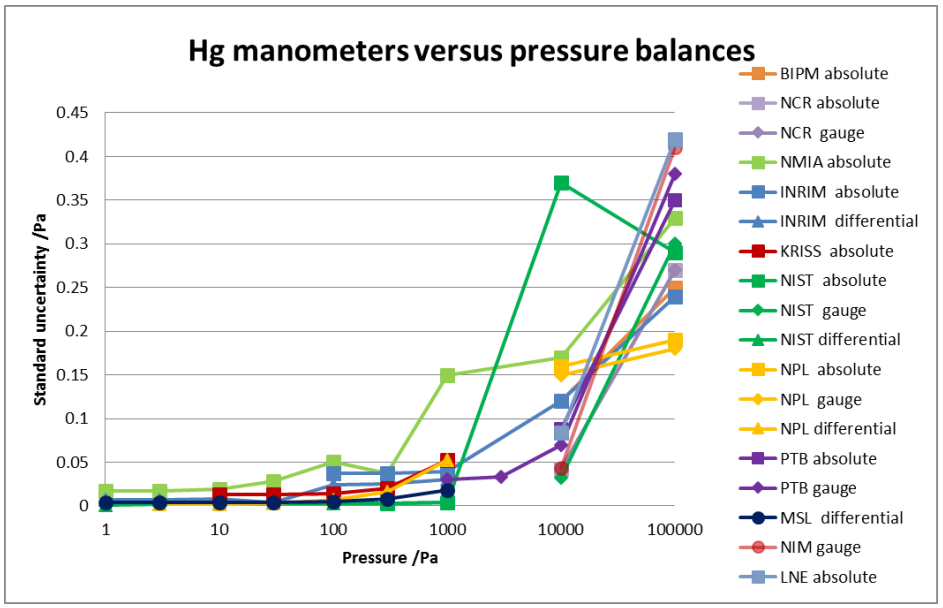

Figure 4.3.2.2: Uncertainties of pressure measurements carried out within international comparisons.

Among the participants of those comparisons, there were only two cases where pressure balances were used: MSL and NIM. Their uncertainties are very similar or even lower than the uncertainties obtained with Hg

manometers. These are two isolated cases, which however demonstrate that similar or even lower uncertainties can be achieved than with Hg manometers. The results clearly show that traceability is ensured for pressure balances in this pressure range with the target uncertainty of $1.5\times10^{-5}\times|p| + 0.5$ Pa.

Conclusion

International key comparisons with application of piston gauges and mercury manometers were analysed, conditions stated and methods described, with which piston gauges can serve as alternatives to mercury manometers with comparable or even better measurement capabilities. This enables users of mercury-containing pressure devices to make an appropriate replacement and, thus, to meet the restrictions for the use of mercury in pressure measurements.

4.4 Establishing a calibration service

With the development of new reference standards, calibration methods and approaches within the project, new measurement and calibration capabilities were created by the project partners, and an extended high-accuracy calibration service was established. This calibration service covers the range of approximately -10^5 Pa to 10^4 Pa of gauge pressure and approximately 1 Pa to 10^4 Pa of absolute pressure. For the calibration service, measurement capabilities are used which are based on absolute and gauge pressure type pressure balances, mercury manometers, force-balanced piston gauges and diving bell manometers. In the pressure range below 15 kPa, calibration service is offered which involves state-of-the-art reference standards and instruments under calibration with a resolution as low as 1 mPa. Over the whole pressure range of -10^5 to 10^4 Pa, the calibration service is provided with an uncertainty better than $3\cdot10^{-5}\times|p| + 1$ Pa. This uncertainty level satisfies the needs of the accredited calibrating laboratories and industrial companies. The measurement techniques, methods and reference standards used for the calibration service are listed in the following.

- FPG piston gauges of the NMIs have been calibrated and allow them to provide calibration service in the range of 15 kPa of absolute and gauge pressure. CMI has set up a calibration service, using FPG, for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa. PTB has characterised an FPG as primary standard which, after a validation against vacuum standards and a comparison against pressure balances and a mercury manometer, is used to provide the required calibration service for absolute and gauge pressure up to 15 kPa with an uncertainty lower than 30 ppm + 20 mPa. At CEM, an FPG was calibrated against a dead-weight pressure balance Ruska 2465 and the mercury column as reference. The characterised FPG is now used to provide a calibration service for absolute pressure up to 15 kPa with an uncertainty of 30 ppm + 20 mPa. Moreover, CEM has successfully tested capability of their mercury manometer to measure negative pressures and herewith can provide an improved calibration service in the range from approximately -100 kPa to -15 kPa. With piston gauges, comparable or partially even lower uncertainties for negative gauge pressure calibrations were obtained than with the mercury manometer at CEM, and, therefore, a pressure balance Ruska 2465 and an FPG were implemented there for providing calibration service, too.
- LNE has developed and validated an automated set up for calibration of negative and positive gauge pressures with an uncertainty below $3\cdot10^{-5}\times|p| + 1$ Pa. Moreover, a service for the calibration of specific instruments for customers allowing automatic calibration of pressure measuring instruments at a level better than $3\ 10^{-5}p + 1$ Pa was implemented.
- At PTB, a new negative gauge pressure balance (UDKM) was designed and built which can accommodate high-quality piston-cylinder assemblies (PCAs) of different types and manufacturers, including primary PCAs, in upside-down orientation. In addition, the primary mercury manometer was successfully tested and is used to provide negative gauge pressure calibrations, complementarily to absolute and gauge pressure calibrations in the 200 kPa range commonly available with the mercury manometer.
- LNE has set up a calibration service, based on the use of pressure balances of the type PG7000 and FPG, for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa. LNE has set up a calibration service, based on the use of pressure balances of the type PG7000 and FPG, for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa.
- IMT has established an accredited calibration service for negative gauge pressure in the range from 0 down to approximately -100 kPa, based on absolute pressure balance and absolute pressure transducers, with uncertainty better than $3\ 10^{-5}p + 1$ Pa. Target users for this service are secondary accredited laboratories and other NMIs in South-East Europe as well as industrial users in this geographic area.

- CMI has developed a transportable middle vacuum range calibration apparatus, based on a transportable vacuum chamber and a pumping set together with an SRG and a set of the CDGs for the absolute pressure range up to ca. 13 kPa, and provided a calibration service at an end user site.
- Guidelines were developed and calibration service was established at CMI for gauge pressures in the range 20 Pa to 15 kPa using conical piston gauges (e.g. Pressurements V1600/4).

Information about the new measurement capabilities and calibration services was disseminated to potential customers, mostly accredited and industrial calibrating laboratories. An example of calibrations provided within the new calibration services is given below.

4.4.1 Calibration service for FPG

Customers' FPGs can be calibrated using dead-weight pressure balances and FPG as reference. At PTB, the traceability chain for both, the reference dead-weight pressure balance (Ruska PG) and FPG, is realised as shown in Fig. 4.4.1.1. Herewith, the effective areas of the Ruska PG and the FPG could be determined with relative standard uncertainties of 5.7 ppm and 8 ppm, respectively.

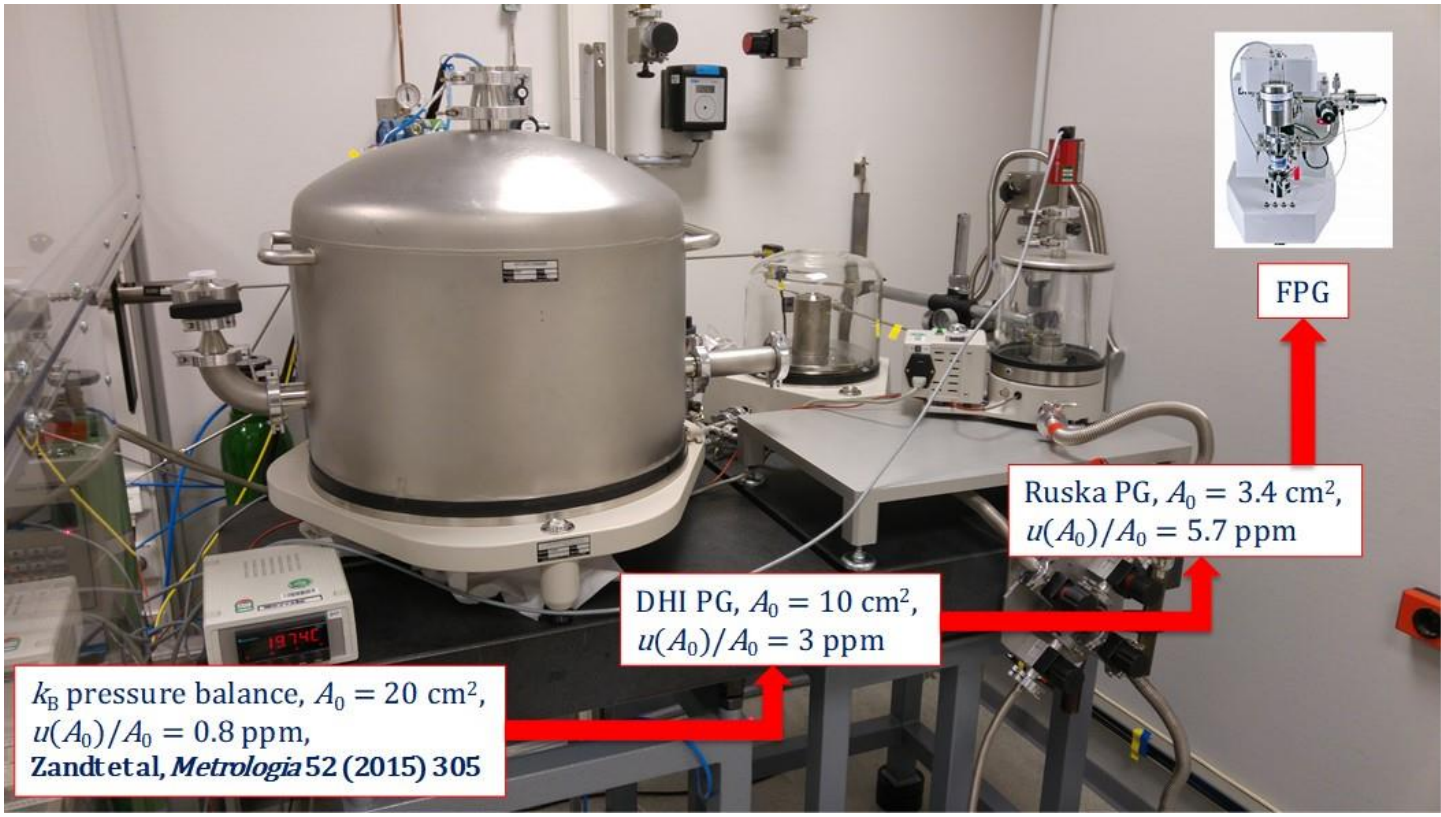

Figure 4.4.1.1: Traceability for reference dead-weight pressure balance (Ruska PG) and FPG used for calibration of customers' FPGs.

In addition, the FPG was characterised as a primary pressure standard by dimensional measurements and flow modelling as described in section 4.1.2. With the results of the experimental and theoretical studies of the FPG, its effective area could be determined with a relative standard uncertainty of 4.3 ppm. The combined relative standard uncertainty of FPG is 4.3 ppm + 5 mPa.

First, the load cell of the customer's FPG was calibrated using reference weights. Second, the effective area of the customer's FPG was determined by a calibration against the pressure balance in the pressure range 3 to 15 kPa, Figure 4.4.1.2 left. Finally, the instrument was compared with the reference FPG over the whole pressure range from 1.3 Pa to 15 kPa, Figure 4.4.1.2 right.

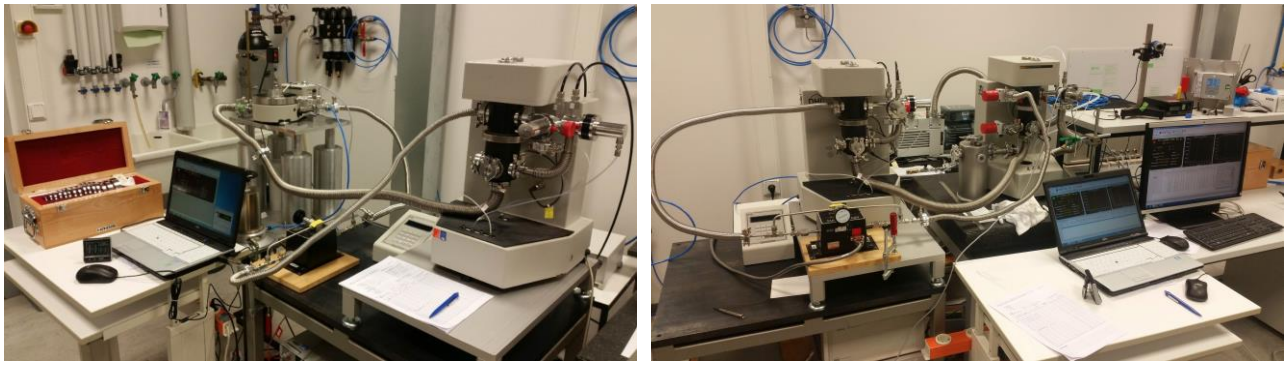

Figure 4.4.1.2: Calibration of a customer's FPG in gauge mode against a reference dead-weight pressure balance (left) and an FPG (right).

Based on the results of all measurements, the standard uncertainty of pressure measurement of the calibrated FPG was found as 15 ppm + 10 mPa.

Conclusion

The project has established a calibration service in the range of approximately -10^5 to 10^4 Pa of gauge pressure and approximately 1 Pa to 10^4 Pa of absolute pressure with an accuracy level of $3 \cdot 10^{-5} \times |p| + 1$ Pa which is required by accredited calibrating laboratories and industrial companies. The calibration service includes new calibrating capabilities for state-of-the-art pressure measurement instrumentation such as force-controlled piston gauges with a resolution of 1 mPa.

4.5 Summary

- Three different techniques were applied to develop and characterise primary pressure standards for low pressure below 100 kPa: liquid column manometry, force-balanced piston gauges and refractometry. A new interferometric liquid column micromanometer for 2 kPa range of absolute and gauge pressure was developed. Force-balanced piston gauges of FRS and FPG type, operated in the range 15 KPa of absolute and gauge pressure, were characterised as primary pressure standards by applying dimensional measurements on their piston-cylinders and state of the art flow modelling. A dual Fabry-Perot cavity (DFPC) to measure gas densities by measuring the refractive index was designed and investigated, which, after further studies, can serve for a primary realisation of the pressure unit in the range of about 100 kPa of absolute pressure.
- Methods were developed for accurate, weather-independent calibration of low gauge pressure instruments. These new methods allow calibration with uncertainties below 3 Pa + $2 \cdot 10^{-4} \cdot p$ even at unstable weather conditions. It has also been shown that there was a possibility to carry out calibrations, despite of the severe ambient conditions, by circumventing the constraints related to the instability of the ambient atmospheric pressure by means of either a closed reference volume or a hermetic chamber. The methods are applicable to calibrate mechanical and electronic low gauge pressure measuring instruments.
- Methods were developed for negative gauge pressure calibration based on absolute and gauge pressure balances, FPGs and liquid column manometers. Provided instruments capable of measuring absolute pressure are available, negative gauge pressures can be measured as accurately with balances as with mercury column manometers. This alleviates the needs for the latter, allowing such instruments to be phased out in line with EU policy. The uncertainties obtained for negative pressure measurements are 0.1 Pa + $1.0 \times 10^{-5} \times$|p| and equal those obtained for positive gauge pressure. They allow industrial needs for pressure measurements to be met.
- A study was carried out of different methods, devices and technologies with the goal of developing new primary standards for the intermediate pressure range, to establish a mercury-free pressure metrology scale in the pressure ranges traditionally covered by mercury manometers and to develop or improve pressure measuring instruments that allow more accurate and efficient traceable pressure measurements in industrial processes. FP based refractometry was demonstrated to be a very promising technique for high precision assessment of refractivity, density, and pressure (the latter under the condition that also the

gas temperature can be accurately assessed). It was shown that when realized with GAs MOdulation Refractometry (GAMOR) sub-ppm precision of pressure in the kPa range can be achieved. Furthermore, the new SI-system in which the Boltzmann constant, k_B, will be fixed, provides a possibility to realize a primary pressure standard based upon refractometry and the ideal gas law (instead of the ratio of a force and an area). The optical methods in general, and refractometric techniques in particular, will have a large impact on pressure metrology in the future. International key comparisons with application of piston gauges and mercury manometers were analysed, conditions stated and methods described, with which piston gauges can serve as alternatives to mercury manometers with comparable or even better measurement capabilities. This enables users of mercury-containing pressure devices to make an appropriate replacement and, thus, to meet the restrictions for the use of mercury in pressure measurements.

- A calibration service in the range of approximately -10^5 to 10^4 Pa of gauge pressure and approximately 1 Pa to 10^4 Pa of absolute pressure with an accuracy level of $3 \cdot 10^{-5} \times |p| + 1$ Pa, which is required by accredited calibrating laboratories and industrial companies, was established. The calibration service includes new calibrating capabilities for state-of-the-art pressure measurement instrumentation such as force-controlled piston gauges with a resolution of 1 mPa.
- The collaborative approach produced added value that individual partners could not achieve by themselves. Especially, the characterisation of force-balanced piston gauges as primary standards required and was performed using specific key expertise and measurement capabilities of the partners in such areas as dimensional metrology, liquid column manometry, pressure balance manometry, vacuum metrology and gas flow modelling by the methods of the Rarefied Gas Dynamics. Furthermore, complementary contributions by the partners in the fields of liquid density and viscosity, and solubility, permeability and diffusivity of gases in oils were crucial for the development of the primary interferometric oil micromanometer.

5 Impact

The project impacts many industries. The reliability and accuracy of low gauge and the differential and absolute pressure measurements were improved at NMIs, accredited commercial laboratories, in industry and with end-users. It also has a direct and indirect positive influence on the European economy, environment and society.

The concept, design and construction of the new oil-based interferometric micromanometer serving as a primary standard were produced. Main uncertainty sources were identified and prerequisites for operation of the micromanometer at the required uncertainty level derived. The key impact factors on the uncertainty, namely interferometric determination of the position of the floats, temperature homogeneity, stability of the density and residual pressure of the manometric oil, were identified and their uncertainty contributions estimated. The density uncertainty related to the oil properties and realisation of the temperature-of-floatation method contributes less than 10 ppm. The uncertainty of the difference of the liquid columns' heights is below 10 nm, corresponding to 0.1 mPa uncertainty in pressure. Herewith, the combined uncertainty of absolute and gauge pressure measured with the new micromanometer is smaller than $1\ \text{mPa} + 2 \times 10^{-5} \times p$. This new measurement capability allows providing traceability to other NMIs and accredited calibration laboratories in the pressure range below 2 kPa on a significantly lower uncertainty level.

The effective area of three FPG8601, one FRS4 and one FRS5 in the whole operational pressure range of each device varying between 1 Pa and 15 kPa was computed using state of the art methods of flow modelling and dimensional measurements. These results are in full agreement with the experimental effective areas obtained by cross-float and with the areas reported by the manufacturer. As a result of this characterization, it was possible to reduce the relative standard uncertainty of the effective area to below 10 ppm. With the theoretical and experimental characterisation of the force-balanced piston gauges as primary and secondary standards, the pressure uncertainty below the target uncertainty of $0.01\ \text{Pa} + 1.4\ 10^{-5}\ p$, was achieved. Finally, these instruments can be operated as primary pressure standards and serve for improved dissemination of the pressure scale to the industry in the range up to 15 kPa.

With the development in the field and the increased knowledge of optical methods, in particular the FP refractometer, it became possible to start taking advantage of more advanced modes of operation of instruments which can yield advantages over conventional static measurements. The optical methods are by design fast. Therefore, the methods, and in particular FP based refractometer systems, can also provide continuous measurement information. These features are of significant interest, in particular, when investigating the dynamic response of various types of pressure gauges, which is required in many industrial

processes. The study of the optically based methods for pressure measurement provides the basis for stakeholders to accept new potential standards. It allows for more input from other fields and research areas, for industrial development and commercialization from European industry, and, finally, for the overall development in the field to contribute to the societal grand challenges. When the instrumentation is to be used for transfer of calibration, high accuracy can be obtained even if not all gas and cavity material characterizations have been performed. In this case, to allow for assessment of the non-linear contributions from these entities, the system needs to be characterized using a standard at a set of pressures. When this is done, the system will inherit the accuracy of the standard. To allow for a broader implementation of the technique (e.g. for the benefit of potential stakeholders), fully computer-controlled instrumentation that is based on off-the-shelf optical and electrical components and sturdy components will be developed in the future. NMIs and the academic community were encouraged to contribute to the ongoing development of optical techniques for the assessment of the presence of gas and its properties to enable collective advances in the field.

PTB has characterised an FPG as primary standard which, after a validation against vacuum standards and a comparison against pressure balances and a mercury manometer, can be used to provide the required calibration service. For the new primary oil micromanometer, selection and characterisation of manometric oils has been finished, a concept of the micromanometer developed and laser interferometric systems produced. LNE has set up a calibration service, based on the use of pressure balances of the type PG7000 and FPG, for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa. CMI has set up a calibration service, using FPG, for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa.

Methods for calibration of FPG against pressure balances and other primary standards in the range from (1 to 15) kPa were studied and provide a basis for the extended calibration service (i.e. in the range of approximately (-10^4 to 10^4) Pa of gauge pressure and approximately (1 to 10^4) Pa of absolute pressure). The calibration methods and techniques are now used to provide the calibration service for negative gauge pressures in the range (-10^5 to 0) Pa. Engagement with industries that utilise pressure in the intermediate range (1 to 10^4) Pa facilitates the uptake of the technology and the measurement infrastructure developed by the project. This was achieved by local meetings with stakeholders in Spain, Germany, Czech Republic, Turkey, Netherlands. New or improved calibration services were provided based on the use of dead weight pressure balances and FPG, for absolute pressures in the range (0 to 15) kPa with an uncertainty of $3 \cdot 10^{-5} \times p$ + 20 mPa.

In particular, PTB finished characterisation of an FPG as a primary pressure standard and established a calibration service for absolute pressure up to 15 kPa with an uncertainty of 30 ppm + 20 mPa. This service uses FPG and, in the future, will be traced to the new primary standard - oil micromanometer. The calibration service is used to provide German accredited pressure labs with traceability from PTB with an appropriate measurement uncertainty. LNE set up a calibration service for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa. This service is based on the use of pressure balances of the type PG7000 and FPG. CMI set up a calibration service for absolute pressures in the range 0 to 15 kPa with an uncertainty of 30 ppm + 20 mPa focusing on utilisation of FPG. PTB finished characterisation of an FPG as a primary pressure standard and can provide a calibration service for absolute pressure up to 15 kPa with an uncertainty lower than 30 ppm + 20 mPa. Furthermore, CMI developed a transportable middle vacuum range calibration apparatus, based on a transportable vacuum chamber and pumping set together with an SRG and a set of the CDGs for the absolute pressure range up to ca. 13 kPa, and provided a calibration service at an end user site. PTB contacted German companies and provided knowledge about the new calibration methods developed in this project with improved accuracy of the pressure measurement to better than $2 \cdot 10^{-4} p$ + 3 Pa.

A transfer standard for pressures in the range 1 Pa to 10 kPa was developed, built and evaluated. It is composed of a resonant silicon gauge (commercially available RSG), of 130 kPa full scale in absolute pressure, and three capacitance diaphragm gauges (CDGs) of 130 Pa, 1.3 kPa and 13 kPa full-scale respectively. In the most pressure range, the uncertainty of the transfer standard is as low as $1 \times 10^{-4} \times p$, except for pressures lower than 30 Pa. For the latter, the resolution of the standard and the contribution of the thermal transpiration effect are getting preponderant. This relatively low contribution, compared with the usual stability performances of capacitance diaphragm gauges, is provided by the stability of the slope correction of a commercially available resonant silicon gauge, with which the CDG of 10 kPa full range is rescaled. As the slope correction of the RSG is stable, but an offset drift can occur, several rescaling pressure points were required and could be set up. This transfer standard is suitable to demonstrate metrological capabilities of calibration services, which are rarely lower than a few $10^{-4} \times p$. It is suitable for comparisons between National metrology institutes that need to validate uncertainties as low as a few times $10^{-5} \times p$, knowing that the transfer standard can be improved. This transfer standard keeps open options for further improvement, e.g. by taking

into account its drift in appropriate way. This development has also shown that instruments composing the transfer standard with the associated rescaling procedure can be used as a working standard in the range 5-130 kPa. By extension, the standard developed within the project is suitable to be either a working or a transfer standard in the absolute pressure range from 1 Pa to 130 kPa. The uncertainty contributions depend on the linearity and stability of the linear correction coefficient of the RSG and the reproducibility of the shape of the calibration curves of the CDGs. The transfer standard was characterised within a comparison organised by LNE with measurements performed at FCT-UNL, CEM, IMT, TUBITAK and LNE. The new transfer was demonstrated to have an uncertainty of $1\times10^{-4}\times p$, where p is the measured pressure, and can now be used by national metrology institutes for comparison purposes and to provide traceability to calibrating laboratories and industry in the pressure range up to 130 kPa.

Based on the CDG technology, advanced transfer standards utilising 10 mTorr gauges by INFICON LI, model STRIPE CDG45Dhs, for absolute pressures below 10 Pa were developed and their performance was tested. These transfer standards provide improved traceability for companies and laboratories working under vacuum conditions below 10 Pa. The resolution of 3×10^{-4} Pa obtained with the digital output represents a similar characteristic as with the common, metrology quality's CDG widely used for traceability in the metrological laboratories. The long-term stability of the CDG sensitivity coefficient at level of $5\times10^{-4}\times p$ was proved achievable for laboratory conditions and the same gas. For different gases and wider ranges of the temperature, the transfer standard can serve with an additional uncertainty when respective corrections are made. This new transfer standard opens a new traceability ways for industrial processes which run under semi-dynamic conditions or with different gases for which application of SRGs is not possible. The reason is that, in some special cases of metrology laboratory praxis, for pressures below 1×10^{-1} Pa, CDGs can perform better than SRGs when demanding a quick response or when measuring gas mixtures of roughly known components ratio (mixture composition strongly affects SRGs, but for CDGs, it only affects the thermal transpiration correction). The ambient temperature study and thermal transpiration coefficient examination provide the information for the limits of using CDGs in this special cases. A STRIPE CDG45Dhs based transfer standard package would be relatively cheap and easy to be transported from NMI or other calibration laboratory to the users in industry.

New capabilities were created for realising a transfer standard utilising optical methods, in particular the FP refractometer. Herewith, it became possible to start taking advantage of more advanced modes of operation of instruments which can yield advantages over conventional static measurements. All this make refractometry suitable for assessment of pressure under various conditions. For example, it has a large potential to be a convenient means of accurate transfer of calibration between different pressure regions. This opens up for a variety of applications within metrology, e.g. transfer of calibration and characterization of pressure gauges, including piston gauges. As recently was demonstrated, the FP refractometer technique can be referenced to existing standards, for example a pressure balance or a manometer. Due to the excellent precision of FP refractometry, when referenced, and under the condition that the non-linear contributions (refractivity and density virial coefficients) are known with sufficient accuracy, the FP system can inherit the accuracy from the standard. If primary standards are available, which is often the case at NMIs, and thanks to its large dynamic range, the refractometer can be used to transfer accuracy from a standard working in one pressure range to another system working in another range, only limited by the resolution of the refractometer. With more detailed investigation of the potential of optical methods for transfer of calibration, accuracy of pressure assessments can be improved, in particular, at low pressure and vacuum applications. When the instrumentation is to be used for transfer of calibration, high accuracy can be obtained even if not all gas and cavity material characterisations have been performed. In this case, to allow for assessment of the non-linear contributions from these entities, the system can be characterized using a standard at a set of pressures. When this is done, the system will inherit the accuracy of the standard. With a new methodology to be developed in the future, which will allow the system to be characterized using a standard at a set of pressures, the accuracy of optically based pressure-measuring instruments used for transfer of calibration can be improved.

LNE has implemented a service for the calibration of specific instruments for customers allowing automatic calibration of pressure measuring instruments at a level better than $3\ 10^{-5}p$ + 1 Pa. IMT has established an accredited calibration service for negative gauge pressure in the range from 0 down to approximately -100 kPa, based on absolute pressure balance and absolute pressure transducers, with uncertainty better than $3\cdot10^{-5}p$ + 1 Pa. CEM has successfully tested capability of their mercury manometer to measure negative pressures and herewith can provide an improved calibration service in the range from approximately -100 kPa to -15 kPa. LNE, aided by CEM, CNAM, CMI, PTB and TUBITAK has described different methods and recommendations for negative gauge pressure measurements.

The project's outcome was and will be further disseminated to industrial stakeholders such as manufacturers of pressure measuring devices in the corresponding pressure range as well as end-users and calibration laboratories. Project results were presented through 13 contributions at several conferences, congresses and meetings such as the 20th International Vacuum Congress, the 18th International Metrology Congress, the 6th CCM International Conference on Pressure and Vacuum Metrology in conjunction with the 5th International Conference IMEKO TC 16, and 5th European Conference on Microfluidics µFlu18 in conjunction with the 3rd European Conference on Non-Equilibrium Gas Flows NEGF18. Several national workshops were also organised such as the project's workshop hosted by CMI in November 2016 and a stakeholder training workshop that took place in January 2017 in Turkey. The project's final, international workshop, aimed at collaborators and stakeholders, and devoted to measurement and traceability issues in the gauge and absolute pressure ranges below 15 kPa, improvement of pressure measurement accuracy at variable ambient atmospheric conditions and industrial environment, was held in May 2018 in Borås, Sweden, Knowledge was disseminated to end-users through training courses, and industrial stakeholders were contacted to create an advisory group in order to exchange information with the consortium and to ensure that the project is delivering relevant information to end-users. The participation of industrial partners in the project also helped to align the project with industrial needs.

Impact on industrial and other user communities
The project established new primary standards and supports the dissemination of the pressure scale in the intermediate pressure range (1 to 10^4) Pa. This improves the reliability and accuracy of low gauge, differential and absolute pressure measurements at many levels, from NMIs, through accredited commercial laboratories, to the end-users. This traceability is the basis for more accurate pressure measurement (e.g. for the cleanroom technologies and processes) and will allow realisation of tighter tolerances of non-equilibrium conditions and, as a consequence, reduce energy expense and costs without the loss of safety, sterility and precision. The costs of operation with toxic and nuclear materials as well as of the storage of environmentally dangerous toxic and nuclear wastes should also be reduced and the safety of these processes increased.

The project established a calibration service that gives end-users an access to calibrations in the range (0 to 15) kPa of absolute pressure with uncertainties on the level of $3 \cdot 10^{-5} \times p$ + 20 mPa, which will be improved in the near future by a further reduction of the uncertainties down to $3 \cdot 10^{-5} \times p$ + 5 mPa. Such conditions will be beneficial for example for more efficient and safe use of airspace by aircraft. An accredited calibration service for negative gauge pressure in the range from 0 down to approximately -100 kPa, based on absolute pressure balance and absolute pressure transducers, with uncertainty better than 3 10-5p + 1 Pa was established.

Dissemination of traceability amongst NMIs provides access to improved capabilities for national and accredited laboratories in Europe and supports consistency in measurement capabilities. Additionally, it benefits the industries that rely on such calibration services. Information on the calibration services was disseminated via accredited bodies (for pressure) in Europe, calibration laboratories and their committees of experts for pressure. Transportable middle vacuum range calibration equipment was created to provide a calibration service at an end-user site.

Impact on the metrological and scientific communities
Based on the project results, a recommended *mise en pratique* for assuring traceability in the range 1 Pa to 15 kPa using FPGs in both absolute and gauge mode were derived. This created a large impact on calibration laboratories and was presented to the accreditation authorities in Europe as well as to end-users and manufacturers of FPGs.

In the area of FPGs, knowledge transfer from experienced NMIs to those less experienced on how to use this new type instruments was proven to be very beneficial. On a broader scope, the project strengthened the collaboration of European NMIs and increased their competitiveness with NMIs outside Europe. Secondary accredited commercial laboratories gain now a better calibration service from the European NMIs, avoiding high costs for calibration of their standards abroad and increasing their calibration capabilities. A draft calibration guide including instructions for calibrating FPGs in both absolute and gauge mode was produced and submitted to EURAMET for revision and publishing as a EURAMET calibration guide.

Furthermore, improved calibration methods for positive and negative gauge pressure standards in the range from approximately -100 kPa to 15 kPa were developed. Accordingly, another EURAMET calibration guide, this time for positive and negative gauge pressure standards, was drafted which describes different calibration systems, conditions under which they are to be operated, procedures to be followed, uncertainties aimed at and the best working practices. The draft guide was presented to EURAMET TC-M members and will be, after approval, made available to end-users.

Impact on relevant standards
One of the project's main impacts is related to the Commission Regulation (EU) No 847/2012 of 19.9.2012 which restricts the use of mercury in barometers and sphygmomanometers for industrial and professional use. The execution of the Directive is facilitated by providing equivalent alternative pressure standards to mercury manometers. It supports the reduction in the number of mercury-containing pressure-measuring devices in Europe.

In addition, the consortium promoted the results of the project within the standardisation community and provided input into the standardisation process CCM WGPV (pressure and vacuum), COOMET TC 1.6 "Mass and related quantities", DIN NATG-D Standard Committee Technical Basics - pressure, flow, temperature and IMEKO TC 16 "Pressure and Vacuum Measurement".

Longer-term economic, social and environmental impacts
By improving the pressure scale at the NMI level in the range of lower gauge, absolute and differential pressure this project provides a better measurement capability. In combination with new calibration methods, a more adequate dissemination of the unit "pressure" is obtained. Further to this, European calibration laboratories and industry are able to engage with the new calibration services and to have their instruments calibrated within Europe without the need to send their devices to the US. This meets the demand of industry to obtain high accuracy calibration services in Europe, whilst making calibrations less time and cost consuming.

The following industries benefit from smaller uncertainties for low gauge, absolute and differential pressure measurement:

- The clean room technique is directly affected by smaller uncertainties of pressure measurement. To establish clean room conditions in e.g. pharmaceutical, semiconductor or nanotechnology industries different zones are separated by different local ambient pressure levels which prevent contaminated air entering a critical zone. With smaller uncertainties in pressure measurements, smaller pressure differences between these zones are possible which enables the use of more zones at a time but with the same effort in energy and costs. This makes new, more efficient, complex and energy saving clean room productions possible.

  Manufacturers developing measurement equipment or components for clean room techniques benefit from the enhanced measurement capabilities at the NMI level and the dissemination of the pressure scale to calibration laboratories.

- The chemical and petrochemical industry is subject to strict international requirements like PED and ATEX [Directives 97/23/EC and 949/EC]. Safety applications therefore benefit from smaller uncertainties in low negative and positive gauge measurement as these are used in fire protection systems in international and European legal standards and regulations [FM Approvals LLC, Class No. 2311, April 2008; Commission Regulation (EC) No 1497/2007]. These regulations guarantee a strong value of safety, protect the industrial infrastructure and mainly beware the environment from fatal situations.

  Manufacturers developing measurement equipment or components for chemical and petrochemical industry benefit from the project by enhanced measurement capabilities at the NMI level and the dissemination of the pressure scale to calibration laboratories.

- In power plants, smaller uncertainties of low gauge, absolute and differential pressure measurement are relevant for safety, efficiency and costs. Such safety systems help to identify environmentally harmful or toxic leakage and prevent pipes or vessels from bursting. In this way, they also protect the infrastructure and the environment. Therefore, efficiently controlled processes using measurands that avoid non-optimal operating conditions, are now more efficient, less cost intensive and avoid the production of unwanted by-products.

  Manufacturers developing measurement and safety equipment for the energy industry benefit from the enhanced measurement capabilities at the NMI level using their services directly or by using the calibration capabilities of associated calibration laboratories.

- Steadily increasing numbers of aircraft within European airspace have made it necessary to reduce the standard vertical separation (RVSM) between aircraft from 600 m to 300 m. Avionic altimeters use absolute pressure measurement for height detection, but only specially certified altimeters and autopilots are allowed to enter the RVSM airspace, and they need to be calibrated traceably to NMI standards. In the future, an even more intensive usage of the airspace will consequently increase the need for smaller uncertainty of low absolute pressure measurements.

Manufacturers developing avionic measurement equipment and the aircraft industry benefit from the enhanced measurement capabilities at the NMI level and the dissemination of the pressure scale to calibration laboratories.

The European mercury strategy [Commission Regulation (EU) No 847/2012 on 19/9/2012] restricts the use of mercury in barometers from 10 April 2014 which is an issue for research institutions and reference laboratories in the avionic industry and weather monitoring and forecast services, which all use mercury barometers. Many European NMIs also realise the pressure scale on low gauge, absolute and differential pressure using mercury based liquid column manometers and these devices usually contain 6 kg to 10 kg of mercury. Therefore, a new primary standard, using alternative manometric liquids such as oil, fulfils the EU demands and reduces the risk of accidental environmental pollution by mercury.

Many industries such as pharma-biotech, semiconductor, micro- and nano-technology, petrochemical, aviation, energy production, weather monitoring and forecast services benefit from the project's output and this strengthen the European industrial infrastructure for the development of new services and products (that rely on pressure). As a wider impact, Europe's innovative capacity is increased, leading to higher employment and wealth for society. Finally, the project has improved collaboration between European NMIs, in particular, between smaller/less experienced NMIs and more experienced NMIs.

6 List of publications

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