



Publishable Summary for 14IND06 pres2vac

Industrial standards in the intermediate pressure-to-vacuum range

Overview

The overall goal of this project was to enable 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. This work 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.

Need

This project enabled the SI traceable measurement of absolute, positive and negative gauge pressure in the intermediate range, 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. An amendment by the Commission Regulation restricts the use of mercury in barometers and sphygmomanometers for industrial and professional use from 10 April 2014. Therefore, support was required for the replacement of primary mercury manometers which are still in use in many research institutions and reference laboratories.

Objectives

The specific objectives of the project were:

- **To develop and characterise primary and transfer pressure standards** - for the 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 $3 \times 10^{-5} p + 1$ Pa independent of variable ambient conditions, and in industrial conditions to better than $2 \times 10^{-4} p + 3$ Pa. 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 will 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.

Progress beyond the state of the art

Primary and transfer pressure standards for a consistent dissemination of the pressure scale in the intermediate range (1 to 10^4) Pa

Primary pressure standards - dead-weight pressure balances and liquid column manometers - allow traceability of the pressure unit to the SI basis units kilogram, meter and second. The operating range of the dead-weight pressure balance is limited by approximately 5 kPa. The lowest pressure accurately measured with mercury manometers is approximately 1 kPa. Oil is an advantageous alternative to mercury due to its low density, low vapour pressure and much better stability of the free surface, but is not widely used because of a relatively large variation of the oil density with pressure. The project went beyond this by the *in-situ* measurement of oil density realised in a novel oil micromanometer.

New force-balanced piston gauges (FPG and FRS) allow gauge and absolute pressures to be accurately measured from 15 kPa downwards to zero, but have been used only as secondary standards so far. This project went beyond this by characterising FPG and FRS as primary standards. Flow models taking into account molecular properties of gas were developed and applied. Dimensional measurements were carried out on piston-cylinders and their 3D data produced. For the first time their effective area was determined theoretically as a function of variable pressure conditions. In this way, the effective area of the FPG and FRS was traced to dimensional measurements and the instruments were characterised as primary pressure standards.

Calibration methods for positive and negative gauge pressure standards in the range from approximately (-10^5 to 10^4) Pa

To solve the problem of inaccurate pressure calibrations due to unstable ambient conditions, new procedures and techniques for low differential pressures calibrations were developed and a calibration guide drafted. Herewith, the calibration uncertainty can be reduced down to $3 \cdot 10^{-5} \times p + 1$ Pa independently of variable ambient conditions. These methods and techniques are beneficial for accredited and industrial calibration laboratories.

EU strategy on restrictions of mercury use in measuring devices (barometers, sphygmomanometers)

Mercury manometers are operated by very few European NMIs, but are still used by numerous calibration, industrial and research laboratories. Commission Regulations restrict the use of mercury in barometers and sphygmomanometers for industrial and professional use. Within the project, two strategies were followed: first, an investigation of alternative standards based on refractometry techniques, and second: comparisons between mercury-containing and existing mercury-free pressure standards. The refractometry techniques were demonstrated to provide sufficient resolution and, under specified conditions and low pressures, accuracy comparable or even better than that of mercury manometers. 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.

Calibration service in the range of approximately (-10^5 to 10^4) Pa of gauge pressure and approximately (1 to 10^4) Pa of absolute pressure

Advanced FPGs, which accurately measure pressure in the range 15 kPa downwards to zero, could only be calibrated against dead-weight pressure balances or mercury manometers at pressures above a few kilopascals. Below these pressures, there were no alternative pressure standards. With the development of

new secondary pressure standards and characterisation of existing primary standards, as well as the development of new calibration methods, a reference was provided and conditions created to offer an adequate calibration service in Europe. Herewith, the traceability for industrial calibration services in the range of approximately $(-10^5 \text{ to } 10^4)$ Pa of gauge pressure and approximately $(1 \text{ to } 10^4)$ Pa of absolute pressure is now provided. Preconditions were created to establish and maintain a calibration service with an uncertainty better than $2 \cdot 10^{-4} \times p + 3 \text{ Pa}$ under industrial conditions.

Results

Primary and transfer pressure standards for a consistent dissemination of the pressure scale in the intermediate range (1 to 10^4) Pa

Design of a new interferometric liquid column micromanometer as a primary standard for 2 kPa range of absolute and gauge pressure was developed, test floats manufactured and a differential interferometer produced. The analysis of oils suitable as manometric liquids was performed, and the density, compressibility and thermal expansion coefficients of seven selected oils – candidate liquids for the micromanometer were measured. Three oils were identified as candidate liquids for the micromanometer, and their capillary properties, gas absorption/desorption dynamics and density dependence on gas saturation were determined. Performance tests of the twin-interferometer system were carried out which demonstrated stability of the interferometric measurement on the level of 30 nm over 12 hours equivalent to a pressure stability of better than 0.25 mPa.

Further to this, two methods for centring the piston in the cylinder of the Furness Rosenberg Standard (FRS) piston gauges were developed and tested which improve the reproducibility of pressure measurements and create preconditions for a characterisation of these piston gauges as primary pressure standards by dimensional measurement. Subsequently, reproducibility of the effective area after assembly was improved by a factor of more than 2. Dimensional measurements on four Force-balanced Piston Gauges (FPG) and two FRS were performed. Gas flow models based on the Dadson theory (viscous flow) and Boltzmann equation and reliable kinetic model equations such as the Bhatnagar–Gross–Krook (BGK) approach and the discrete velocity method (DVM) were adopted and software was developed, validated and applied to calculate the pressure distribution in the piston-cylinder as well as to determine the effective area of two FRS and four FPG piston gauges when operated in absolute and gauge mode with a standard uncertainty lower than 9 ppm. FPG and FRS piston gauges were calibrated in the absolute and gauge mode at pressures above 1 kPa against national primary pressure standards such as pressure balances and mercury manometers and, in the low-pressure operation range, against vacuum standards such as static expansion systems. In addition, the influence of the lubrication pressure on the initial conditions of the FPG was studied. Results of the theoretical and experimental studies were compared and are in reasonable agreement.

A dual Fabry-Perot cavity (DFPC) to measure gas densities by measuring the refractive index was designed and investigated. The system was found to be best suited for short measurement times. It provides the basis for an alternative pressure standard in the range from $(1 \text{ to } 10^4)$ Pa. A new methodology, based on a drift-free mode-of-operation of DFPC was developed, and promising experimental results were achieved showing how well this technique can be used to assess pressure. The progress was made as part of the development of a calibration bench to be used as a transfer standard in the range for 1 Pa to 13 kPa. The newly built and characterised calibration bench is currently being used for the characterisation of different gauges. A special capacitance diaphragm gauge (CDG) was developed, in which the heater can be switched off and can be temperature-controlled up to 80°C. This CDG was characterised in the temperature range between 30 °C and 55 °C. Also, 10 mTorr CDGs were investigated at different environmental conditions and found to be suitable for providing traceability to industrial vacuum sensors below 10 Pa.

Calibration methods for positive and negative gauge pressure standards in the range from approximately $(-10^5 \text{ to } 10^4)$ Pa

Two techniques for weather-independent calibration of low gauge pressure were developed. The first technique involves a hermetic chamber in which the standard and instrument to be calibrated can be enclosed and different parameters such as atmospheric pressure, temperature and humidity can be controlled and maintained. The second technique uses the control of the atmospheric pressure in reference ports of the instruments involved. For this, a variable volume chamber either open to the atmosphere or controlled by a pressure controller was used as a source of ambient reference pressure. With the first technique, the hermetic-

chamber technique, the standard deviations of pressure gauge indications, which at unstable ambient conditions were as high as 0.15 Pa, could be reduced to 0.002 Pa. With the second weather-independent calibration technique, the standard deviation of readings at unstable ambient conditions could be reduced by factor 2. Further to this, a new sensor for differential 100 Torr CDG and associated electronics were designed and tested in different environmental conditions.

Three methods for calibrating negative gauge pressure were developed, which use different configurations of absolute and gauge pressure balances and barometers. The three methods include (i) measuring the negative pressure in the bell jar of the balance, (ii) using a gauge pressure balance in an upside down (“hanging piston”) orientation, and (iii) the application of two absolute pressure measuring instruments, one of which is an absolute pressure balance for sub-atmospheric pressure and the other is a barometer controlling a nearly atmospheric pressure in a reference chamber. Performance of these three methods was evaluated and their advantages when compared to each other analysed. The results showed that method (i) is applicable for calibration and for indicating negative gauge pressure instruments, but can have limitations for the pressure range and stability of measurement dependent on the local ambient pressure. It can also be easily automated. Method (ii) can be applied to the calibration of negative gauge pressure piston gauges, for which methods (i) and (iii) are less suitable. This method is simple in realisation, and its equipment is relatively cheap, but similar to method (i) the measurement range for method (ii) can be limited by the local atmospheric pressure. Method (iii) is the most expensive in realisation, and it cannot be used for calibration of negative gauge pressure piston gauges, only for indicating instruments. Its advantage lies in the method’s independency from the ambient atmospheric pressure. Therefore, a calibration can be performed at unstable atmospheric conditions and over the full negative pressure range down to -100 kPa even for laboratories having significant elevations over the sea level, where a full range calibration with other methods is not possible. Thus, depending on the instruments to be calibrated and conditions of the laboratory, a recommended best method can be suggested from either method (i), (ii) or (iii). An automated set-up for the calibration of pressure instruments in the range (-10⁵ to 10⁴) Pa was developed. Using this set-up, calibrations of pressure instruments for secondary laboratories with a target uncertainty better than $3 \cdot 10^{-5} \times p + 1$ Pa became possible thanks to the elimination of the atmospheric pressure's contribution from the measurement uncertainty. Calibrations of pressure balances in positive and negative gauge pressure mode with a mercury manometer as a reference were performed and showed similar uncertainties of $1.3 \cdot 10^{-5} \times p + 0.17$ Pa in both operation modes. The capability of FPG for measuring negative pressure was tested too.

EU strategy on restrictions of mercury use in measuring devices (barometers, sphygmomanometers)

Results of barometric interlaboratory pressure comparisons were analysed and measurements of pressure balances and FPGs in comparison with mercury manometers were performed. In addition, high precision pressure gauges were compared against dead-weight pressure balances, FPGs and mercury manometers. The analysis showed that the mercury manometers performed better than digital piston gauges. However, with dead-weight pressure balances, comparable or even lower uncertainties than those of mercury manometers could be achieved.

Calibration service in the range of approximately (-10⁵ to 10⁴) Pa of gauge pressure and approximately (1 to 10⁴) Pa of absolute pressure

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 (-10⁴ to 10⁴) Pa of gauge pressure and approximately (1 to 10⁴) Pa of absolute pressure). The calibration methods and techniques are now used to provide the calibration service for negative gauge pressures in the range (-10⁵ to 0) Pa. Engagement with industries that utilise pressure in the intermediate range (1 to 10⁴) 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.

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 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 μ Flu18 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 10⁴) 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 $3 \cdot 10^{-5} p + 1$ 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

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 should 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 should be increased, leading to higher employment and wealth for society.

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 benefits 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 which guarantee a strong value of safety, protect the industrial infrastructure and mainly beware the environment from fatal situations.

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.

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, will be more efficient, less cost intensive and avoid the production of unwanted by-products.

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 these 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 will benefit from the enhanced measurement capabilities at the NMI level and the dissemination of the pressure scale to calibration laboratories.

As the European mercury strategy restricted the use of mercury in barometers from 10 April 2014 which was an issue for research institutions and reference laboratories in the avionic industry, weather monitoring and forecast services, a new primary standard, using alternative manometric liquids such as oil, fulfils the EU demands and reduce the risk of accidental environmental pollution by mercury.

List of publications

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1 PTB, Germany	10 CUNI, Czech Republic	14 INFICON LI, Liechtenstein
2 CEM, Spain	11 FCT-UNL, Portugal	15 INRIM, Italy
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4 CNAM, France	13 UTH, Greece	
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