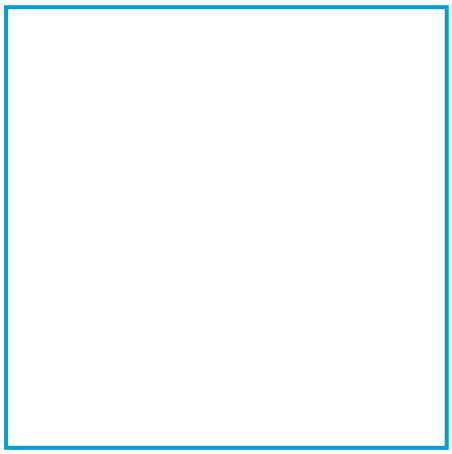
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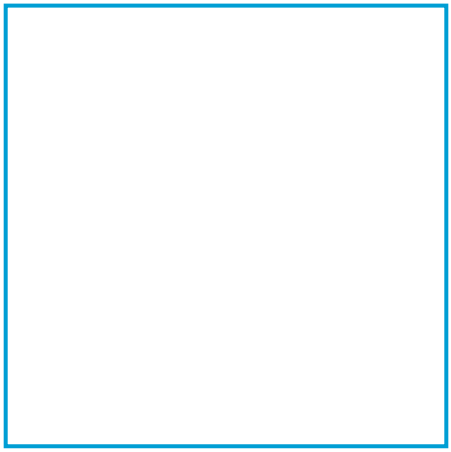
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April 2019

Wladimir Sabuga (Hrsg.)

Industrial Standards in the Intermediate Pressure-to-Vacuum Range







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Wladimir Sabuga (Hrsg.)

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Proceedings of the final pres2vac workshop held at
RISE, Borås, Sweden, May 16–17, 2018

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Physikalisch-Technische Bundesanstalt
Braunschweig und Berlin

Proceedings

of the Final Workshop

"Industrial Standards in the Intermediate Pressure-to-Vacuum Range"

held at RISE, Borås, Sweden
May 16th–17th, 2018

EMPIR
EURAMET
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

pres2vac
14IND06



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INTRODUCTION

This PTB report comprises the presentations held at the Final Workshop on Industrial Standards in the Intermediate Pressure-to-Vacuum Range, which took place in RISE, Borås, Sweden on 16th – 17th May 2018.

The workshop was an output of the joint research project (JRP) entitled “Industrial Standards in the Intermediate Pressure-to-Vacuum Range” carried out within the European Metrology Programme for Innovation and Research (EMPIR), jointly funded by the EMPIR participating countries within EURAMET and the European Union and registered as EMPIR JRP 14IND06 pres2vac. In the project, 10 national metrology institutes (NMIs) collaborate with 4 universities and 2 industrial companies.

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.

SI traceable measurement of absolute, positive and negative gauge pressure are relevant for such industries 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. Conventional calibration procedures applied to instruments for low differential pressures are extremely dependent on weather conditions, especially the stability of atmospheric pressure. Alternative calibration approaches and techniques are needed to ensure a low level of uncertainty, independent of ambient conditions, 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. With the project, support is provided for the replacement of primary mercury manometers, which are still in use in many research institutions and reference laboratories, by mercury-free instruments and techniques.

More than 35 representatives of industry (both manufacturers and users), academic and national metrology institutes from 16 countries were invited and participated. During the workshop, 22 talks on key topics, each of 5 to 35 minutes' duration, were held. These oral presentations are published in the present report.

I would like to thank all contributors for their efforts to organize this successful workshop at RISE and to publish these proceedings.

Braunschweig, February 2019, *Wladimir Sabuga (PTB)*

PROGRAMME
of the Final Workshop
on Industrial Standards in the Intermediate Pressure-to-Vacuum Range
16th – 17th of May 2018

Wednesday, 16th of May

09:30 Registration and coffee

10:00 Welcome and introduction
Martin Zelan – RISE, Sweden, *Wladimir Sabuga* – PTB, Germany

10:30 Overview and perspectives on the development of oil-based interferometric micromanometers
Jens Könemann, Sven Ehlers, Wladimir Sabuga – PTB, Germany

10:40 Design of an interferometric liquid column manometer with in situ density measurement
Sven Ehlers, Wladimir Sabuga, Jens Könemann – PTB, Germany

11:00 Vacuum liquids for use in liquid column manometers
Sven Ehlers, Jens Könemann, Wladimir Sabuga – PTB, Germany

11:20 Measurement of diffusion and solubility of atmospheric gases in candidate oils for oil manometer
Janez Šetina – IMT, Slovenia

11:40 Determination of physical properties of candidate fluids by IPQ
Isabel Spohr, Andreia Furtado, Jorge Pereira – IPQ, Portugal

12:00 Discussion

12:30 Lunch at RISE restaurant

13:30 Digital pressure balances with non-rotating piston - FRS and FPG
Dominik Pražák, Zdeněk Krajíček, Martin Vičar – CMI, Czech Republic

14:05 Dimensional measurements and modelling of FPG and FRS at PTB
Ahmed Hashad – NIS, Egypt, *Ahmed Hashad, Wladimir Sabuga* – PTB, Germany

14:30 Gas flow modelling and calculation of the effective area of FPG and FRS
Nikolaos Vasileiadis, Steryios Naris, Dimitris Valougeorgis – UTH, Greece, *Ahmed Hashad* – NIS, Egypt, *Ahmed Hashad, Sven Ehlers, Wladimir Sabuga* – PTB, Germany

15:00 Coffee

15:15 Experimental study of PTB FPG as secondary pressure standard
Wladimir Sabuga, Ahmed Hashad, Sven Ehlers – PTB, Germany, *Ahmed Hashad* – NIS, Egypt

15:30 Effect of lubrication pressure in FPG
David Herránz, Virginia Marcos, Salustiano Ruiz, Nieves Medina – CEM, Spain

15:45 Calibration techniques- Introduction
Mark Plimmer – CNAM, France*, Djilali Bentouati* – LNE, France

15:50 Methods and instruments for accurate, weather-independent calibration of low gauge pressure instruments
Zdeněk Krajíček, Martin Vičar, Dominik Pražák, – CMI, Czech Republic, *Wladimir Sabuga, Thomas Konczak, Sven Ehlers, Ahmed Hashad* – PTB, Germany, *Ahmed Hashad* – NIS, Egypt

16:20 Methods for the calibration of Force Balance Piston Gauge FPG
Djilali Bentouati – LNE, France, *Mark Plimmer* – CNAM, France

16:40 Discussion

17:00 Tour to the RISE pressure and vacuum laboratory

18:00 Transport to hotels

19:00 Dinner at "Gamla matsalen" Textile Fashion Centre.

Thursday, 17th of May

08:40 Transport from hotels to RISE

09:00 Measurement of positive and negative gauge pressures using an absolute pressure balance
Mark Plimmer – CNAM, France*, Djilali Bentouati* – LNE, France

09:30 Negative gauge pressure calibration by means of a mercury manometer and conventional gauge pressure piston gauges
Sven Ehlers, Thomas Konczak, Wladimir Sabuga – PTB, Germany

09:45 Other methods for negative pressure: FPG, digital manometer, divider
Djilali Bentouati – LNE, France

09:55 Conclusion
Mark Plimmer – CNAM, France

10:00 Coffee

10:30 Advances in pressure measurement using refractometry
Martin Zelan, Thomas Hausmaninger – RISE, Sweden, *Isak Silander, Ove Axner, Thomas Hausmaninger* – UmU, Sweden

11:10 Evaluation of a low absolute pressure transfer standard in the range 1 Pa to 10 kPa
Frédéric Boineau – LNE, France

11:40 Investigation of the use of pressure balances as an alternative to mercury manometers
Virginia Marcos, David Herránz, Salustiano Ruiz, Nieves Medina – CEM, Spain

12:00 Stability study of low-range CDGs
Martin Vičar – CMI, Czech Republic, *Janez Šetina* – IMT, Slovenia

12:10 Transfer standard package for high accuracy pressure transducers with improved temperature stability
Rifat Kangi – UME, Turkey

12:30 Lunch at RISE restaurant

13:15 Summary and final discussions
Martin Zelan – RISE, Sweden

14:00 End of workshop

CONTENTS

W. Sabuga
Introduction – EMPIR JRP 14IND06 pres2vac "Industrial standards in the intermediate pressure-to-vacuum range" **13**

J. Könemann, S. Ehlers, W. Sabuga
Overview and perspectives on the development of an oil-based interferometric micromanometer at PTB **25**

S. Ehlers, W. Sabuga, J. Könemann
Design of an interferometric liquid column manometer with in situ density measurement **31**

S. Ehlers, J. Könemann, W. Sabuga
Vacuum liquids for use in liquid column manometers **49**

J. Šetina
Measurement of diffusion and solubility of atmospheric gases in candidate oils for oil manometer **63**

I. Spohr, A. Furtado, J. Pereira
Industrial standards in the intermediate pressure-to-vacuum range – Determination of physical properties of candidate fluids by IPQ **77**

D. Pražák, Z. Krajíček, M. Vičar
Digital pressure balances with non-rotating piston – FRS and FPG **85**

A. Hashad, W. Sabuga **101**
Dimensional measurements and modelling of FPG and FRS at PTB

N. Vasileiadis, S. Naris, D. Valougeorgis, A. Hashad, S. Ehlers, W. Sabuga
Gas flow modelling and calculation of the effective area of force-balanced piston gauges FPG and FRS **117**

W. Sabuga, A. Hashad, S. Ehlers
Experimental study of PTB FPG as secondary pressure standard **135**

D. Herránz, V. Marcos, S. Ruiz, N. Medina
Effect of lubrication pressure in FPG **151**

M. Plimmer, D. Bentouati
Calibration techniques for the intermediate pressure-to-vacuum range – Introduction **159**

Z. Krajíček, M. Vičar, D. Pražák
Weather-independent low pressure measurements - I **165**

W. Sabuga, T. Konczak, S. Ehlers, A. Hashad
Weather-independent low pressure measurements - II **177**

D. Bentouati, M. Plimmer
Traceable methods for calibration of Force balanced Piston Gauges (FPGs) in absolute and gauge modes **187**

M. Plimmer, D. Bentouati
Measurement of positive and negative gauge pressures using an absolute pressure balance **201**

S. Ehlers, T. Konczak, W. Sabuga
Negative gauge pressure calibration by means of a mercury manometer and conventional gauge pressure piston gauges **213**

D. Bentouati
Calibration of positive and negative gauge pressure – Two examples **229**

M. Plimmer
Calibration techniques for the intermediate pressure-to-vacuum range – Conclusion **239**

M. Zelan, T. Hausmaninger, I. Silander, O. Axner
Advances in pressure measurement using refractometry **245**

F. Boineau
Evaluation of a low absolute pressure transfer standard in the range 1 Pa to 10 kPa **269**

V. Marcos, D. Herránz, S. Ruiz, N. Medina
Investigation of the use of pressure balances as an alternative to mercury manometers **287**

M. Vičar, J. Šetina
Stability study of the low-range CDGs **301**

R. Kangi
Temperature controlled transfer standard package for high accuracy pressure transducers with improved temperature stability **311**

Introduction – EMPIR JRP 14IND06 pres2vac "Industrial standards in the intermediate pressure-to-vacuum range"

Wladimir Sabuga

Physikalisch-Technische Bundesanstalt (PTB), Germany

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

EMPIR

EURAMET

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

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EMPIR 14IND06 "pres2vac"
Call-2014 "Industry"

- Mass and related quantities metrology
- Dimensional metrology
- Flow metrology

Industrial standards in the intermediate pressure-to-vacuum range

Wladimir Sabuga

Consortium and collaborators

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PTB, Germany
CMI, Czech Republic
CEM, Spain
LNE, France
CNAM, France
TUBITAK, Turkey
IMT, Slovenia
RISE, Sweden
IPQ, Portugal
UTH, Greece
UmU, Sweden
FCT/UNL, Portugal
INRIM, Italy
INFICON AG, Liechtenstein
TRESCAL, UK

AIRFRANCE
Snecma SAFRAN Group
IBERIA
AKS MESSTECHNIK GMBH
ENAC Entidad Nacional de Acreditación
ZMK Group
WIKA
delong instruments
VAKUUM PRAHA
europascal
Endress+Hauser
Minerva METROLOGY CENTER

Industrial needs

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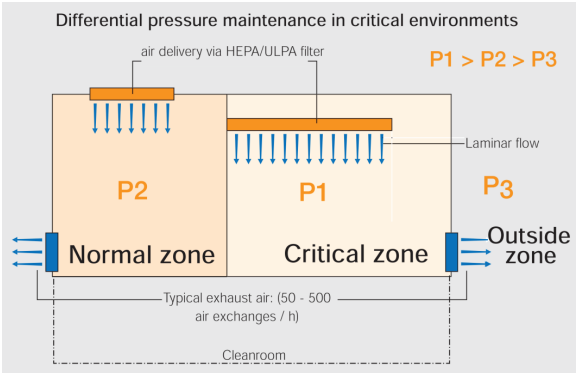
Low absolute, differential, positive and negative gauge pressure measurements play vital role in numerous industrial processes.



Pharmaceutical industry operates in environment with strict requirements on safety, sterility and precision.

Cleanroom requirements:
- 100 to 1000 Pa in the makeup and re-circulation units,
- 50 to 500 Pa drops through HEPA and pre-filters,
- 7 to 12 Pa overpressure in the cleanroom,
- 5 Pa between adjacent rooms.

Energy: 1 Pa in a medium size cleanroom requires 3000 kWh additional energy per annum!



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**,
- **research laboratories**

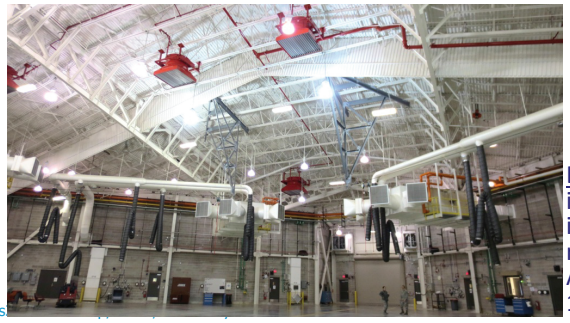
pressure difference (negative pressure) prevents spread of germs, contaminants and dust.

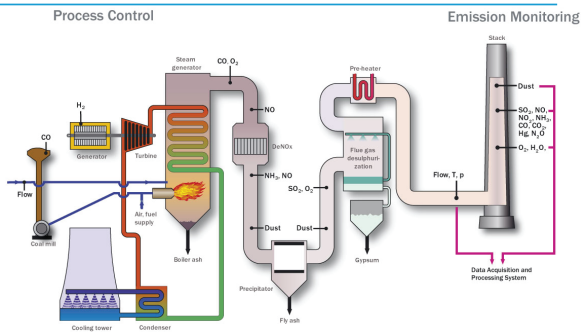
Industrial needs

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Chemical and petrochemical industries subject to strict international requirements like PED, ATEX [Directives 97/23/EC, 94/9/EC]. To meet the safety and performance requirements, reliable, accurate, traceable pressure measurements must be provided to these industries.





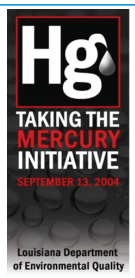
In **power plants**, for safety, efficiency and cost savings: avoiding overpressure situations, preventing release of harmful process into plant or environment. Through differential pressure measurement, flow and level are measured. Absolute, gauge and differential pressures are measured over a wide range from 0 to about 20 MPa.

Low positive and negative pressure measurements 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].

Industrial needs

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Mercury Manometer Investigation and Remediation:

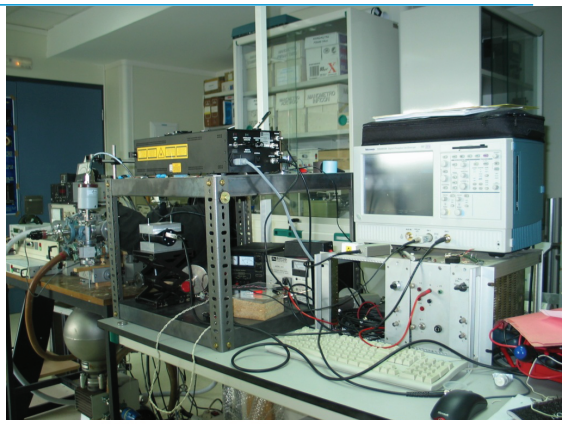
A COOPERATIVE EFFORT WITH THE NATURAL GAS INDUSTRY

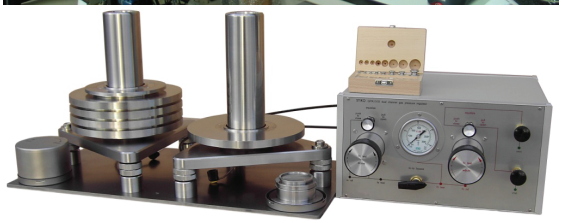
Bill Schramm

EU and global plan to **reduce mercury emissions**: ban of mercury exports, restriction of mercury use in industrial and professional barometers and sphygmomanometers from 10 April 2014 (Commission Regulations (EU) No 847/2012 and 19/9/2012). It presents problem for **research institutions** and **reference laboratories** in the **aviation industry**, **weather monitoring** and **forecast services** where Hg manometers are still widely used as reference pressure standards.



Intensifying **airspace** use by aircrafts → reduction of standard vertical separation. Improved altimeters and autopilots measuring barometric pressure with U<15 Pa, particularly below 30 kPa.



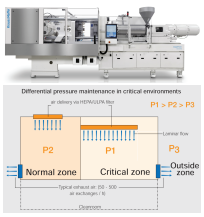


Demands of end-users in Europe are covered by **calibrating laboratories**. Advanced instrumentation became available recently. Target uncertainty of NMIs standards reduces to $3 \cdot 10^{-5} p + 5$ mPa in the range (~1 to ~10^4) Pa.

Workpackages

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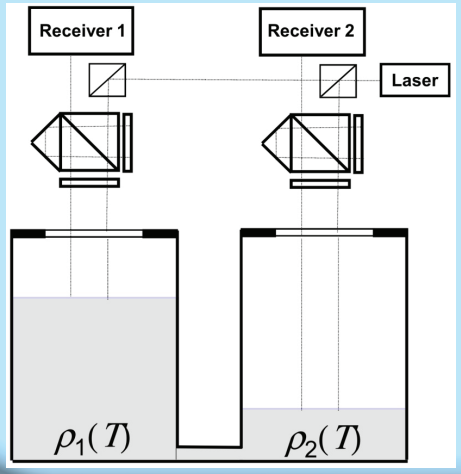
1. Development of an oil-based interferometric micromanometer as a primary absolute and gauge pressure standard (***PTB***, *TUBITAK, IPQ, IMT*)
2. Development of FPG and FRS piston gauges as primary and secondary absolute and gauge pressure standards (***CMI***, *PTB, RISE, INRIM, UTH, CUNI, TUBITAK, CEM, LNE*)
3. Development of calibration techniques in the intermediate pressure-to-vacuum range (***LNE***, *CEM, PTB, CMI, INFICON LI, CNAM, TUBITAK, RISE*)
4. Alternative systems for pressure measurement using optical methods in the range 1 to 10^4 Pa for absolute, positive and negative pressure (***CEM***, *RISE, UmU, PTB, LNE, INFICON LI, CNAM, TUBITAK, IMT, FCT-UNL, CMI*)

WP1

Primary oil micromanometer

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- Thermostated interferometric system - ongoing
- Oils analysis - √
- Density, viscosity, ab- /desorption kinetics - √
- *In-situ* density measurement - ongoing
- Experimental characterisation - ongoing

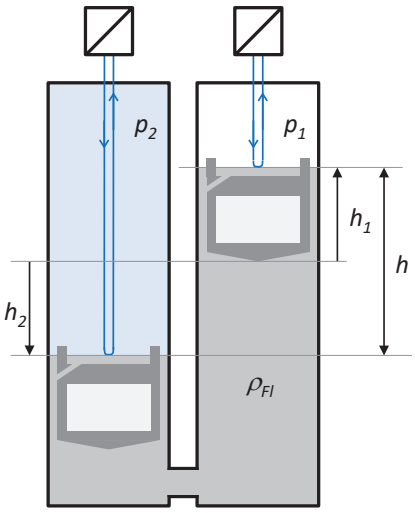




Primary oil micromanometer

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$$p_2 = \rho_{Fl} \cdot g \cdot h + p_1$$



Target: **1 Pa ≤ p ≤ 2 kPa, $U(p)$ = 1 mPa + 2·10^{-5} · p**

Liquid density ρ_{Fl}:

Vacuum oils appear suitable:

+ High sensitivity of the length measurement due to low density (100 µm ≙ 1 Pa)
+ Low vapour pressure (<< 1 mPa)
– Gas absorption
– therm. expansion coefficient
– Long-term stability

} Density change ~ 100 ppm

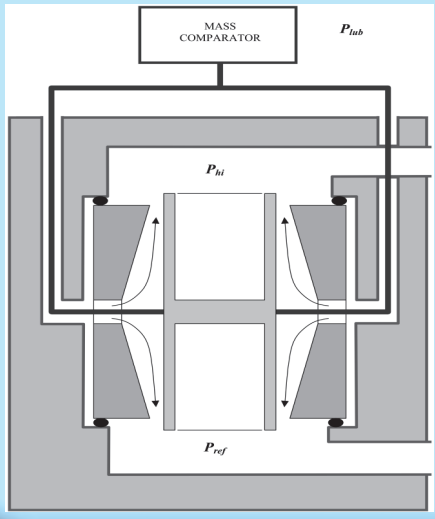
→ **Necessity of integrated density measurement**

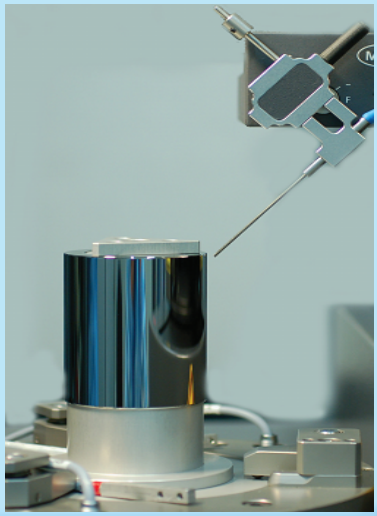
WP2

Force-balanced piston gauges

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- Piston adjustment techniques - √
- Dimensional measurements - √
- Gas flow modelling - √
- Experimental study - √

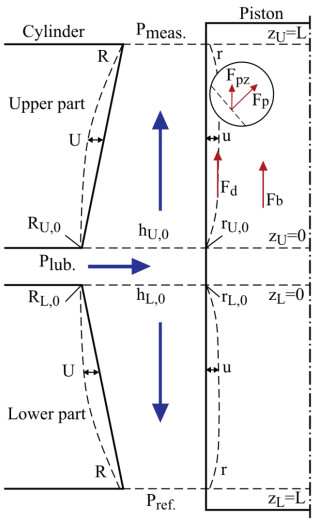




Force-balanced piston gauges

Gas flow modelling

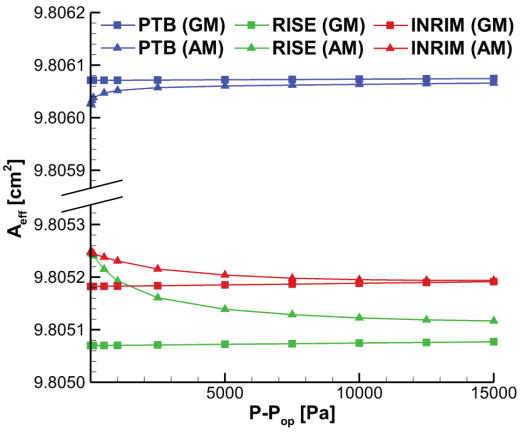
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Total effective area:

$$A_{eff} = \frac{F_L - F_U - F_0}{P_{meas} - P_{ref}}$$

$$A_{eff} = \frac{P_{meas}A_U - P_{ref}A_{U,0}}{P_{meas} - P_{ref}} - \frac{P_{lub}(A_U - A_{U,0})}{P_{meas} - P_{ref}}$$



Operation mode	Gauge			Absolute		
PCA	PTB	RISE	INRIM	PTB	RISE	INRIM
$u_d(A_0)/A_0$ [ppm]	1.0	8.0	1.0	1.0	8.0	1.0
$u_a(A_0(a = 0.9))/A_0$ [ppm]	-0.1	-0.5	-0.4	-0.4	-1.2	-1.1
$u_m(A_0)/A_0$ [ppm]	0.03	0.1	0.1	0.05	0.2	0.2
$u_t(A_0(T = 18\ ^\circ C))/A_0$ [ppm]	0.01	0.03	0.02	0.03	0.1	0.1
$u_t(A_0(T = 22\ ^\circ C))/A_0$ [ppm]	-0.01	-0.03	-0.02	-0.03	-0.1	-0.1
$u(A_0)/A_0$ [ppm]	1.0	8.0	1.1	1.0	8.1	1.6

Uncertainties of all FPGs at maximum pressure →

Force-balanced piston gauges

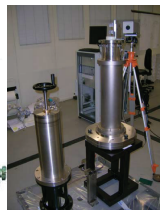
Experimental study

pres2vac
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Using national primary pressure and vacuum standards as reference







1 kPa →

Measurements on FRS in absolute and gauge mode

- Against CMI and PTB pressure balances above 2 kPa
- Against PTB mercury manometer
- Against PTB static expansion system

← 100 Pa

Measurements on FPG in absolute, gauge and differential mode

1 kPa →

- Against LNE APX and PG 7607 pressure balances
- With CDG as transfer standard to pressure balance at UME
- Against INRIM pressure balances
- Against CEM and PTB mercury manometers
- Study of lubrication pressure and stability of gauge pressure measurements at CEM

10 Pa – 5 kPa

- Against CEM static expansion system





← 100 Pa

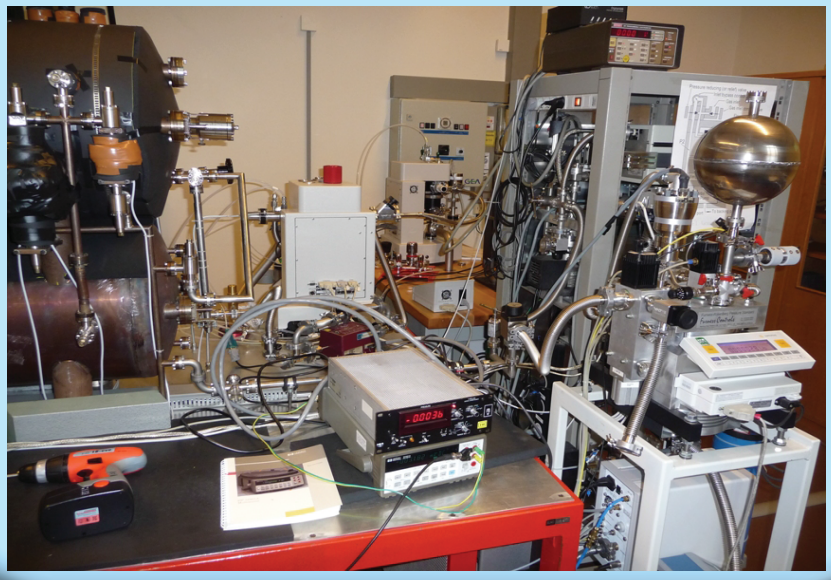
- Against PTB static expansion system
- Against CMI and CUNI continuous flow system
- Against LNE CDG and SRG
- At UME with CDG and SRG as transfer standards to static expansion system

WP3

Calibration techniques

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- Weather-independent calibration - √
- Methods for negative gauge pressure - √

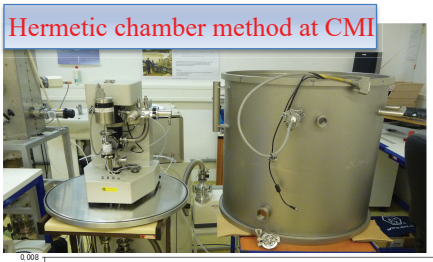


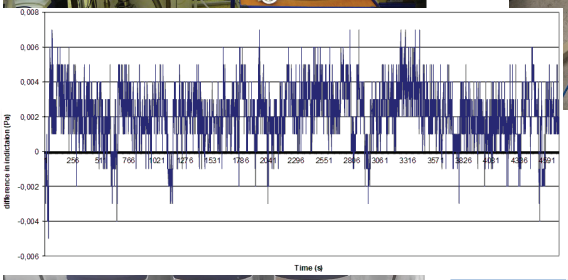
Calibration techniques

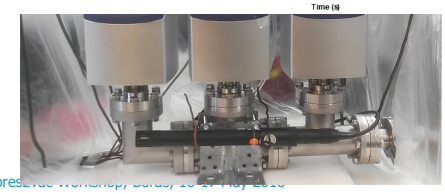
Weather-independent calibration

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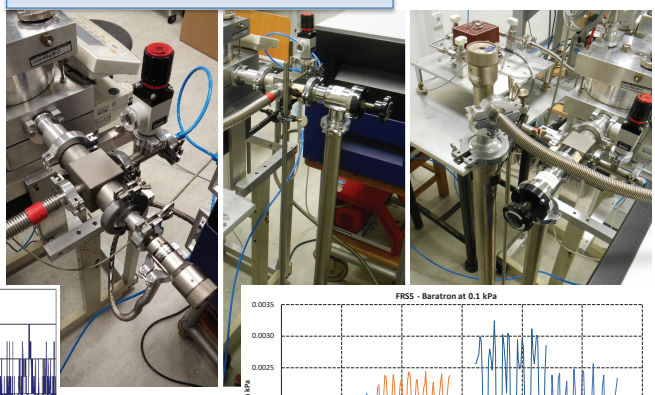
Hermetic chamber method at CMI

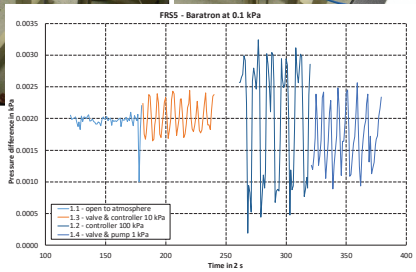






Additional volume method at PTB





Laboratory	*p* / Pa	u(*p*) / Pa	StDev / Pa (no control of ambient pressure)	StDev / Pa (control of 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

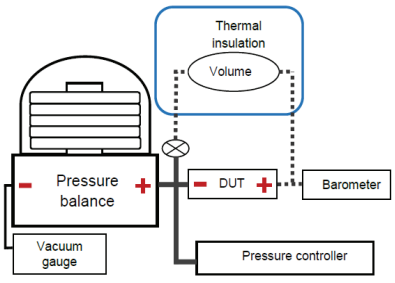
Calibration techniques

Methods for negative gauge pressure

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14IND06

3 methods using pressure balances

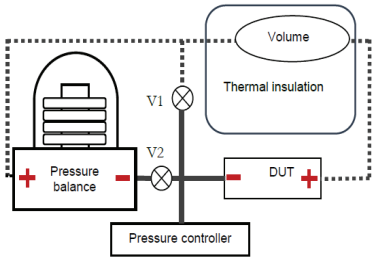
Barometer method



$$P_{neg} = -\frac{\Sigma_i [m_i \cdot g]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})][1 + \lambda \cdot P]} \mu + \rho_f \cdot g \cdot \Delta h - P_{atm}$$

U = 0.20 Pa + 6.6·10^{-6}·IPI

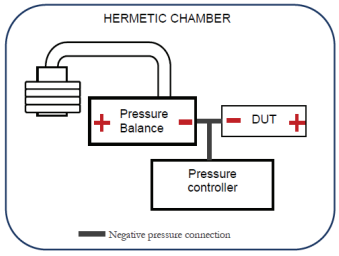
Bell jar method



$$P_{neg} = -\frac{\Sigma_i \left[m_i \left(1 - \frac{\rho_f}{\rho_{m_i}} \right) \cdot g \right]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})][1 + \lambda \cdot P]} + (\rho_f - \rho_a) \cdot g \cdot \Delta h \, .$$

U = 0.10 Pa + 7.0·10^{-6}·IPI

Upside-down method



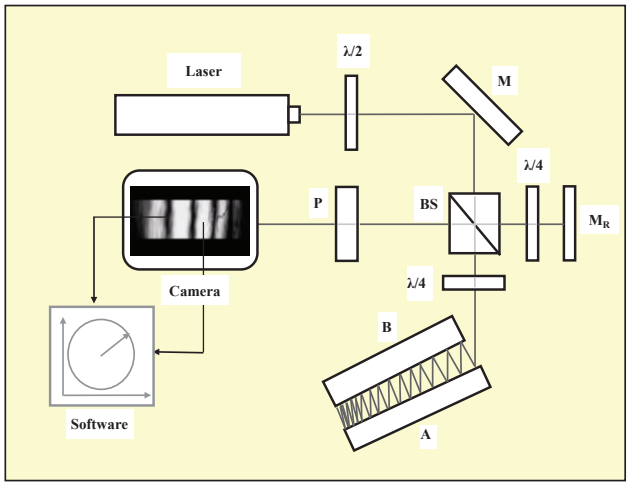
$$P_{neg} = -\frac{\Sigma_i \left[m_i \left(1 - \frac{\rho_a}{\rho_{m_i}} \right) \cdot g \right]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})][1 + \lambda \cdot P]} + (\rho_f - \rho_a) \cdot g \cdot \Delta h \, .$$

U = 0.16 Pa + 1.0·10^{-5}·IPI

WP4 (CEM, 77 PM):
Alternative pressure standards

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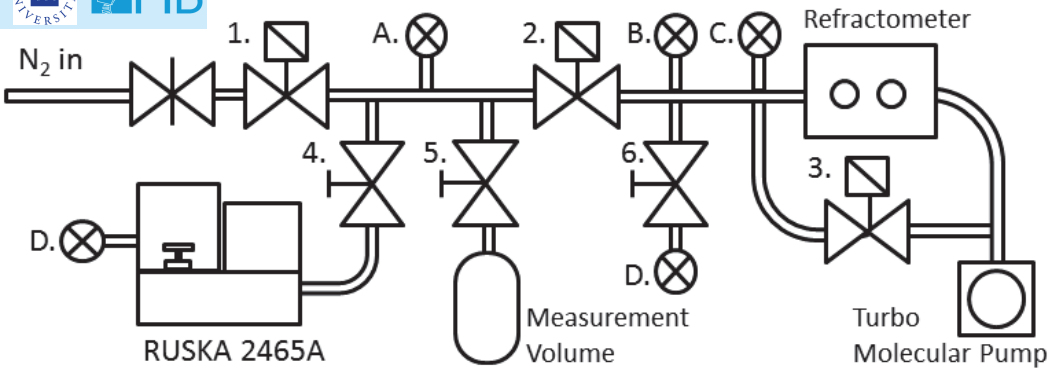
- Optical methods - ongoing
- Transfer pressure standards - ongoing
- Mercury-free barometric standards - ongoing
- Industrial transfer standards below 10 Pa - ongoing



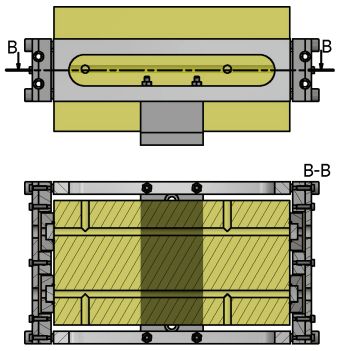


Alternative pressure standards
Optical methods

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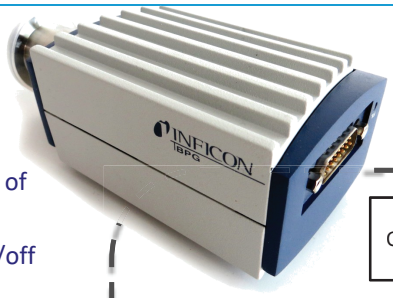




Alternative pressure standards

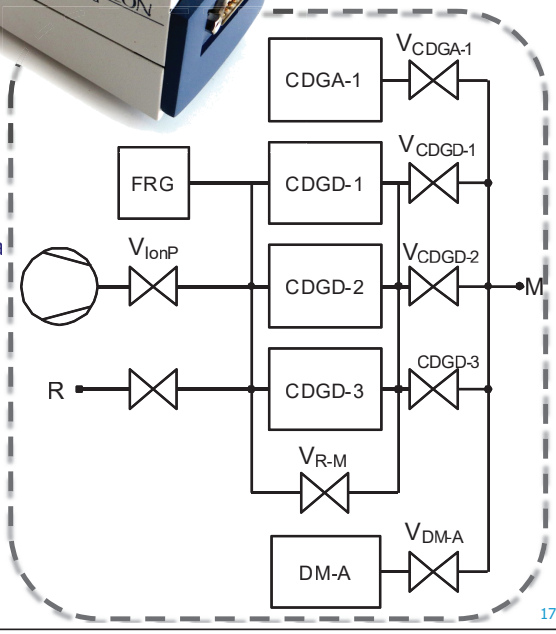
Transfer standards

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Methods and instrumentation

- Transfer standard in the range 1 Pa to 13 kPa of absolute and differential pressure
- Metrology-grade CDGs with optional turn on/off heater by INFICON



Calibration of the transfer standard package against

- UME pressure balance (1.3 kPa to 13 kPa), FPG (10^{-2} Pa to 13 kPa), static expansion system (10^{-3} Pa to 1 kPa)
- IMT pressure balance (2 kPa to 13 kPa) and static expansion system (10^{-2} Pa to 2 kPa)
- CEM FPG (0.01 Pa to 13 kPa), static expansion (10^{-2} Pa to 1 kPa) and dynamic expansion system (10^{-4} to 10^{-2} Pa).
- FCT-UNL 133 Pa and 133 kPa CDGs and SRG (1 to 10 Pa)

Investigation of the thermal transpiration effect

Alternative pressure standards

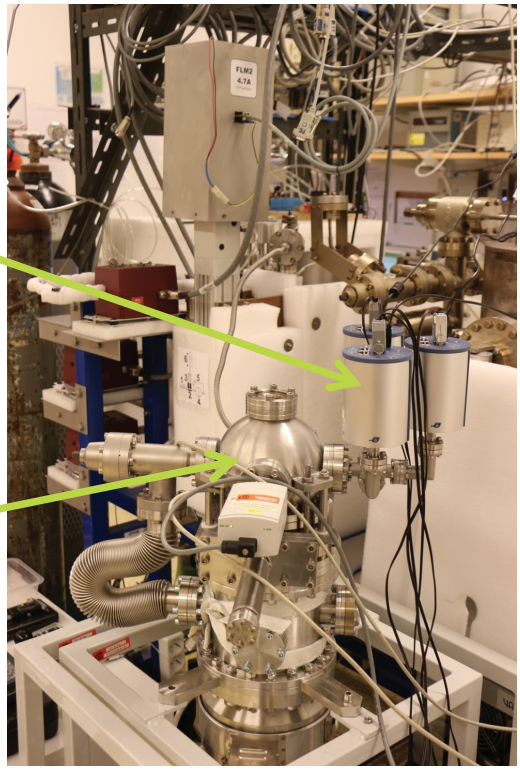
Transfer standards

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INFICON CDGs were used in measurements based on classical and transitional dynamic expansion systems at CMI

INFICON CDGs under study at CMI

Transitional regime dynamic expansion system used for calibration of gauges in the range 0.1 Pa – 1.33 Pa



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EMPIR

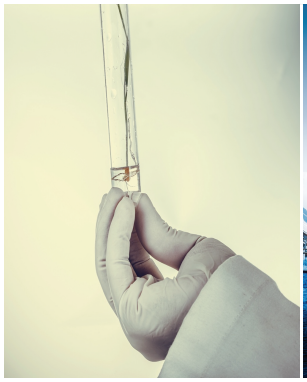
EURAMET

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pres2vac

pres2vac

-A metrology research project within the European Union Horizon 2020 framework.





FINAL WORKSHOP

Thanks to RISE for hosting!





Research Institutes of Sweden

Safety and Transport

Measurement Technology

Overview and perspectives on the development of an oil-based interferometric micromanometer at PTB

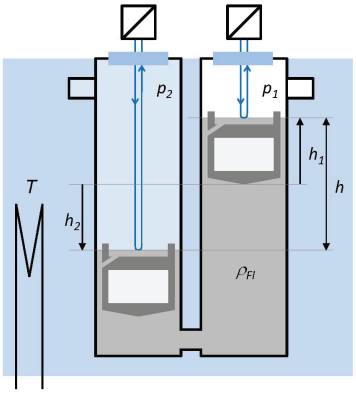
Jens Könemann, Sven Ehlers, Wladimir Sabuga

Physikalisch-Technische Bundesanstalt (PTB), Germany

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Overview and perspectives on the development of an oil-based interferometric micromanometer at PTB

J. Könemann, S. Ehlers, W. Sabuga: *PTB Braunschweig*



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EURAMET

14IND06 pres2vac: Final workshop
RISE Research Institutes of Sweden, Borås
May 16th-17th, 2018

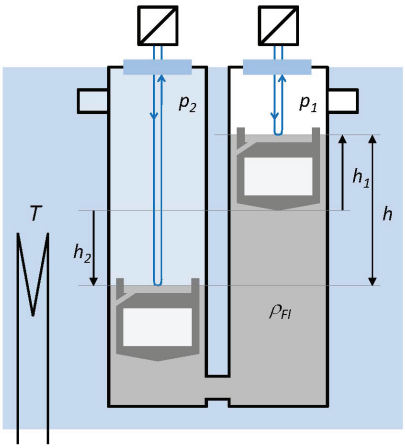
Motivation: Liquid column manometry

- Primary realisation of the pressure unit in the intermediate pressure range
- Relies on fundamental principle
- Should allow calibrations of commercial high-accuracy secondary standards like force-compensated digital piston gauges of FPG and FRS type: challenging accuracy
- Usually, calibrations of these devices done with pressure balances in the higher-pressure range by determining the effective area, in low pressure regime "pressure validation" by comparison with another FPG
- Traditionally primary realizations based on mercury liquid-column manometer-> our approach: oil-based: low vapor pressure, low density, nonhazardeous
- Disadvantage: oil instability, elevated uncertainty of oil density due to saturation effects-> In-situ measurement *of oil density* applied

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Concept



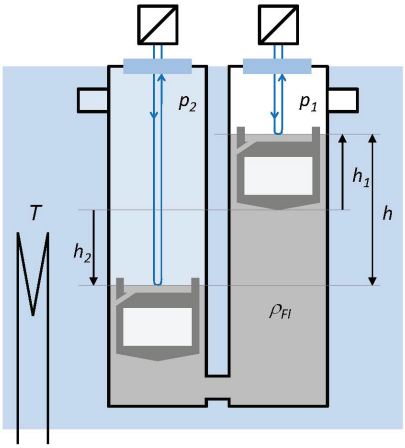
Principle:

- two communicating columns filled with pressure-transmitting liquid (well-known density, low vapor pressure)
- Master equation: $\Delta p = p_2 - p_1 = g \cdot \rho_{Fl} \cdot h$

Basic requirements:

- Pressure regime p_1 p_2
  absolute pressure << 1 mPa 1 Pa – 2 kPa
  gauge pressure p_{amb} 100 kPa – 102 kPa
- Temperature: ≈20 K ± 10 mK
- Interferometer: h_{max}: 2 × 12 cm
  (resolution: 10 nm)

Concept



Principle:

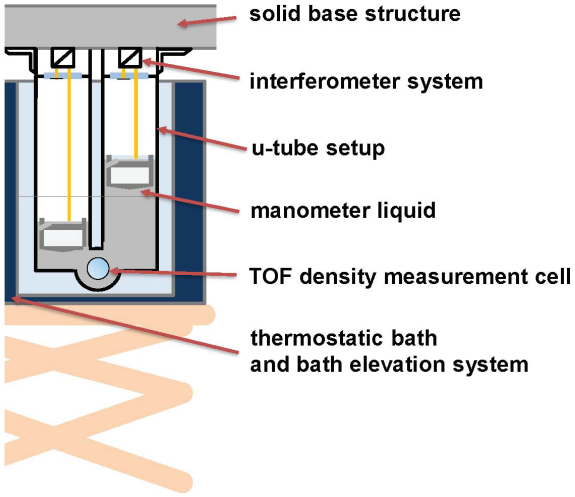
- two communicating columns filled with pressure-transmitting liquid (well-known density, low vapor pressure)
- Master equation: $\Delta p = p_2 - p_1 = g \cdot \rho_{Fl} \cdot h$

Target uncertainty: $1\ \text{mPa} + 20 \cdot 10^{-6}\ p$

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May 16th-17th, 2018

Building blocks of micromanometer



Sven Ehlers/PTB will talk about design and development (task 1.1 of EMPIR project)

- Selection of suitable oils as manometer liquids will be presented in another talk by Sven Ehlers (tasks 1.2 & 1.3)

- Measurements of solubility and diffusion of atmospheric gases in candidate oils will be presented in a talk by Janez Šetina/IMT (activity 1.3.4)

- Andreia Furtado/IPQ will present investigation of density and viscosity of the candidate oils in a following talk (activities 1.3.2 & 1.3.3)

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Summary & outlook

- overall-design of the mechanical parts of the micromanometer is almost finished
- commercial differential interferometer has been selected and purchased
- construction schemes have been created right now in collaboration with engineering-office
- purchase process of all commercial parts and materials for the micromanometer still in progress
- assembling of the mechanical parts takes place in second half of 2018.
- Micromanometer shall provide traceability for calibration service up to 15 kPa with an uncertainty below 20 mPa + 30 ppm

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Design of an interferometric liquid column manometer with in situ density measurement

Sven Ehlers, Wladimir Sabuga, Jens Könemann

Physikalisch-Technische Bundesanstalt (PTB), Germany

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Braunschweig and Berlin
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Sven Ehlers, Wladimir Sabuga, Jens Könemann
PTB Braunschweig, Pressure laboratory, Germany

Final Workshop, EMPIR Research Project “pres2vac”

Borås, Sweden
May 16th-17th, 2018

The task

What do we need?

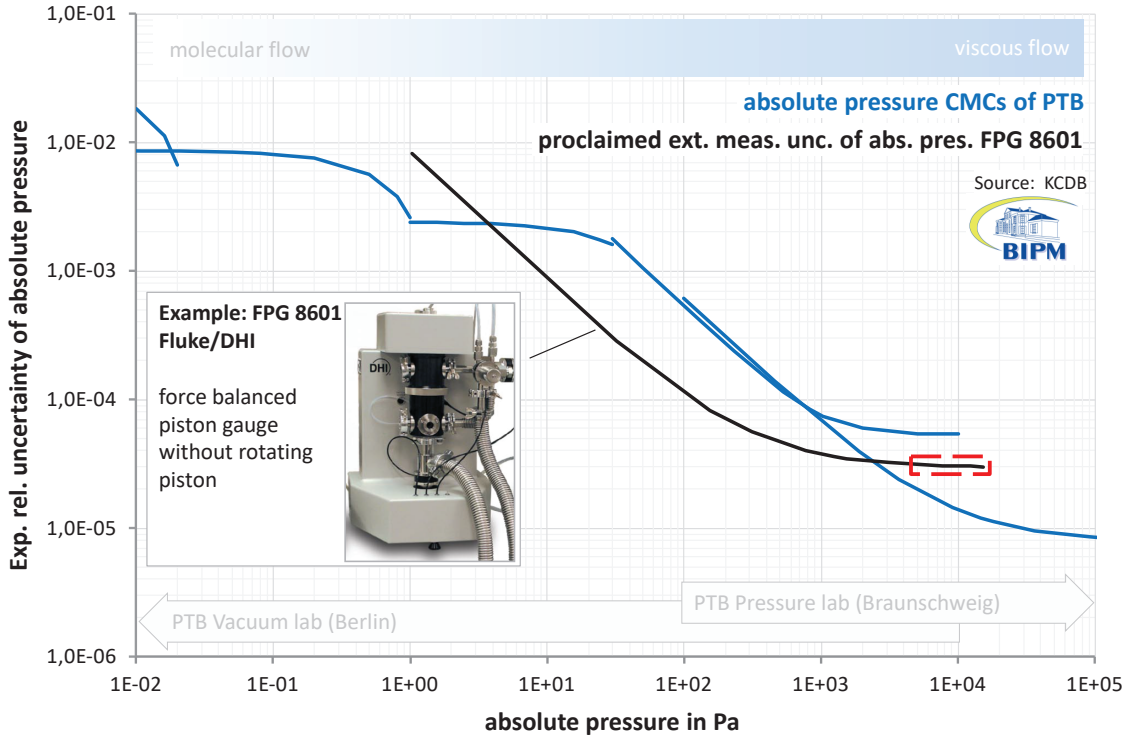
A couple of components

Summary

The task

PTB

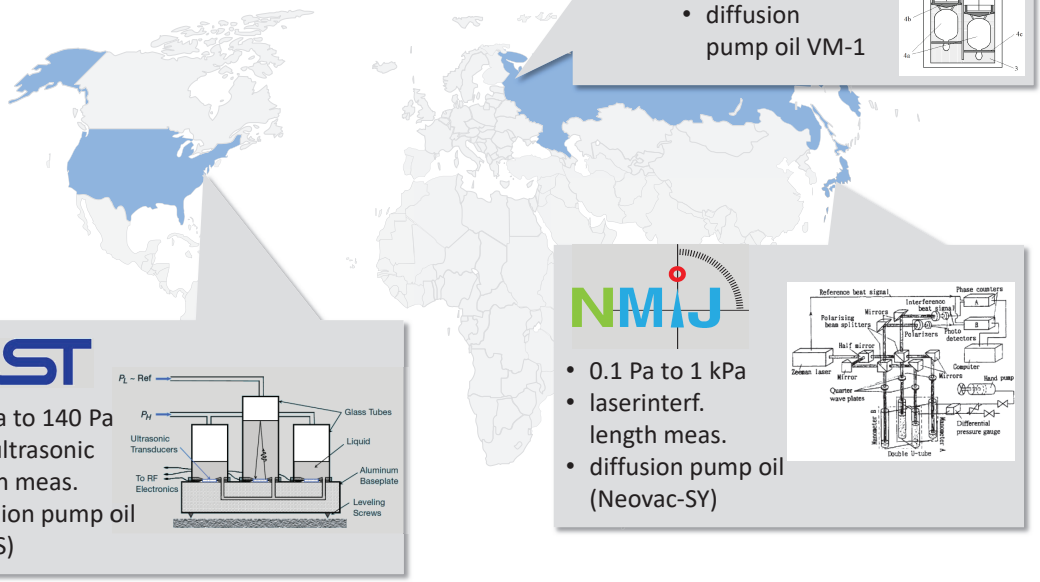
Calibration of a force balanced piston gauges



Sven Ehlers, PTB Pressure Lab

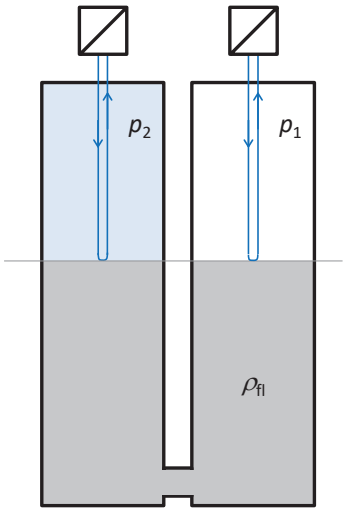
4

Liquid column manometers



Liquid column manometer

$$p_2 = \rho_{fl} \cdot g \cdot h + p_1$$



Gravitational acceleration *g*:
in our lab: $u(g)/g = 10^{-7}$

Length measurement *h*:
- laser interferometer length measurement: resolution 10 nm ≙ 0.1 mPa.
- direct reflection on the liquid surface.

Liquid density ρ_{fl} (vacuum oils):
+ high sensitivity of length measurement, low density (1 Pa ≙ 100 µm)
+ low vapor pressure possible (<< 1 mPa)
– gas absorbing
– thermal expansion
– long-term instability

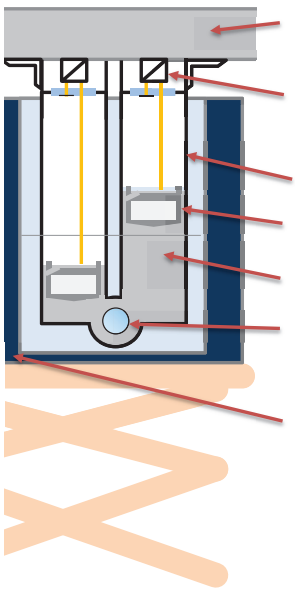
} change in density ~ 100 ppm

→ ***in-situ* density measurement**

What do we need?

PTB

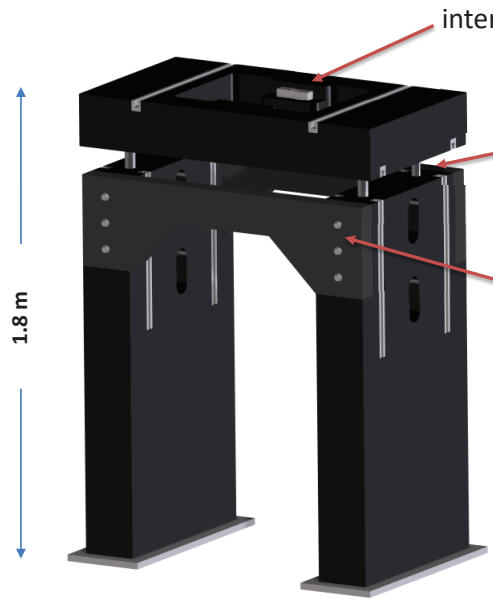
Design concept



Solid base structure

PTB

Solid base structure



Material: Granite
- density: 2900 kg/m³
- thermal expansion: 5-7 µm/(m K)
- elastic modulus: 60-90 kN/mm²

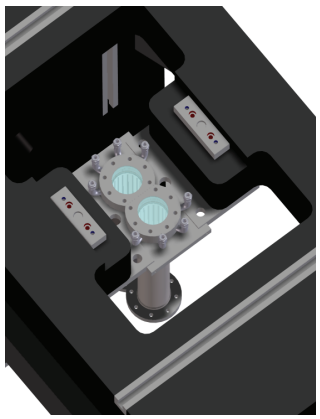
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U-tube body

PTB

U-tube body







U-tube body:
- welded vacuum flanges
- deep drilled main tubes
- oleophobic inner coating

pres2vac

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Sven Ehlers, PTB Pressure Lab

12

Twin differential interferometer

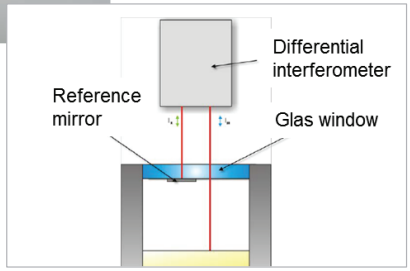
PTB

Twin differential interferometer

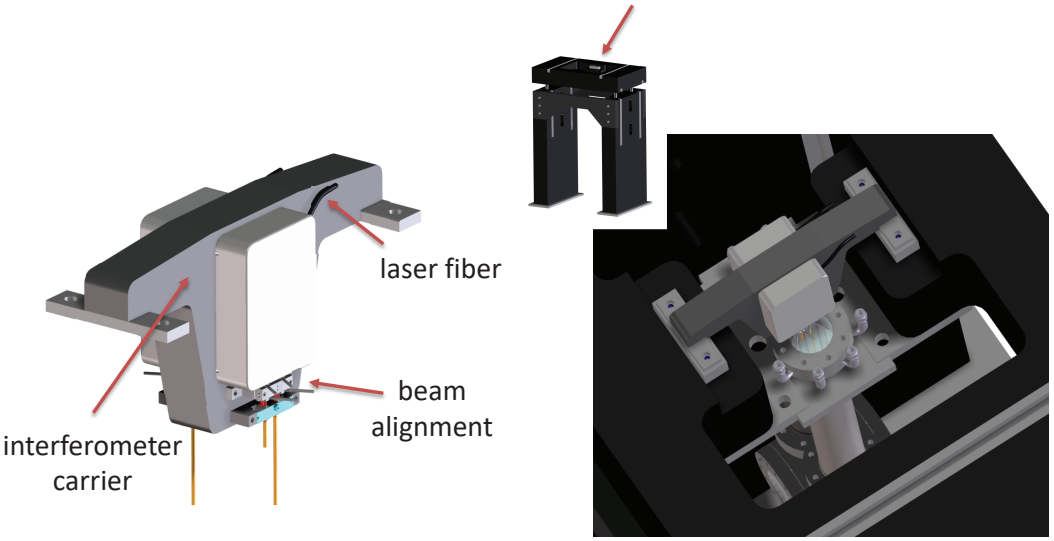


Differential interferometer
- resolution: 1 nm ≙ 0.01 mPa
- fibercoupled He-Ne laser
- no internal electrical components

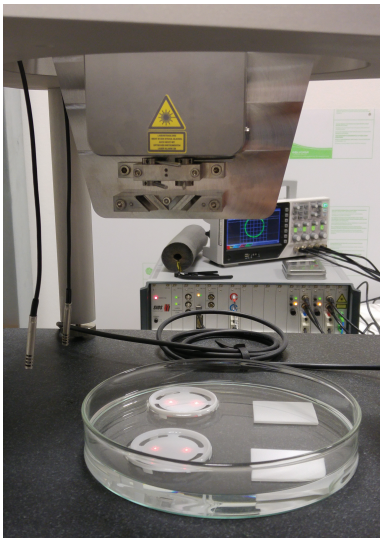


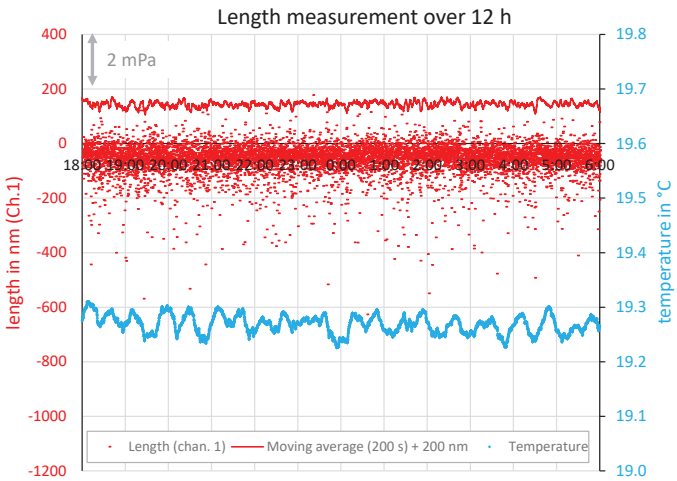


Twin differential interferometer



Twin differential interferometer test setup



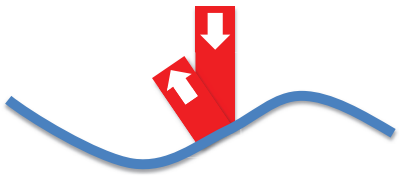


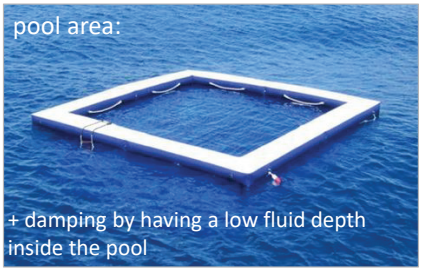
Interferometer characterization on oil surface:
- stable signal over 12 h
- interferometer drift < 30 nm ≙ 0.25 mPa

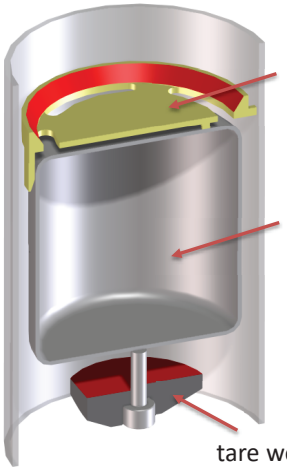
Room temperature stability: ± 0.03 K

Facing surface wafes

possible signal loss:







Manometric liquid

Manometric working liquid

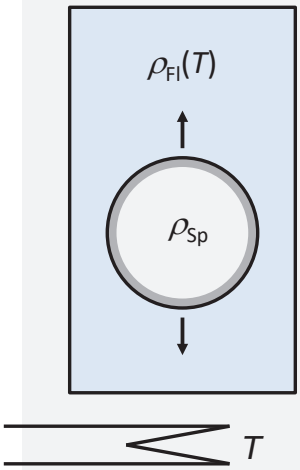
		PTB04	PTB09	PTB10
Vapor pressure				
Potential		4E-08	4E-06	1E-05
	15%	10	8	6
Pressure level found		5E-05	2E-05	1E-05
	15%	4	6	6
Gas solubility				
Change in mass (N2)		205	117	286
Change in mass (dry air)		259	196	425
	10%	6	8	4
Change in mass (moist air)		722	1319	468
	10%	6	2	8
Density change with gas content		-89	-145	-57
	20%	6	4	8
Wetting properties				
		295	347	102
Wall flow	15%	4	4	8
Column oscillation characteristic	5%	6	6	4
Density level				
Density		1095	1178	829
	10%	6	6	8
	100%	6,0	5,4	6,8



– PTB10 was chosen for further developments.
– Long-term stability density measurements are ongoing.

Density standard

Temperature-of-flotation method

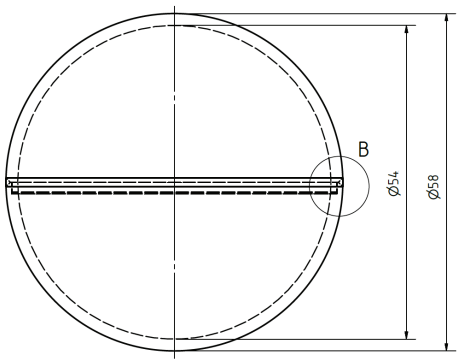


Vary the fluid temperature,
when floating:

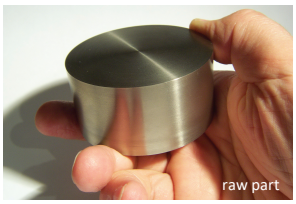
$$\rho_{Fl}(T) = \rho_K$$

- $\Delta T = \pm 0.1$ K
- homogenity = 3 mK
- stability = 3 mK

Density standard



Titan hollow sphere
- diameter: 58 mm
- inner diameter: 54 mm
- volume: 100 cm³



Density standard



Production process:

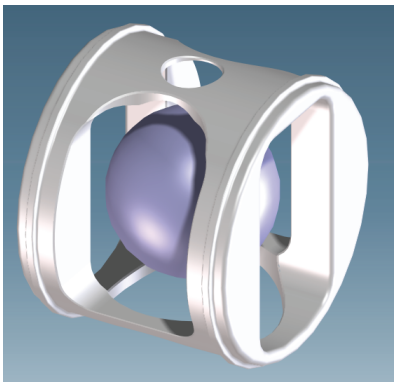
- half shells turning from solid titan
- vacuum tight laser welding
- fine polishing

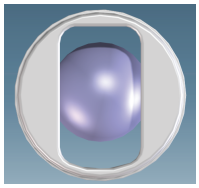
beutter
PREMIUM PRÄZISIONS-KOMPONENTEN

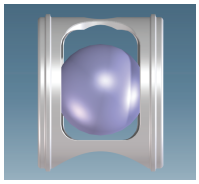
Fraunhofer IWS
Fraunhofer-Institut für Werkstoff- und Strahltechnik

PTB 

Density standard centering cage

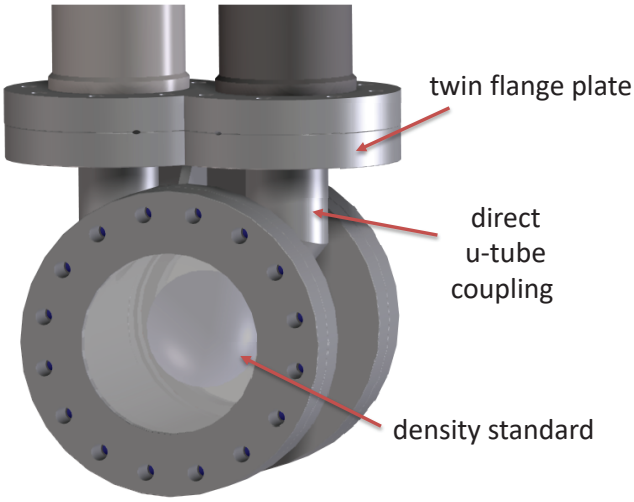






material: solid PTFE
density: 2200 kg/m³
inert with vacuum oils
bubble repellent

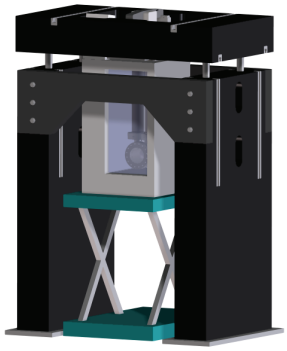
Temperature-of-flotation method (TOF)





Thermostatic bath

Temperature bath and elevation system



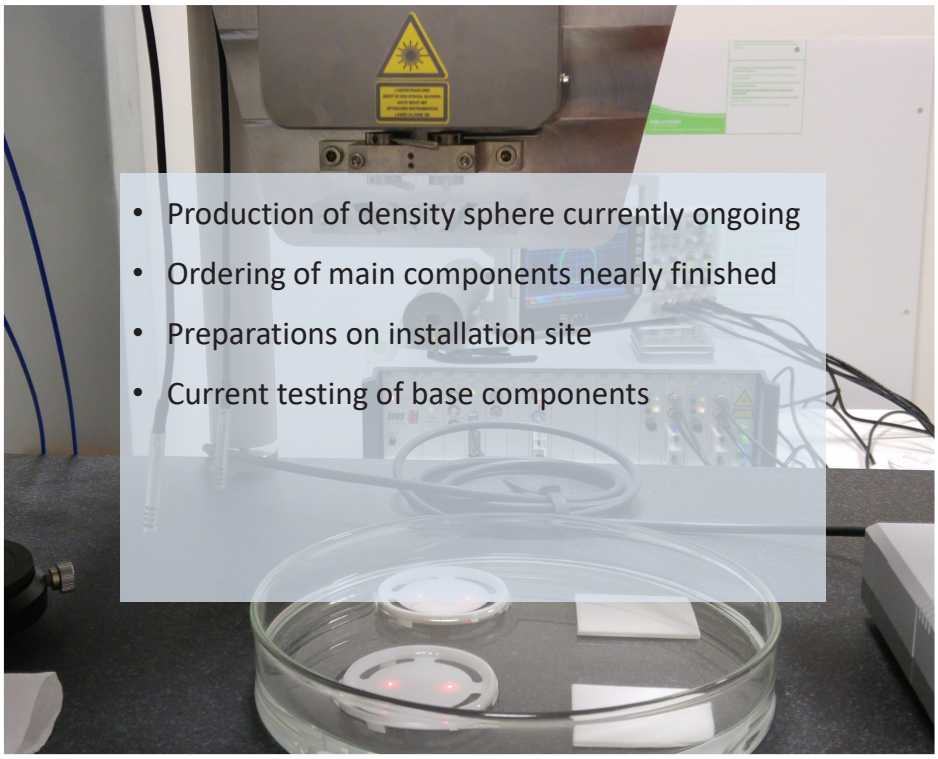


Commercial temperature bath:
- type: Tamson 7000 DC (NL)
- volume: 70 l
- homogenity: 0.01 °C @ 50 °C
- stability: <0.01 °C
- additional cooling bath for temperature control at room temperature level (20 °C).

Bath elevation system:
- non hydraulic spindle lift table

Summary

Summary



- Production of density sphere currently ongoing
- Ordering of main components nearly finished
- Preparations on installation site
- Current testing of base components

EMPIR
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

EURAMET

pres2vac
-A metrology research project within the European Union Horizon 2020 framework.

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Dr. Robin Wegge
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Norbert Ensemeier

Thank you!

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Companies are identified in this presentation to foster understanding. Such identification does not imply endorsement by PTB nor does it imply that the services or products of this companies are necessarily the best for the purpose.

Vacuum liquids for use in liquid column manometers

Sven Ehlers, Jens Könemann, Wladimir Sabuga

Physikalisch-Technische Bundesanstalt (PTB), Germany

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Vacuum liquids for use in liquid column manometers

Sven Ehlers, Jens Könemann, Wladimir Sabuga,
PTB Braunschweig, Pressure laboratory, Germany

Final Workshop, EMPIR Research Project “pres2vac”

Borås, Sweden
May 16th-17th, 2018

Concept

Requirements

Candidate liquids

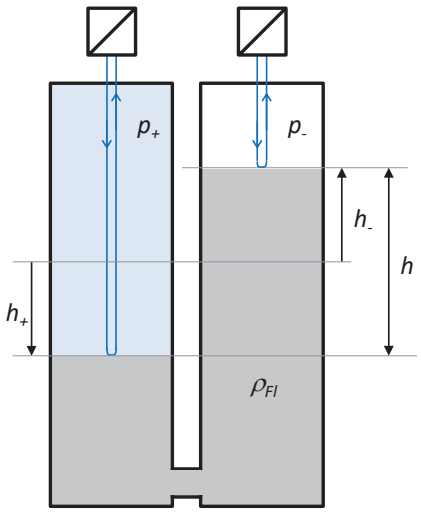
Measurements

Summary

Concept

Liquid column manometer - Functional principle

$$p_+ = \rho_{Fl} \cdot g \cdot h + p_-$$



Pressure range: 1 Pa to 2000 Pa

Main uncertainty contributions:

- *length measurement*
- *working fluid density*

Working fluids: Mercury vs. vacuum liquids

Mercury:

High vapour pressure 0.17 Pa

Very high surface tension 485 mN/m

High density 13 546 kg/m³

Poisonous 

+++

Very stable density

Low thermal expansion coefficient 0.2 ppm/mK

Vacuum liquids:

High therm. exp. coefficient 1 ppm/mK

Unstable density ±50 ppm/0.1 MPa

Pollution of apparatuses

+++

Very low vapour pressure <1 x 10^{-5} Pa

Low surface tension <50 mN/m

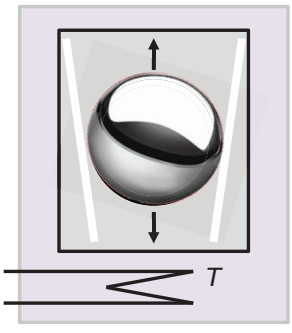
Low density >800 kg/m³

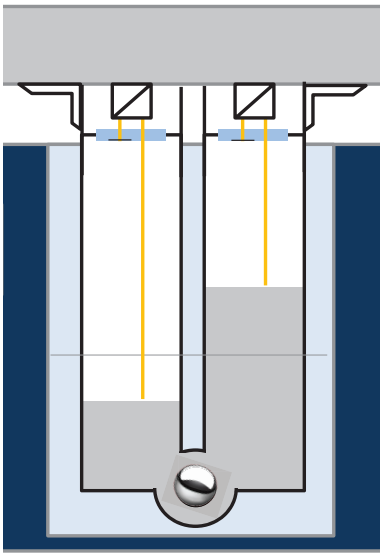
Easily measurable density ±5 ppm

In-situ density measurement (example)

Titanium density standard (D 58 mm)

- Temperature range: ±0.1 K
- Movement space: ±10 mm



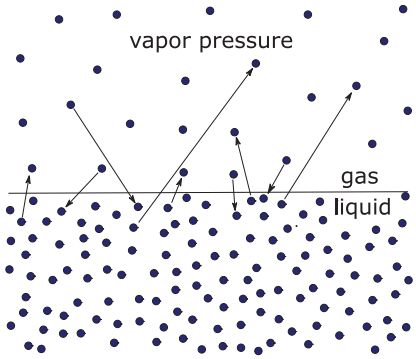


Density uncertainty: 10 ppm · ρ (in-situ)
Pressure uncertainty: 0.002 Pa + 20 ppm · p

Requirements

Vapor pressure

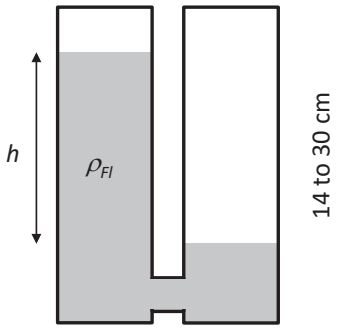
- better 2 magnitudes smaller than lowest measurable pressure.
- uncertainty contribution small or neglectable (absolute pressure measurements).
- low, to prevent internal fluid transport and pressure gradients.



Vapor pressure:
p_{vap} <= 0.1 mPa

Density

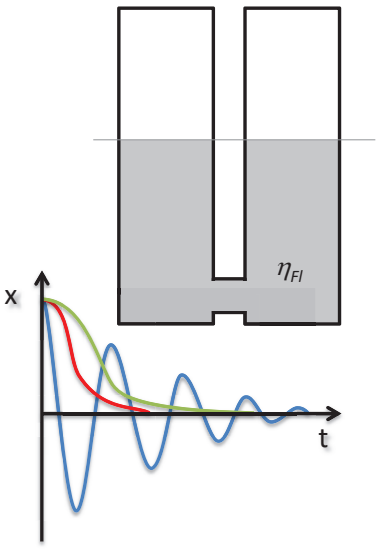
- Measurement range, geometric and thermal restrictions define the tolerated density range.



tolerated liquid density range:
ρ_{Fl} = 800 kg/m³ to 1500 kg/m³

Viscosity

- Dynamic liquid column displacement is a function of viscosity, density and geometry.
- Underdamped, critically damped or overdamped oscillation characteristics can occur.
- Critical damping reaches equilibrium the fastest.



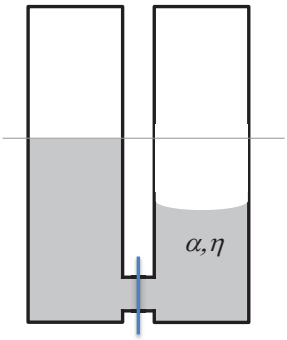
$$\ddot{x} + 2\beta\dot{x} + \omega_0^2 x = 0$$

$$\beta = 16\eta/(\rho d^2) \qquad \omega_0 = \sqrt{2g/l}$$

Critical damping: η_{Fl} = 830 mPa s to 1790 mPa s
ρ_{Fl} = 700 kg/m³ to 1500 kg/m³, l = 0.7 m, D = 6 cm (idealized!)

Capillary properties

- After a pressure change a thin liquid layer will cover the inner wall.
- When neglecting fluid exchange between both legs, a deviation of the measured pressure will occur.

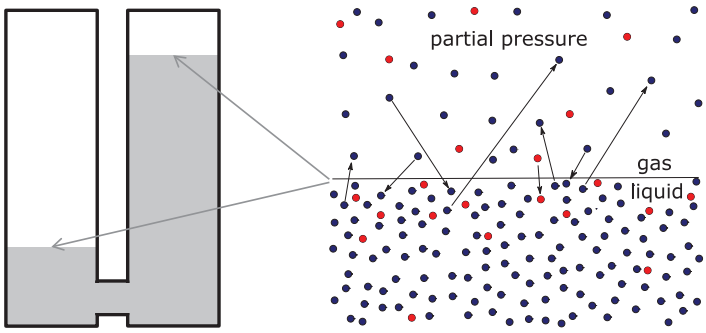


time t to reach min. pressure deviation Δp =0.1 Pa / min

contact	dynamic viscosity η / mPa s							
angle α / °	10	20	50	100	200	500	1000	2000
0,1	6,4	12,8	32,1	64,1	128,3	320,7	641,4	1282,8
1	6,4	12,8	32,0	64,0	128,0	320,1	640,1	1280,3
5	6,1	12,2	30,5	61,1	122,1	305,3	610,6	1221,3
10	5,4	10,7	26,8	53,5	107,1	267,7	535,4	1070,7
20	3,7	7,3	18,3	36,6	73,2	182,9	365,9	731,7
30	2,5	4,9	12,3	24,6	49,2	123,0	246,0	492,0
40	1,7	3,4	8,6	17,2	34,5	86,2	172,4	344,8
50	1,3	2,5	6,3	12,7	25,4	63,4	126,9	253,7
60	1,0	2,0	4,9	9,8	19,5	48,8	97,5	195,0
70	0,8	1,6	3,9	7,8	15,6	38,9	77,9	155,7

Gas solubility (1)

- Pressure transmitting medium (N_2/air) will be absorbed/desorbed by the working fluid.
- Gas solubility ~ p_{part}
- Absolute gas pressure level:
  gauge mode: 102 kPa max. / ambient pressure
  absolute mode : 2 kPa max. / vacuum



Gas solubility (2)

- literature sources:
  - [1] reports, a density decrease for a wide set of alkenes of around 70 ppm to 130 ppm after saturation with air or nitrogen (around 100 kPa)
  - In [2], Di-2-ethylhexyl sebacate (DEHS) density decreased:
    - 42 ppm after 24 h of air exposure (101 kPa)
    - no significant change after low pressure N2 exposure (1.3 kPa)

Data on gas absorption and desorption kinetics of other vacuum liquids have to be characterized.

[1] **S J Ashcroft, M Ben Isa,** Effect of Dissolved Gases on the Densities of Hydrocarbons, *J. Chem. Eng. Data*, **1997**, *42* (6), pp 1244–1248
[2] **J H Hendricks, J R Ricker, J H Chow & D A Olson**, Effect of Dissolved Nitrogen Gas on the Density of Di-2-Ethylhexyl Sebacate, *NCSLI Measure*, **2009**, *Volume 4*

Candidate vacuum liquids

List of candidate liquids

Seven candidate liquids are identified:

Company	Product name	Fluid	Class of the chem. compound	Vapor pressure p_{vap} / mPa	Density ρ / kg/m³	Dynamic viscosity η / mPa s
Merck	Bis(2-ethylhexyl) sebacate	F01	Carboxylate ester	5×10^{-2}	914	24
Edwards	E705	F04	Siloxane	4×10^{-5}	1090	190
Edwards	E704	F05	Siloxane	3×10^{-3}	1070	40
Vacom	InlandTW-1	F06	Hydrocarbon	1×10^{-3}	860	48
Santolubes	SantoVac 5	F08	Ether	3×10^{-5}	1195	2900
Santolubes	SantoVac 9	F09	Ether	4×10^{-3}	1170	210
Edwards	45	F10	Hydrocarbon	1×10^{-2}	830	43

Measurements

Density / Thermal expansion / Compressibility / Kinematic viscosity

- 7 liquids were analyzed with a oscillating type densimeter.
- 3 liquids (out of 7) were characterized using PTBs hydrostatic weighing apparatus.
- Kinematic viscosity was determined with an Ubbelohde-type capillary viscometer.

The accuracy of the hydrostatic weighing results allows a pressure measurement with an uncertainty of nearly 3 ppm (assuming stable condition).

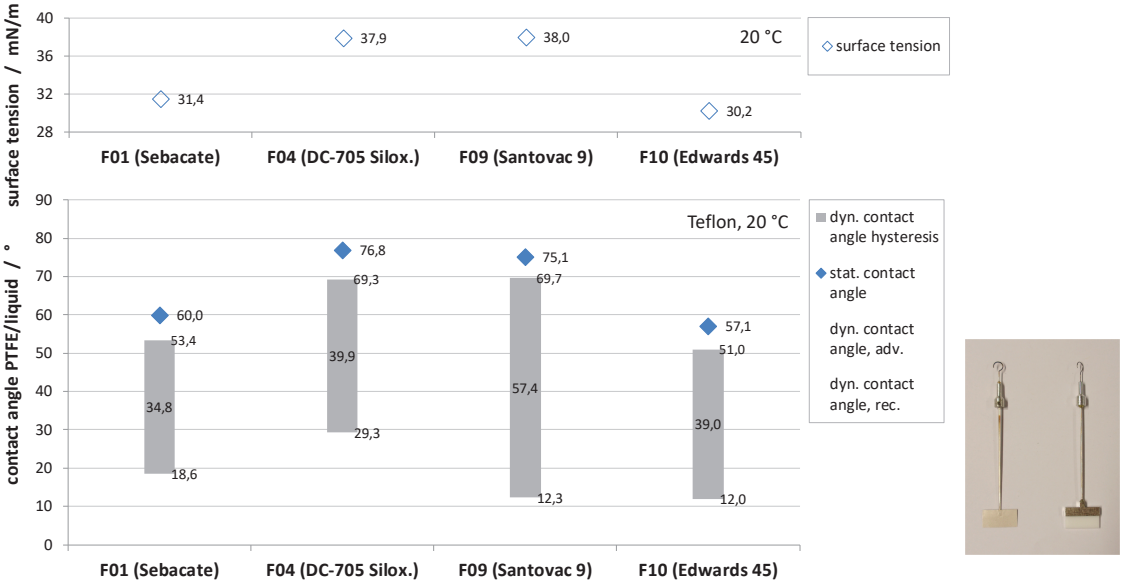


® Dr. H. Wolf, PTB Department 3.3



17

Surface tension and wettability



The combination of PTFE's weak wetting characteristic and its application like surface may explain the pronounced contact angle hysteresis.

Sven Ehlers, PTB Pressure Lab

18

Degassing of liquids

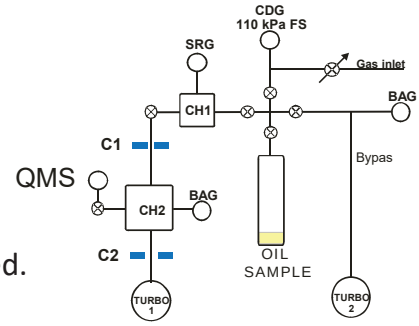
- A first degassing test using an oscillation type densimeter and F04 was performed.
- Degassing procedure:
  - A flat glas bowl was filled 5 mm high with F04.
  - Inside a vacuum oven a pressure of 0.3 kPa was applied 24 h a day.
  - An additional 110 °C treatment of 5 h each day.
- Time variation 1 day / 2 days / 3 days.



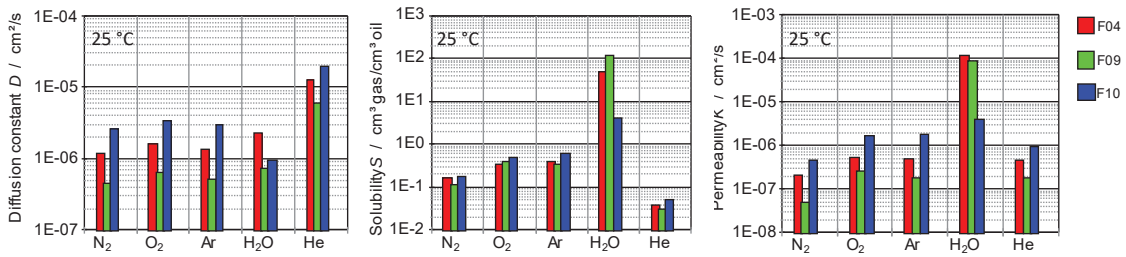
→ Up to 90 ppm rel. density increase when degassing.
→ New questions:
- ➢ Which gas fractions are leaving the liquid?
- ➢ How fast is this process?

Gas absorption and desorption kinetics (1)

- Outgassing rate of F04/F09/F10 was determined using a quadrupole mass spectrometer (QMS).
- Nearly saturated liquid was connected under vaccum with a QMS which detects the gas flow leaving the liquid.
- Nitrogen, argon, oxygen, helium (1000 mbar) and water vapor (19 mbar, eq. 60 % rH) were used.
- Finite Difference Method (FDM) allows the calculation of the diffusion constant *D*, gas solubility *S* and gas permeability *K*.

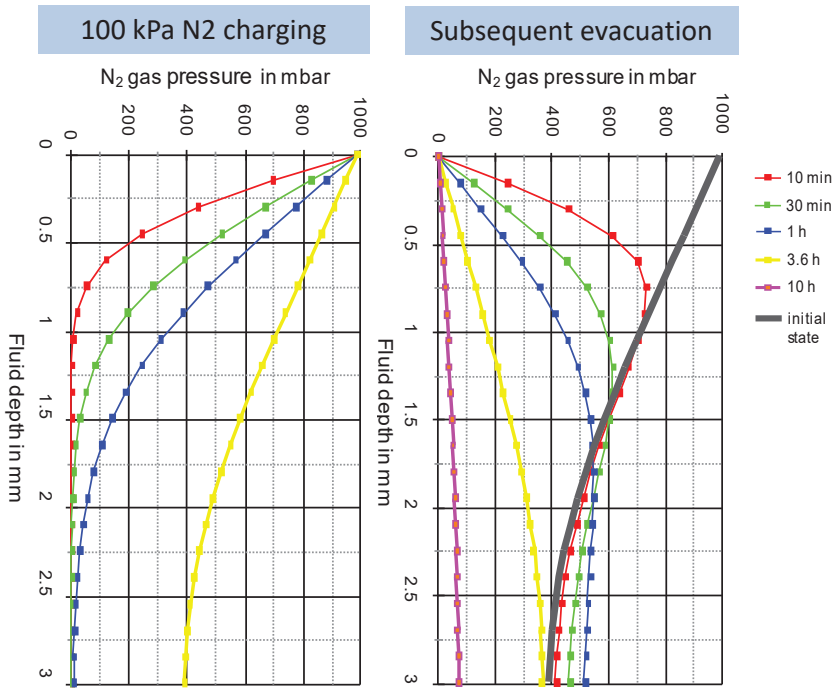


$K = D \times S$



Gas absorption and desorption kinetics (2)

With *D* and *S*, the calculation of the fluids inner gas pressure in dependence of fluid depth is possible.



Summary & Outlook

Summary & Outlook

- Seven vacuum oils out of four chemical classes were selected.
- Their liquid properties were determined.
- The liquids
  - F04 Edwards E705 - siloxane, vapor pressure and wettability
  - F09 Santolubes SantoVac 9 - ether lowest nitrogen solubility
  - F10 Edwards 45 – synth.hydrocarbon low density, viscosity and water solubility

  are considered as the best candidates for use.
- Surface tension and contact angle on PTFE was measured.
- Density change with gas saturation, absorption and desorption kinetics and diffusion coefficients of nitrogen, oxygen, argon, helium and water were determined.

→ Long term density measurements are ongoing.
→ F10 was chosen for further experiments.

pres2vac
-A metrology research project within the European Union Horizon 2020 framework.

EMPIR EURAMET
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Wladimir Sabuga
Henning Wolf
Jens Könemann
Oliver Ott
Thomas Konczak
Frank Lorenz

Isabel Spohr
Andreia Furtado

Janez Setina

Thank you!

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Measurement of diffusion and solubility of atmospheric gases in candidate oils for oil manometer

Janez Šetina

Institute of Metals and Technology (IMT), Slovenia

Measurement of diffusion and solubility of atmospheric gases in candidate oils for oil manometer

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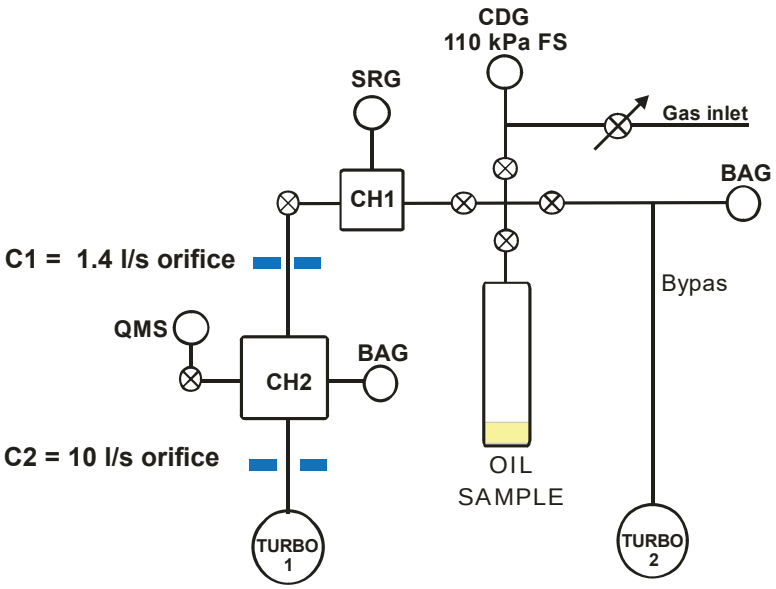
A1.3.4

From the JRP protocol:

Using input from A1.3.2 and A1.3.3, PTB will select at least 3 of the candidate oils as the most suitable fluid for the oil-based micromanometer and will then provide them to IMT for a study of the gas absorption/desorption dynamics.

- Assembly of "dedicated" vacuum system with a quadrupole mass spectrometer for measurement of outgassing rate of N_2, O_2 and Ar from the oil
- De-gassing of oil by exposure to vacuum for sufficiently long period (to be determined by experiment)
- Exposure of oil to selected gases (N_2, O_2 , Ar), He and H_20), at defined pressure until saturation
- measurement of outgassing rate as a function of time and extraction of diffusivity and solubility (for modeling of transient phenomena induced by gas exposure)
- Estimation of vapor pressure of oil

IMT system for outgassing measurements from oil samples



$A/\,Calibration:$

$$q_{cal,i} = C1_i \times p_{SRG,i}$$

$B/\,Measurement:$

$$q_i = C2_i \times p_{QMS,i}$$

$$q_i = \Psi_i \times I_i^+$$

$where:$

$$\Psi_i = \frac{C2_i}{S_i}$$

Ref :

EMRP IND12 project

Oils provided by PTB:

OIL	Vapour pressure [mbar] (at 25° C)
PTB04	$4x10^{-10}$
PTB09	$4x10^{-8}$
PTB10	$1x10^{-6}$

Oil PTB04 3 minutes
after start of evacuation



Oil PTB09 during
“degassing”



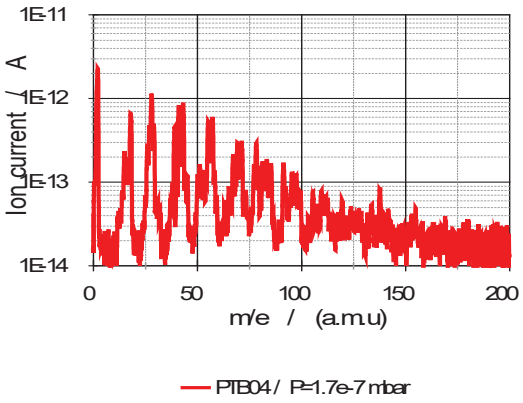
Condensation of oil vapours on cold surfaces (25 °C)

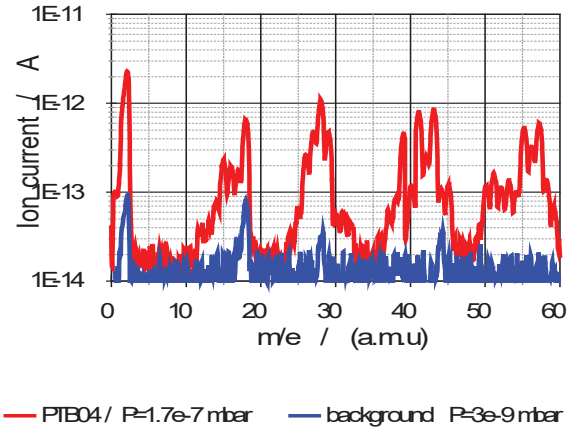
Heated in water bath at 50 °C

How much is vapour pressure of oil ??

for PTB04: 4×10^{-10} mbar (declared)

QMS spectrum of PTB04 oil after 3 days of pumping ($P_{tot}=1.7 \times 10^{-7}$ mbar)



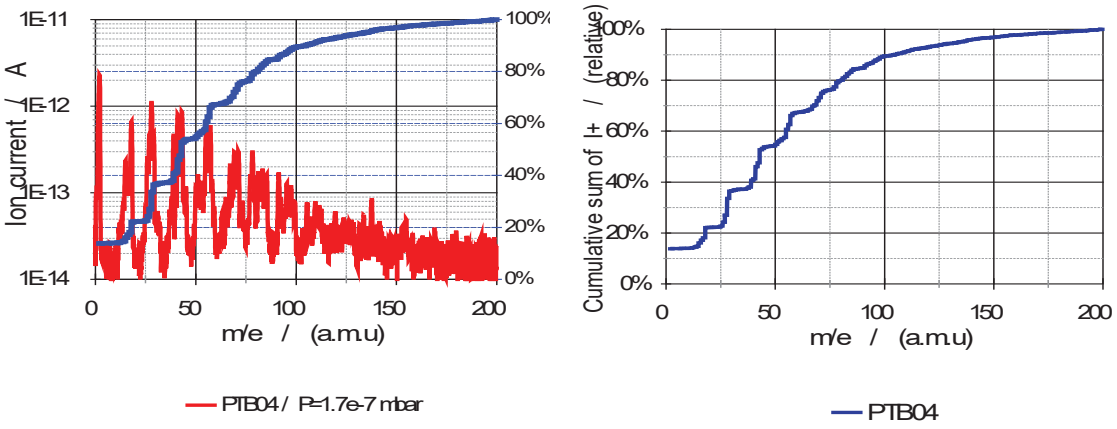


Masses between 25 and 200 represent more than 75 % of ion current

Rough estimate of partial pressure of hydrocarbons above m/e=25:

0.75 x 1.7x10^{-7} mbar = **1.3x10^{-7} mbar (300 times more than declared!)**

Good news: partial pressure of hydrocarbons is slowly decreasing with time)



Measurements of gas diffusion in oil sample PTB04

Measurements were performed with: **N_2, Ar, O_2, He and H_2O**

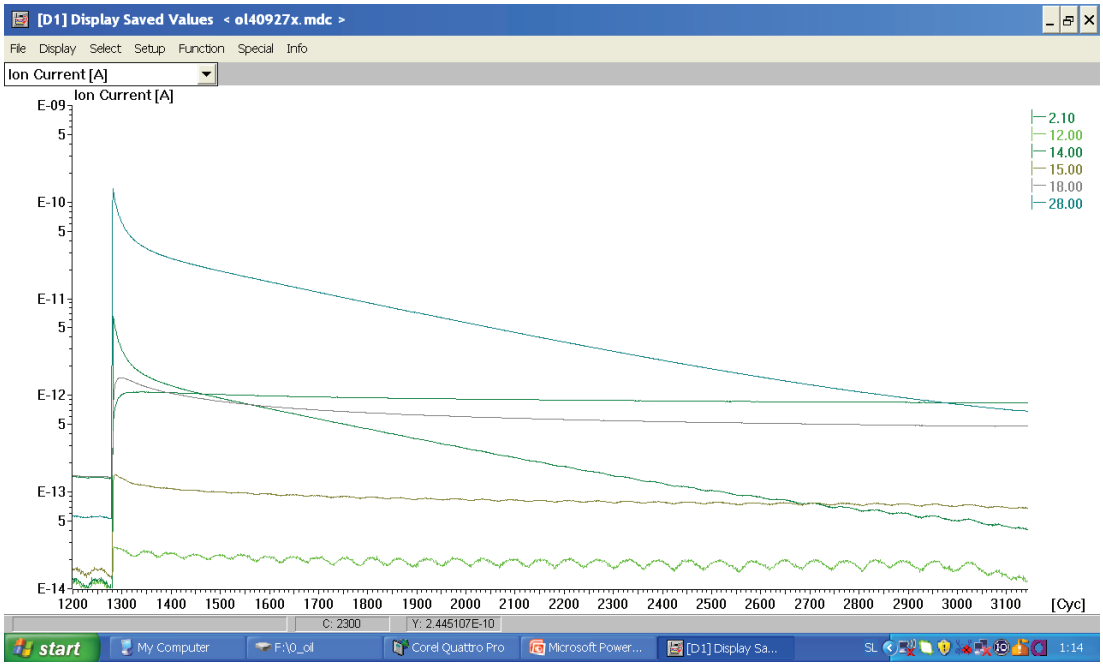
3 mm thick layer of **pre-degassed** oil was introduced in SS reservoir

Procedure:

- Sample was first pumped (degassed) for 3 days
- Selected gas was introduced above the oil surface

  (pressure around 1000 mbar, exposure duration few hours)
- After that the gas was pumped away by a bypass pumping line
- After few minutes (in case of H_2O after 20 minutes) the bypass was closed and pumping was switched to the QMS system

Raw data:

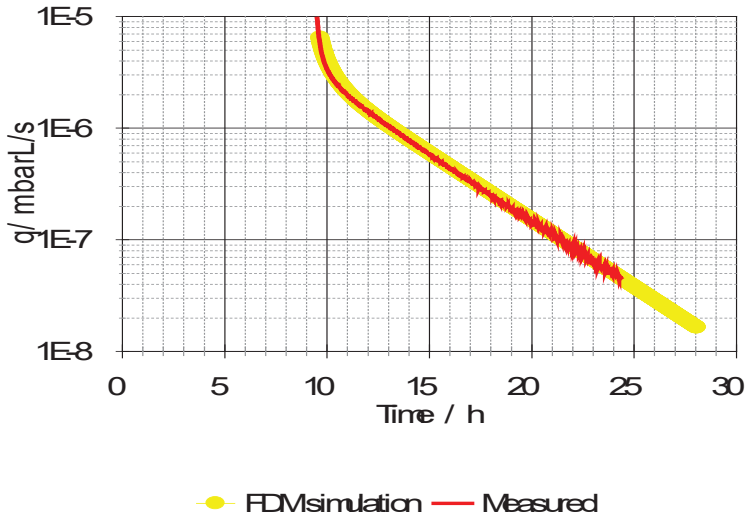


Procedure (continued):

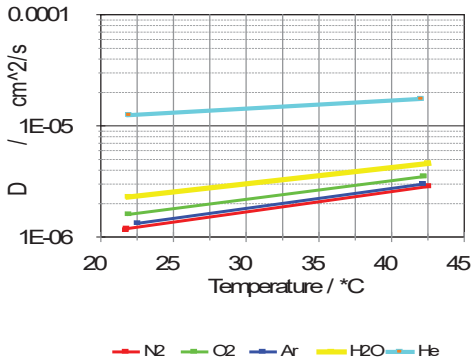
- From ion current of QMS the gas flow from the sample (outgassing rate) was calculated)
- Time dependence of the gas flow from the sample was modelled assuming pure diffusion process
- Modelling was performed numerically using finite element method (FEM)
- Modelling included gas dissolution into the previously degassed oil sample during gas exposure
- From the model the diffussion constant D, gas permeability K and gas solubility S was determined

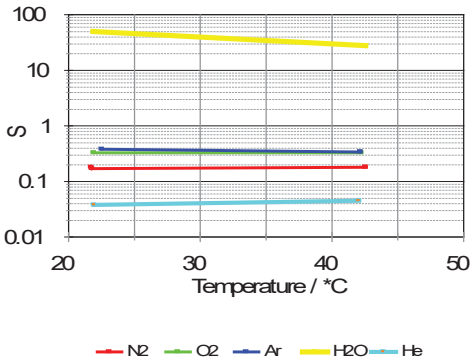
Example of measured and modelled outgassing of N2 after 9.4 h exposure at P = 311 mbar

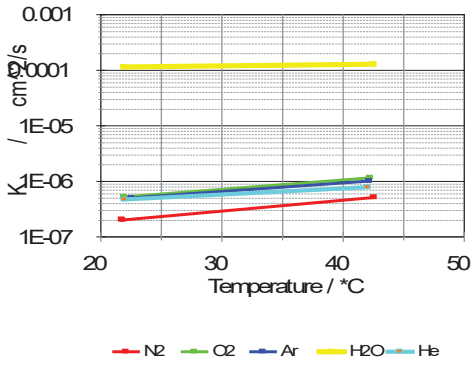
D=1.19 x 10^{-6} cm^2 K=2.02 x 10^{-7} cm^2 S= 0.17



gas	T	D	K	S=K/D
N_2	21.7	1.15E-06	2.07E-07	0.18
	21.8	1.19E-06	2.02E-07	0.17
	42.6	2.85E-06	5.13E-07	0.18
O_2	21.9	1.60E-06	5.28E-07	0.33
	42.2	3.50E-06	1.16E-06	0.33
Ar	22.5	1.33E-06	5.05E-07	0.38
	42.2	3.00E-06	1.02E-06	0.34
H_2O	21.9	2.30E-06	1.15E-04	50
	42.5	4.60E-06	1.29E-04	28
He	21.9	1.25E-05	4.75E-07	0.038
	42.0	1.75E-05	7.87E-07	0.045





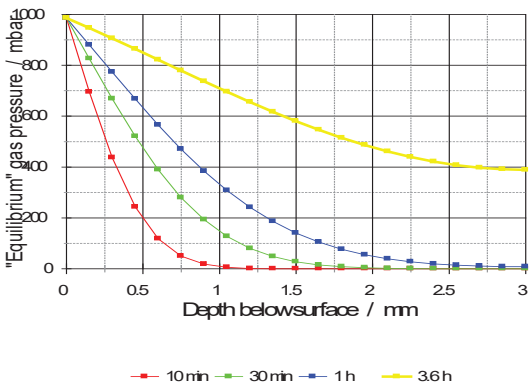


$K = D \times S$

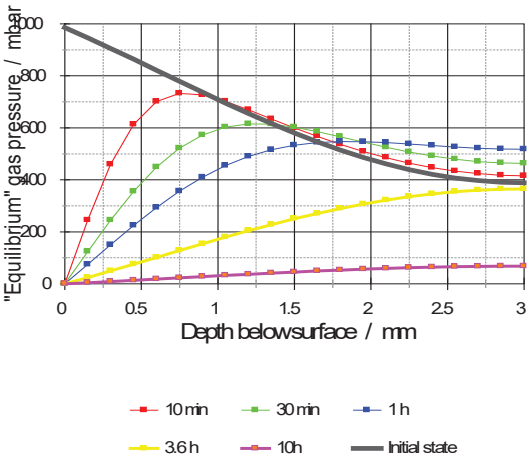
PTB04 oil, N_2

Layer thickness: 3 mm **loading pressure/time: 986 mbar/3.6 h**

Gas loading phase

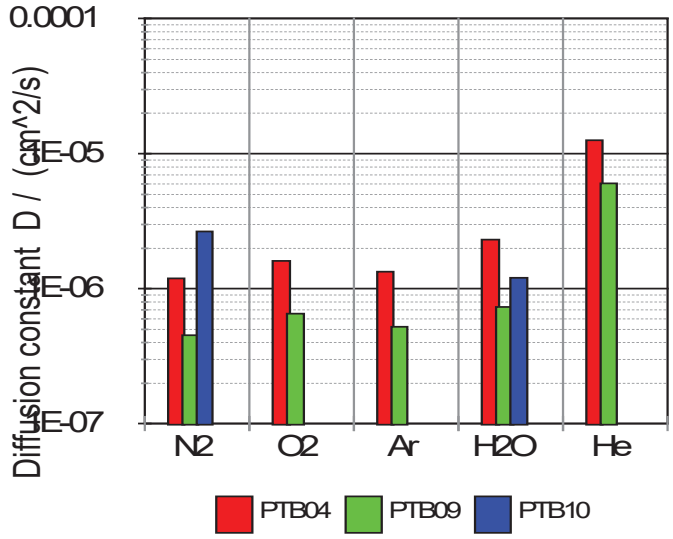


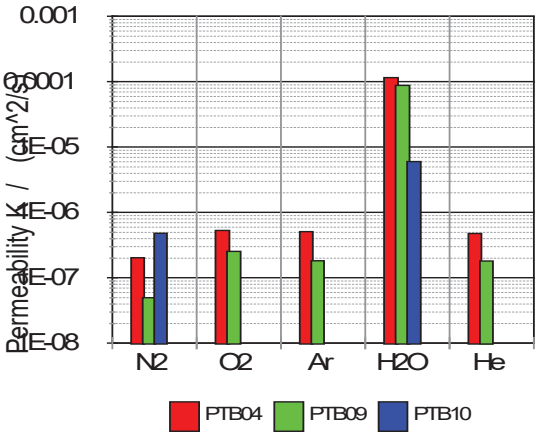
Degassing phase

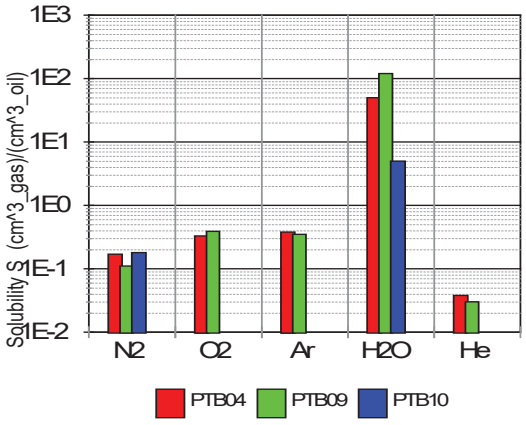


Results of diffusion constant D, permeability K and solubility S for oil PTB04 have been already presented in Tres Cantos.

During this period we measured samples
PTB09 and **PTB10** at **temperature 25*C**

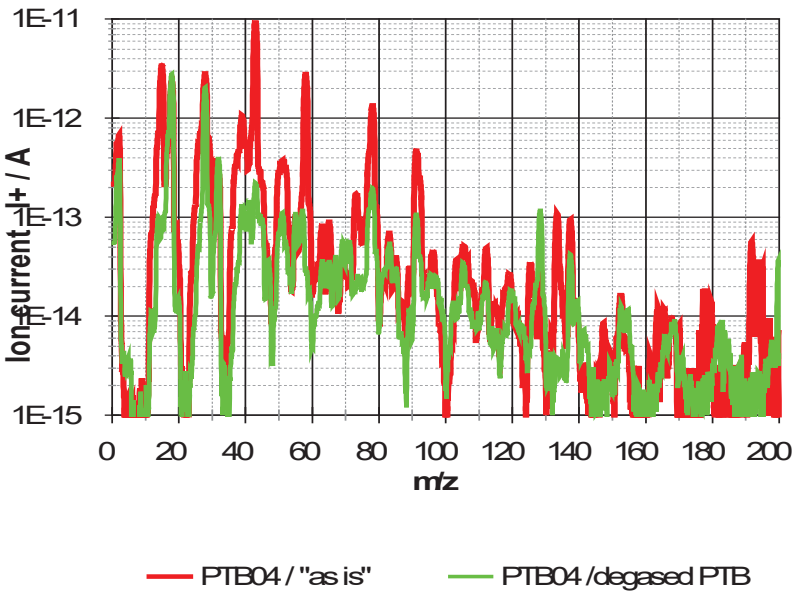


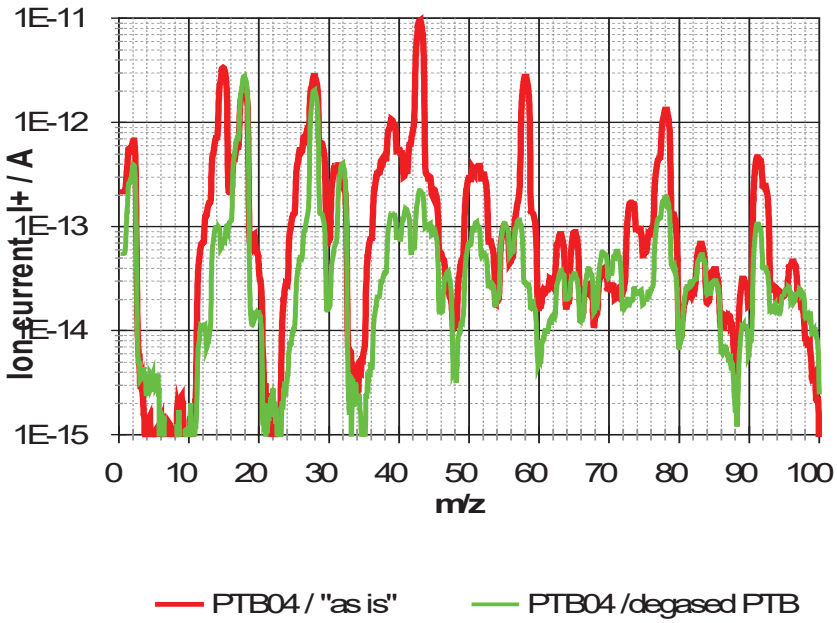




In addition we performed comparison of "hydrocarbon outgassing" from different oil samples

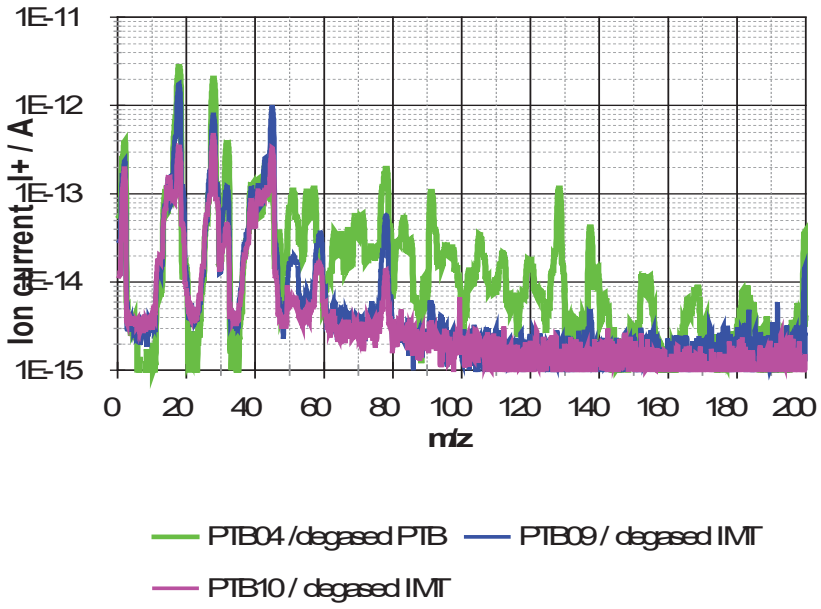
Mass spectrum after 20h of pumping:
(comparison of PTB04 without degassing and after degassing)

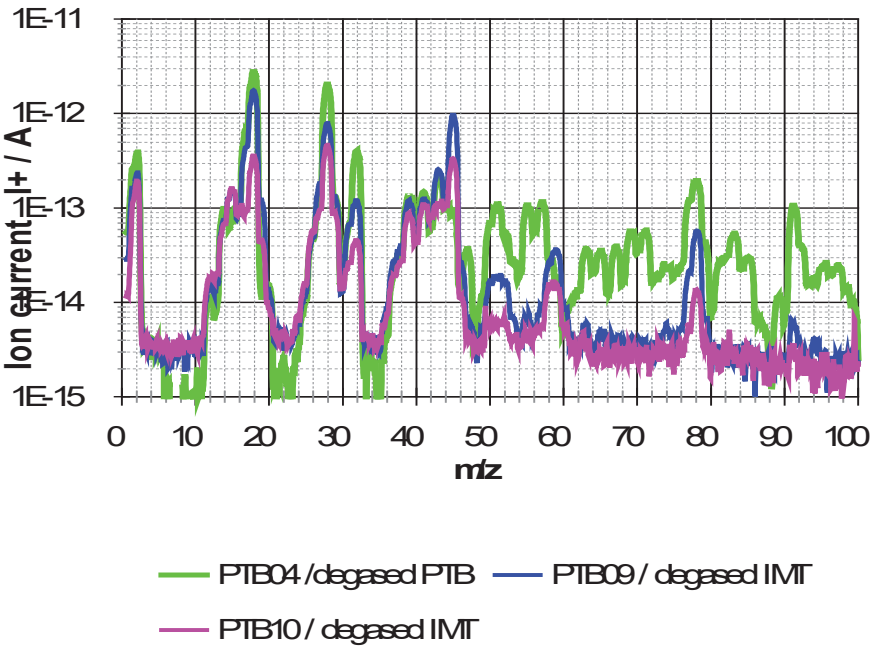




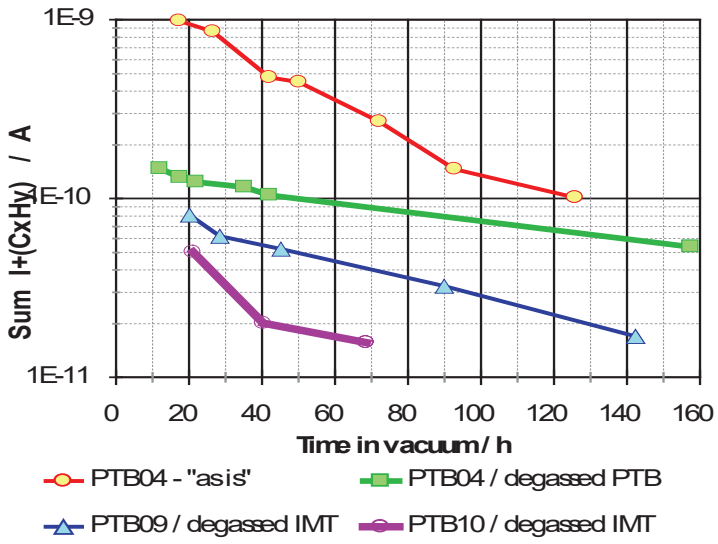
Mass spectrum after 20h of pumping:

(comparison of PTB04, PTB09 and PTB10 after degassing)





Total amount of hydrocarbons C_xH_y was estimated as sum of ion currents in mass ranges: **[4 to 16] + [20 to 26] + [33 to 43] + [45 to 200]**



Degassing in IMT:

Pumping by oil diffusion pump

PTB09
3 days at 120°C
+ 15 days at room temp.
$P<1.5\times10^{-6}$ mbar

PTB10
5 days at 120°C
+ 1 day at room temp.
$P<2\times10^{-6}$ mbar

Industrial standards in the intermediate pressure-to-vacuum range – Determination of physical properties of candidate fluids by IPQ

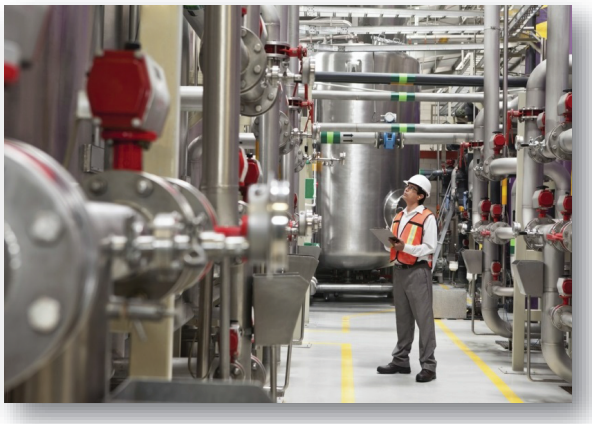
Isabel Spohr, Andreia Furtado, Jorge Pereira

Instituto Português da Qualidade (IPQ), Portugal



Instituto Português da Qualidade

Industrial standards in the intermediate pressure-to-vacuum range



Isabel Spohr, Andreia Furtado, Jorge Pereira

Pres2Vac (EMPIR 14IND06) Final Workshop
16 May 2018, Borås

IPQ

Background

PTB defined the **parameter ranges** of candidate oils suitable as manometric liquids for the oil-based interferometric micromanometer for:

density **thermal expansion coefficient** **compressibility** **viscosity**

vapour pressure density variability with gas content surface tension
reflectivity long-term stability

TUBITAK and PTB have independently search for candidate oils that fulfil the parameters previously defined (sources included literature data, databases and data sheets of manufacturers).

UME –TUBITAK presented a list of candidate oils for manometric liquids together with their properties.

PTB have chosen at least 6 of the best potential candidate oils from the list. Criteria for the selection was the optimal properties of the oils and the possibility to acquire them.

The **selected oils** were studied experimentally at **IPQ** and the results obtained are given in this presentation.

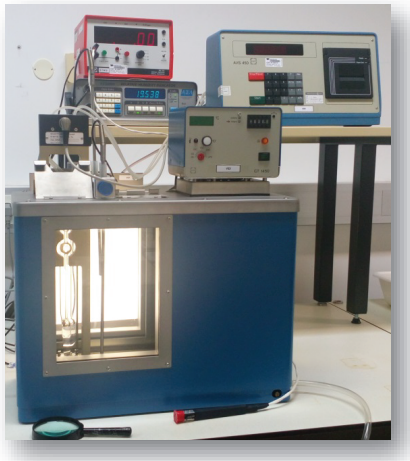
Task 1.3 - Characterization of the candidate oils (Density)

A1.3.2 **Part I**	**7 candidate oils** ▪ Measure density: (15, 20 and 25) °C, atmospheric pressure ▪ Uncertainty ~ 0.03 kg/m^3 ▪ Oscillation-type density meter (DMA 5000, Anton Paar) ▪ **Determination of thermal expansion coefficient**



Task 1.3 - Characterization of the candidate oils (Viscosity)

A1.3.3	**Measure the viscosity of 7 oils at temperature 20 °C** • Capillary viscometers • Uncertainty below 0.6%



Density, Thermal Expansion Coefficient and Kinematic Viscosity

The density and the thermal expansion of the fluids were determined with an oscillation-type densimeter DMA 5000 from Anton Paar.

To determinate the thermal expansion of each fluid it was performed three temperature cycles in the temperature interval from 15 °C to 25 °C, with 0.5°C steps.

It was necessary to determine the viscosity-temperature dependence in the temperature interval from 15 ºC to 25 ºC using a Stabinger viscometer from Anton Paar to correct the damping effect observed in density measurements with the oscillation density meter.

The kinematic viscosity of the fluids, at 20 ºC and ambient pressure, was measured with Ubbelohde-type capillary viscometers.

Resume of the determined physical properties, at 20 C and atmospheric pressure, of the candidate fluids including the expanded measurement uncertainty (U) for a coverage factor k =2.

Fluid	Density (ρ) at 20 ºC ± U_ρ kg/m³	Thermal expansion coefficient (γ) at 20 ºC ± U_γ/ 10^{-4} K^{-1}	Kinematic viscosity (υ) at 20 °C ± U_υ / mm^2/s
F01	914.203 ± 0.030	8.0 ± 0.33	23.21 (1 ± 0.31 × 10^{-2})
F04	1095.268 ± 0.030	6.9 ± 0.28	268.5 (1 ± 0.26 × 10^{-2})
F05	1064.210 ± 0.030	7.3 ± 0.28	51.72 (1 ± 0.30 × 10^{-2})
F06	844.106 ± 0.030	7.2 ± 0.36	128.9 (1 ± 0.30 × 10^{-2})
F08*	1200.476 ± 0.030	6.6 ± 0.25	4882 (1 ± 0.31 × 10^{-2})
F09	1178.468 ± 0.030	6.9 ± 0.26	295.20 (1 ± 0.26 × 10^{-2})
F10	829.405 ± 0.030	7.4 ± 0.36	122.38 (1 ± 0.30 × 10^{-2})

* not possible to measure the temperature dependence of the viscosity of the F08 fluid in a Stabinger viscometer because of its high viscosity value

Task 1.3 - Characterization of the candidate oils (Compressibility)

A1.3.2

Part II

5 oils (PTB 1, PTB4, PTB5, PTB9 and PTB10)

- Density: 20 ºC, *p* (0.1, 0.5 and 1.0) MPa
- Samples: normal and degassed
- Oscillation-type density meter for high *p* (DMA HP, Anton Paar)
- Uncertainty ~ 0.1 kg/m^3
- **Determination of compressibility**

IPQ's apparatus developed for density measurements up to 10 bar

Density measuring cell (DMA HP- Anton Paar + DMA 500- Anton Paar) pressurization with **nitrogen**

Oils degassing was performed with a **membrane degasser**



Coefficient of Isothermal Compressibility

It was determined the coefficient of isothermal compressibility (cL) of five oils (F01, F04, F05, F09 and F09), in the pressure interval from atmospheric pressure up to 1 MPa, at 20 ºC. These measurements were done with these oils in saturated (s) and in degassed (d) forms, to compare the influence of the gas content in the compressibility.

Resume of the coefficient of isothermal compressibility (cL), at 20 ºC in the pressure interval from ambient pressure up to 1 MPa, of the candidate fluids including the expanded measurement uncertainty (UcL) for a coverage factor k =2.
Legend: s – saturated sample; d – degassed sample.

The values of the coefficient of isothermal compressibility of the saturated samples are greater than for the degassed sample.
As the differences are within the uncertainty, it is not possible to make further conclusions for the differences obtained between the two states of each sample.

Sample	Sample state	c_L at 20 ºC /10^{-5} bar^{-1}	U_{cL} /10^{-6} bar^{-1}
F01	s	8.05	1.8
	d	7.98	1.8
F04	s	6.81	2.4
	d	6.67	2.4
F05	s	7.34	2.9
	d	7.14	2.8
F09	s	6.70	5.3
	d	6.23	5.0
F10	s	8.76	3.0
	d	7.64	2.6

Density Pressure dependence

For density pressure dependence, five oils were tested at 20 ºC in the pressure interval from 0.1 MPa to 1 MPa. Measurements were performed for each oil with saturated samples and degassed samples.
The expanded uncertainty for:
density measurements 0.14 kg/m^3
pressure measurements 1.2 kPa

Resume density measurements at 20 ºC in the pressure interval from 0.1 MPa up to 1 MPa, of the candidate oils.
Legend: s – saturated sample; d – degassed sample

Sample →	F01		F04		F05		F09		F10	
Nominal p / MPa	ρ/ kgm^3									
	s	d	s	d	s	d	s	d	s	d
0.10	914.203	914.254	1095.268	-	1064.210	1064.327	-	1178.468	829.418	829.487
0.25	914.309	914.361	1095.424	1095.352	1064.356	1064.439	1177.628	1178.644	829.527	829.561
0.50	914.493	914.541	1095.599	1095.544	1064.542	1064.629	1177.848	1178.813	829.705	829.742
0.75	914.679	914.727	1095.776	1095.725	1064.733	1064.821	1178.033	1178.981	829.875	829.921
1.0	914.863	914.909	1095.954	1095.900	1064.923	1065.009	1178.224	1179.150	830.056	830.096

Conclusion

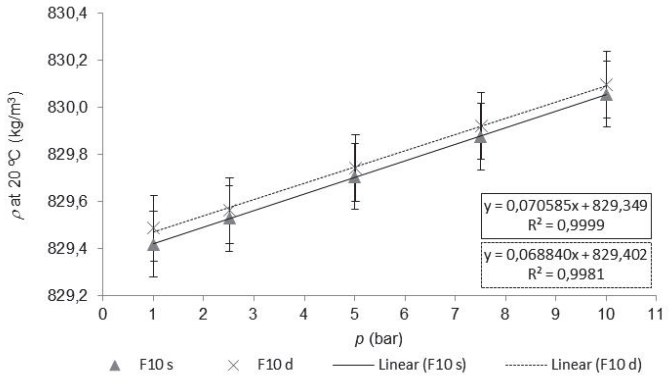
PTB selected 3 oils from the oils tested at IPQ:

- ✓ Then PTB repeated the measurements done at IPQ with hydrostatic weighing method.
- ✓ And IMT perform a detailed study of gas absorption/desorption in/from oil when it is exposed to pressure of up to 100 kPa or is evacuated.

Finally from the 3 oils tested the oil chosen is **F 10**.

Fluid	Density (ρ) at 20 ºC ± U_ρ kg/m³	Thermal expansion coefficient (γ) at 20 ºC ± U_γ/ 10^{-4} K^{-1}	Kinematic viscosity (υ) at 20 °C ± U_υ / mm²/s
F10	829.405 ± 0.030	7.4 ± 0.36	122.38 (1 ± 0.30 × 10^{-2})

Sample	Sample state	c_L at 20 ºC /10^{-5} bar^{-1}	U_{cL} /10^{-6} bar^{-1}
F10	s	8.76	3.0
	d	7.64	2.6



Density pressure dependence of the test fluid F10 at 20 ºC in the pressure interval from 0.1 MPa to 1 MPa.
The vertical bars represent the expanded uncertainty of density measurements 0.14 kg/m³.
Legend:
s – saturated sample;
d – degassed sample.

Thank you for your attention!



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Digital pressure balances with non-rotating piston – FRS and FPG

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Český metrologický institute (CMI), Czech Republic

Digital pressure balances with non-rotating piston - FRS and FPG

Dominik Pražák, Zdeněk Krajíček, Martin Vičar

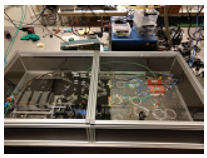
Czech Metrology Institute

pres2vac

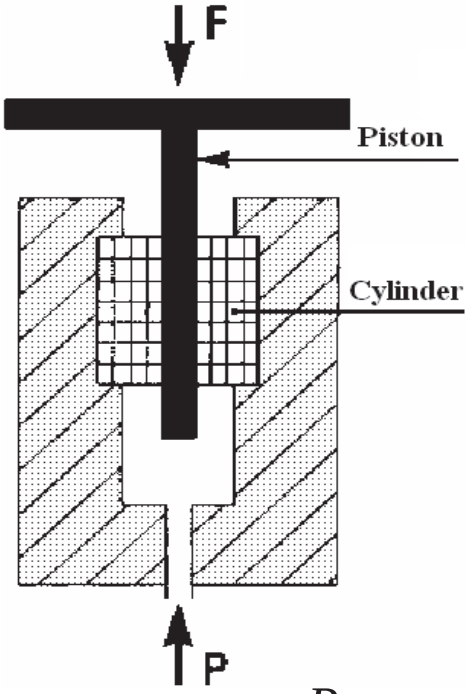
-A metrology research project within the European Union Horizon 2020 framework.

Micro-manometers

- The micromanometers are designed for the pressures below 6650 Pa (50 mmHg), [Brombacher, W.G.: *Survey of Micromanometers*. NBS Monograph 114. Washington: NBS, 1970].
- Low gas flows, leaks, air-conditioning, aircrafts.
- There are many principles, but we focus on those primary:

1. liquid columns (*in the morning*), 
2. diving bells, 
3. **digital pressure balances (mainly FRS and FPG)**,
4. photonic realisation of pressure unit (*tomorrow*). 

Pressure balances



Measurement of low-pressure differences and low absolute pressures using classical pressure balance with rotating piston has two basic limits:

1. It is possible to measure only pressures that are at least big enough for balancing of the weighting mass of the piston. This does not enable us to measure pressures lower than that of the order of kilopascals. (*Tilting in 50s*)
2. The periodic pressure fluctuations caused by rotation of the piston also contribute to the uncertainty of low-pressure measurements.

$$P = \frac{F}{A(P,T)} = \frac{m(1-\rho_a/\rho_m)g}{A(P,T)}.$$

Digital pressure balances

digital piston design with electronic dynamometer for measurement and compensation of gravity force of the piston

+ basic advantages:

- low range is limited only to resolution and accuracy of force transfer from rotating piston-cylinder unit and non-rotating head of dynamometer,
- possibility use digital piston gauge for very accurate pressure measurement, not only pressure generation.

− basic disadvantages:

- limited accuracy of force transfer from rotating piston-cylinder unit to non-rotating head of dynamometer,
- low long-term stability of dynamometer.

Non-rotating digital pressure b.

Piston manometer utilizing connection of a non-rotating piston to an electronic dynamometer can measure even very small changes of equilibrium state of the piston

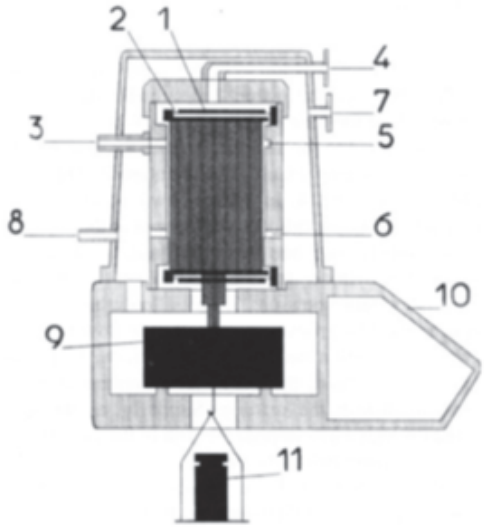
+ basic advantages:

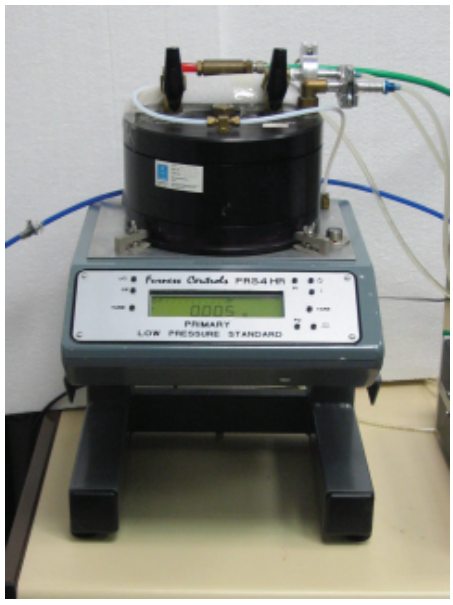
- no problems with force transfer from the piston to the dynamometer
- possibility of large effective area (today maximum 100 cm^2)

− basic disadvantages:

- problem with dynamometer stability
- problem with centralisation of piston position in the cylinder (whilst a rotating piston is self-centering, in this case it is necessary to ensure its centering by means of *lubrication gas flow in the gap between piston and cylinder* or using *flexible hinges*)

Furness Controls FRS4



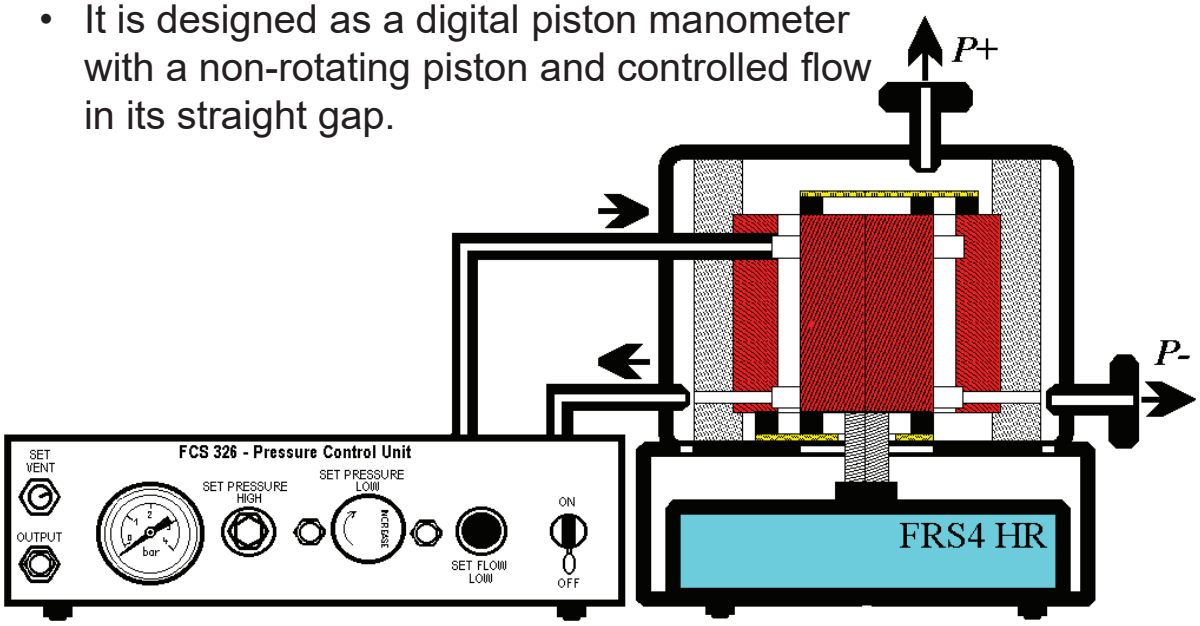


[Rendle, C. G.: A Large Area Piston Gauge for Differential Gauge Pressure from Zero to 3.2 kPa. *Metrologia*, Vol. 30, 1994, p. 611 – 613.]

[Rendle, C. G., Rosenberg, H.: New absolute pressure standard in the range 1 Pa to 7 kPa. *Metrologia*, Vol. 36, 1999, p. 613 - 615.]

Description

- CMI uses type Furness FRS 4 HR with range from 1 Pa to 3200 Pa in the gauge and negative gauge (differential) modes, U = 0.02 Pa + 80 ppm.
- It is designed as a digital piston manometer with a non-rotating piston and controlled flow in its straight gap.

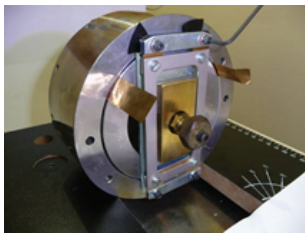


Description

- Nominal area of the piston is 100 cm^2, the width of the gap is cca 35 μm.
- Piston is directly mounted to a digital electronic balance with the range of 3.2 kg and resolution 1 mg in the range (0 – 1) kg and 10 mg in the range (1 – 3.2) kg.
- The calibration of the balance can be made using an external mass before each measurement (plus zeroing).
- Calibration is performed in such a way the output in grams to be equal to the generated pressure in pascals.
- This is ensured by adjusting the calibration mass taking into account the efective area of the piston-cylinder and local value of acceleration due to the gravity.

- Material of both the piston and the cylinder is invar - linear temperature expansibility coefficient is low ($\alpha = 2 \cdot 10^{-6}\ K^{-1}$).
- The centering of the piston must be ensured by suspension flexure hinges that allow free axial movement.
- The gas medium flow is controlled by the means of an external regulator (or the MFCs), the flow through the gap of cca 3.3 cm^3/min generates the full range value of generated pressure.
- The clean dry air or nitrogen can be used as pressure medium. In the case of nitrogen there is the necessity of rinsing of the whole system properly before the measurement.

Piston centering



- Firstly, the original Furness Controls centering method using 25μm feeler gauges.
- CMI added two own methods:

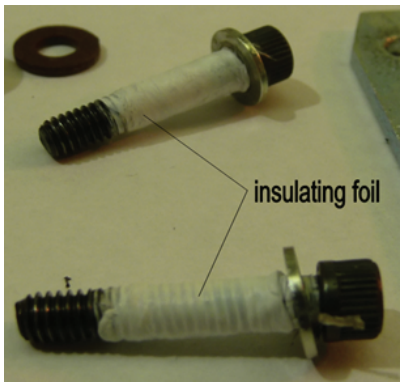
o *capacitance measurement method*,

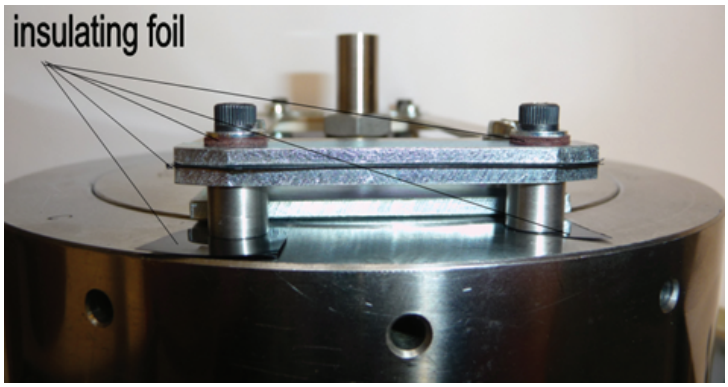
o *lower illuminance method*.



Capacitance method

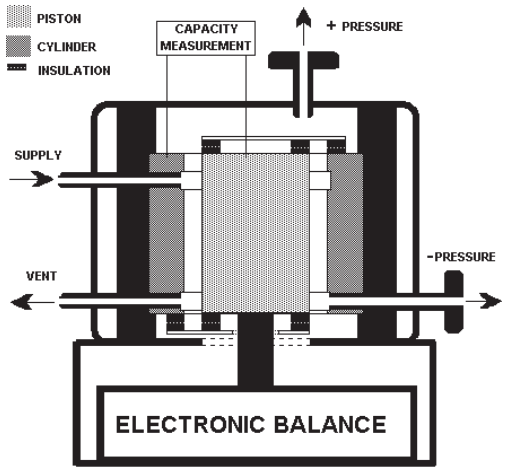
- Piston and cylinder are electrically insulated from other parts of the instrument.
- Then the capacity of that system is monitored, while the position of the piston is being changed.
- The minimal measured capacity indicates the best-centered position.

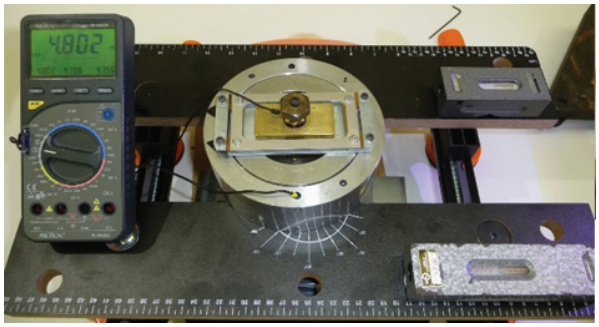




Capacitance method

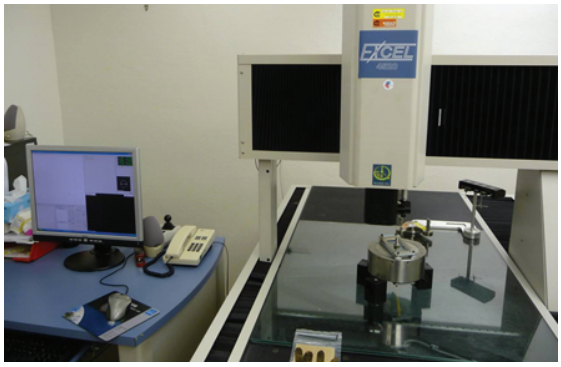
- With the repeated best-centrings we got 25 ppm A_{ef} repeatability with FPG.
- An intentional ±10 μm wrong-centering brought the additional differences up to 20 ppm.

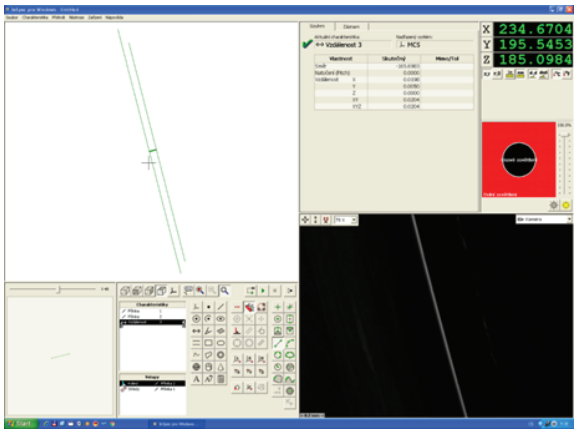




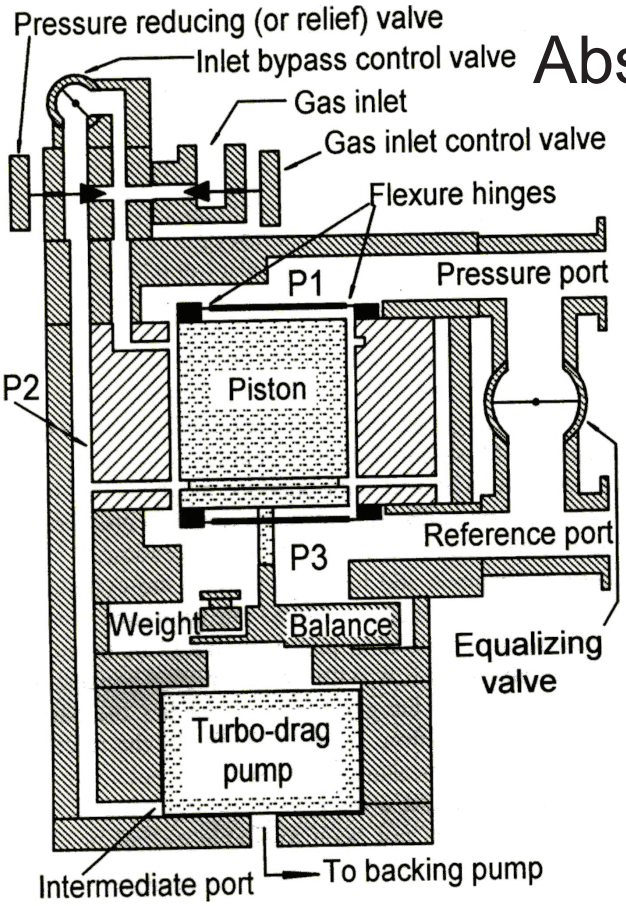
Lower illuminance method

- An optical profile-meter can be utilised for such a check.
- CMI used profile-meter Excel 4520 of the technical length department.
- It easily managed to check and measure the entire length of the free part of the gap, including a check of a presence of the impurities.





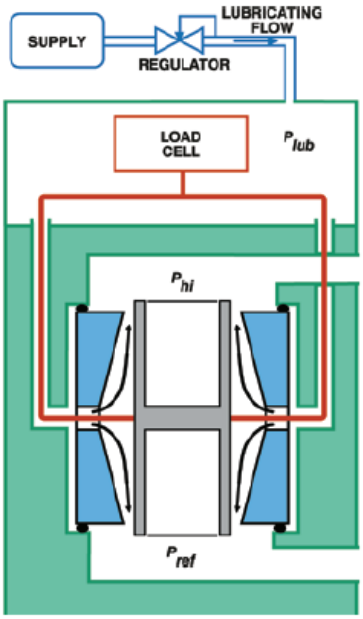
Absolute mode FRS5

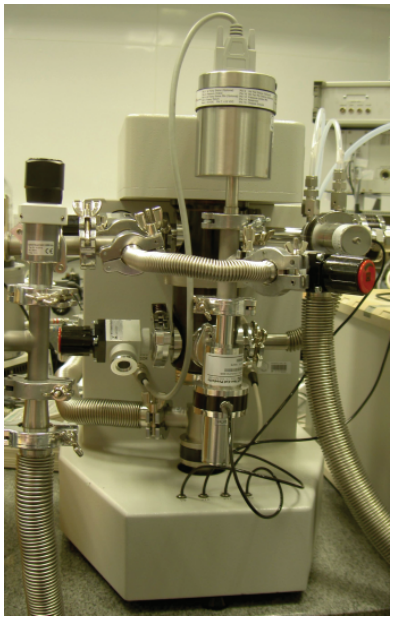




range: (1 – 11000) Pa of absolute and gauge
ref. pressure: 0.01 Pa (0.001 Pa below 2 kPa)
gap: cca 30 μm
effective area: 45 cm^2
no water vapour

DHI FPG 8601

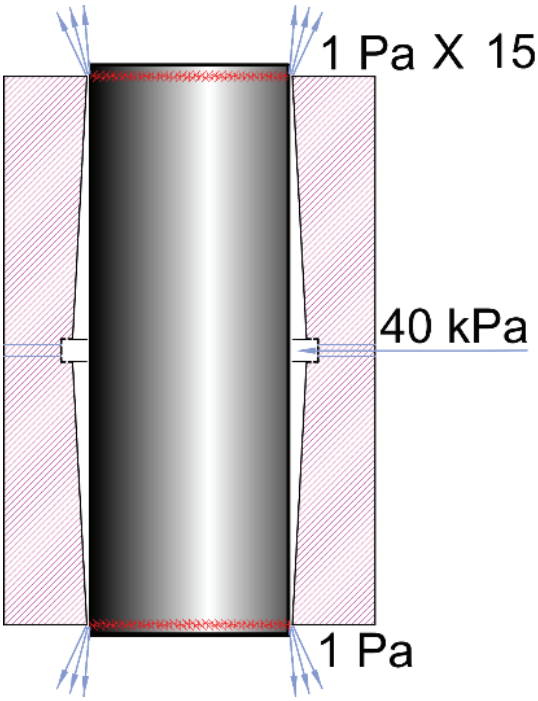




[Ooiwa, A.: Novel Nonrotational Piston Gauge with Weight Balance Mechanism for the Measurement of Small Differential Pressures. *Metrologia*, Vol. 30, 1994, p. 607 - 610.]
[Delajoud, P., Girard, M.: Kolbenmanometer mit Kraftmesszelle zur Kalibrierung sehr kleiner Drücke. *Vakuum in Forschung und Praxis*, Vol. 15, 2003, p. 24 - 29.]

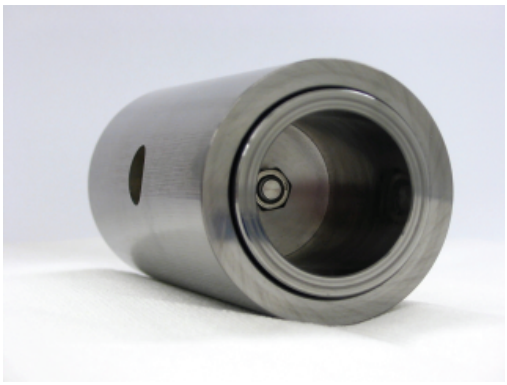
Description

- CMI uses instrument DHI FPG 8601 for the absolute, gauge and differential pressures (at line pressures lower then atmospheric) from 1 Pa to 15 kPa.
- Very low uncertainty of cca 0.02 Pa + 28 ppm.
- A non-rotating tungsten-carbide piston of 35 mm diameter is situated in a tungsten-carbide cylinder.
- It is connected to an electronic balance and centred by means of lubricating gas flow (flowrate less then 1 cm^3/min) in a gap between piston and cylinder.
- The gap has shape of a double taper and width from 1 to 6 μm.



$Pl \approx Pr$ + 40 kPa

$Ph \approx Pr$ + (0 – 15) kPa



- The temperature of the piston-cylinder is measured by two PRTs, serving also for determination of the temperature of gas.

- The force acting on the piston is balanced by the means of the electronic balance. Its range is 2 kg (0.5 kg needed for tarring the mass of the piston and its coupling parts, the rest serves for force measurements) and resolution 0.1 mg. It is placed on a massive aluminum plate for good temperature stabilization and the instrument itself ensures 50 % relative humidity of the lubricating gas to eliminate influence of the humidity changes on it.
- Pressure in the upper chamber is adjusted by an automated control unit VLPC (very low pressure controller) that is equipped with two parallel mass flow controllers (MFC) for coarse and fine adjustment and a PC, which calculates and keeps generated pressure at a chosen value.

Theory of operation

$$dP = \frac{dF}{A(P,T)} .$$

$$dP = \frac{dF}{A} = \frac{dF}{A_{20}\left[1+\left(\alpha_p + \alpha_c\right)\left(T-20\right)\right]}.$$

Assuming linear dependence of mass comparator output (number of counts) N on an acting force:

$$K_{cal} = m_{cal}\left(1-\rho_{lcal}/\rho_{mcal}\right)g/N_{cal} .$$

Forces acting by tarring:

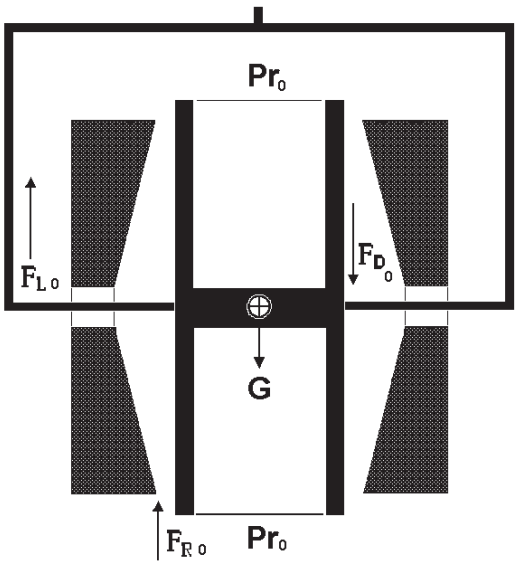
G - weight of the piston and its coupling parts,

F_{L0} - buoyancy caused by the lubricating gas in the time of tarring,

F_{R0} - buoyancy caused by the gas in chambers in the time of tarring,

F_{D0} - drag force due to the flow of the lubricating gas in the time of tarring,

P_{r0} - reference pressure in the time of tarring.



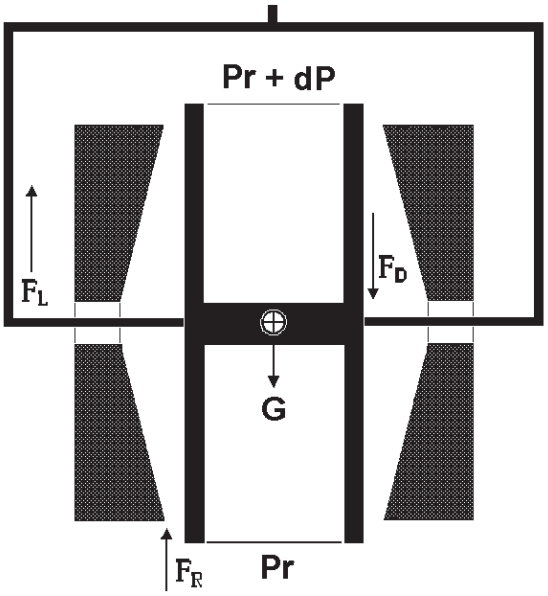
$$N_0 = \left[G + F_{L0} - F_{D0} + F_{R0}\right]/K_{cal}$$

$$N_0 = N_C + \left[F_{L0} + F_{R0} - F_{D0}\right]/K_{cal}$$

Forces acting by measurement:

The forces and pressure in the time of measurement lack zero indexes.

This convention will be kept also in the further marking of lubricating and reference pressure and reference temperature.



$$N_0' = N_C + \left[F_L + F_R - F_D\right]/K_{cal}$$

$$N_0' = N_0 + \left[F_L - F_{L0} + F_R - F_{R0} - F_D + F_{D0}\right]/K_{cal}$$

$$N_0' = N_0 - \delta N_1 - \delta N_2 - \delta N_3$$

- K_b *and* K_d are experimentally determined constants of the instrument for used piston and calibration gas.
- The last term equals zero in absolute pressure measurement.
- All three terms remain zero if the operating conditions do not change since the system was zeroed.

$$\delta N_1 = -K_b(P_l - P_{l0})$$

$$\delta N_2 = K_d\left[(P_l - P_r) - (P_{l0} - P_{r0})\right]$$

$$\delta N_3 = Vg\left[\frac{P_r}{T_r} - \frac{P_{r0}}{T_{r0}}\right]\frac{M_{gas}}{K_{cal}Z_{r0}R}$$

Assuming reference pressure P_r and pressure head P_h :

$$P_A = \frac{K_{cal}\left(N_m - N_0 + \delta N_1 + \delta N_2 + \delta N_3\right)}{A_{20}\left[1 + \left(\alpha_p + \alpha_c\right)\left(T - 20\right)\right]} + P_r + P_h,$$

where $P_h = -h\left(\rho_h - \rho_r\right)g.$

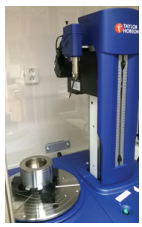
Further $\alpha_p = \alpha_c = \alpha$ and denoting $N = N_m - N_0$ we get:

$$P_A = \frac{K_{cal}\left(N + \delta N_1 + \delta N_2 + \delta N_3\right)}{A_{20}\left[1 + 2\alpha\left(T - 20\right)\right]} + P_r + P_h$$

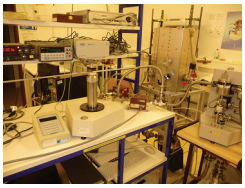
$$P_A = \frac{m_{cal}\,g}{N_{cal}}\left(1 - \frac{\rho_{lcal}}{\rho_{mcal}}\right)\frac{\left(N + \delta N_1 + \delta N_2 + \delta N_3\right)}{A_{20}\left[1 + 2\alpha\left(T - 20\right)\right]} + P_r + P_h$$

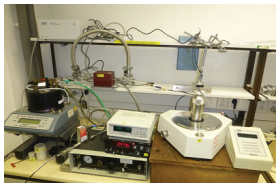
Traceability

- First it is necessary to ensure traceability of all auxiliary measurements (calibration mass, balance linearity, temperature, …) and to know the value of the local acceleration of gravity.
- The most important is to ensure the traceability of the effective area. This can be done by two ways:
  - The primary traceability through the geometrical measurements ***(next pres.)***.



  - The secondary traceability based on cross-floating with another pressure standard ***(next pres.)***.







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Thank you very much for your attention.

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Dimensional measurements and modelling of FPG and FRS at PTB

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Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Dimensional Measurements and Modeling of FPG and FRS at PTB

Ahmed Hashad, Wladimir Sabuga

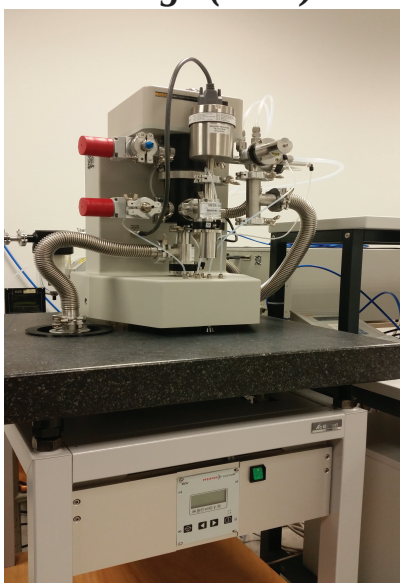
Content

- *Introduction on FPG and FRS*
- *Dimensional measurements*
- *Effective area determination*
  - *Theoretically*
- *Effective area uncertainty*
- *2-Dimentional analysis.*
- *Conclusion*



Pressure measurement principles
Non-rotating pressure balance

Force-balanced Piston Gauge (FPG)



Furness Rosenberg Standard (FRS)

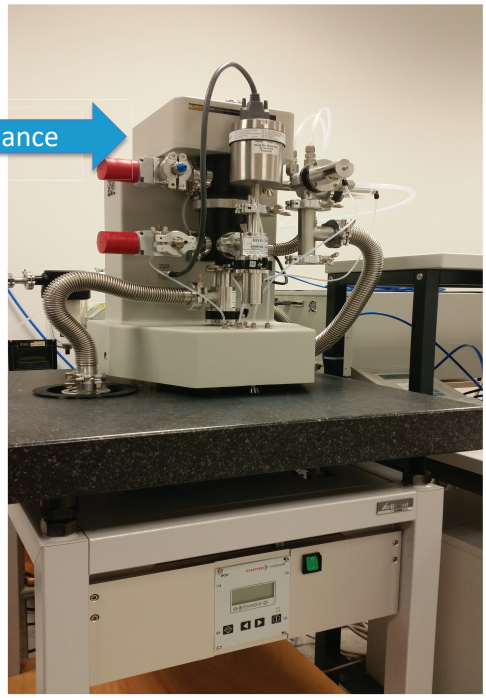


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20.02.2019 3

Pressure measurement principles
Non-rotating pressure balance

- *Force-balanced Piston Gauge (FPG).*
- *Piston mass compensation.*
- *Measurement range (1 Pa – 15 kPa).*

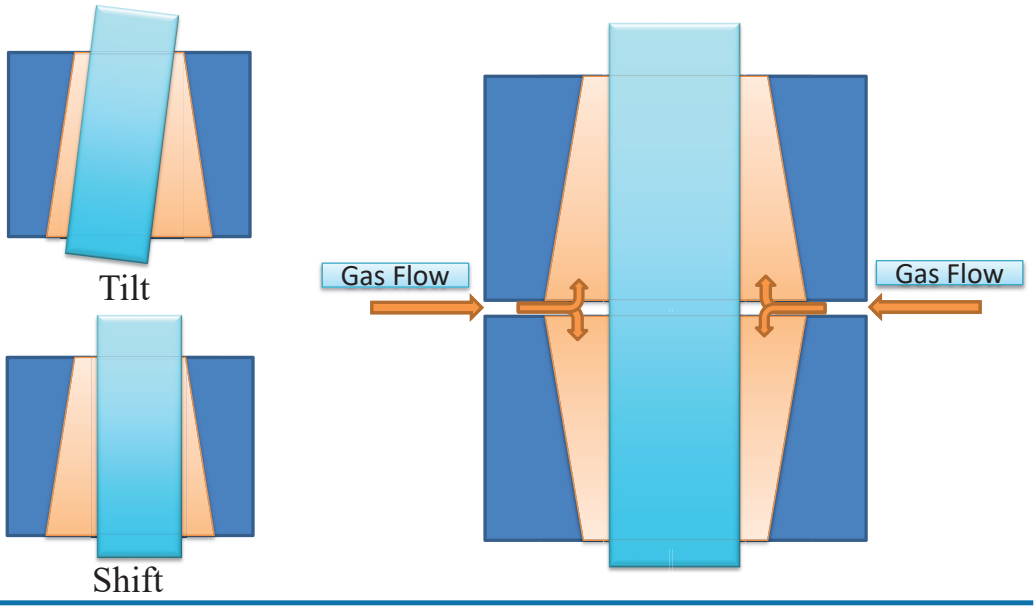


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What is the FPG ?

- The principle of operation is presented in original work by [A. Ooiwa 1986].



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Pressure calculation equations FPG

Calibration coefficient of load cell

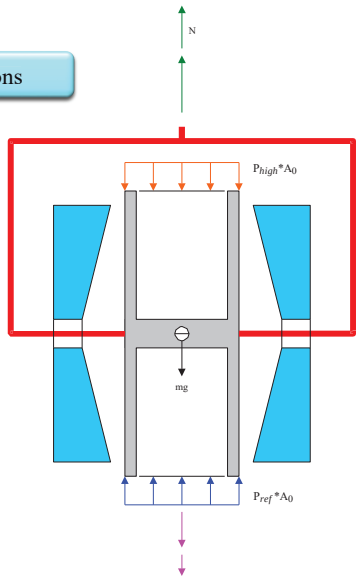
Reading

Corrections

$$\Delta P = \frac{K_{cal} \cdot (N + \delta N_1 + \delta N_2 + \delta N_3)}{A_0 \cdot \left[1 + (\alpha_p + \alpha_c) \cdot (t - 20)\right]}$$

Effective area

Thermal expansion coeff. of the piston and cylinder

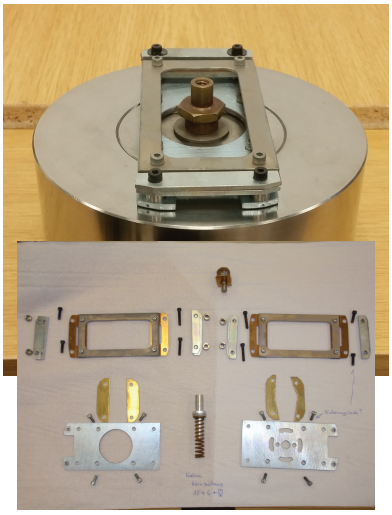


* FPG 8601 manual

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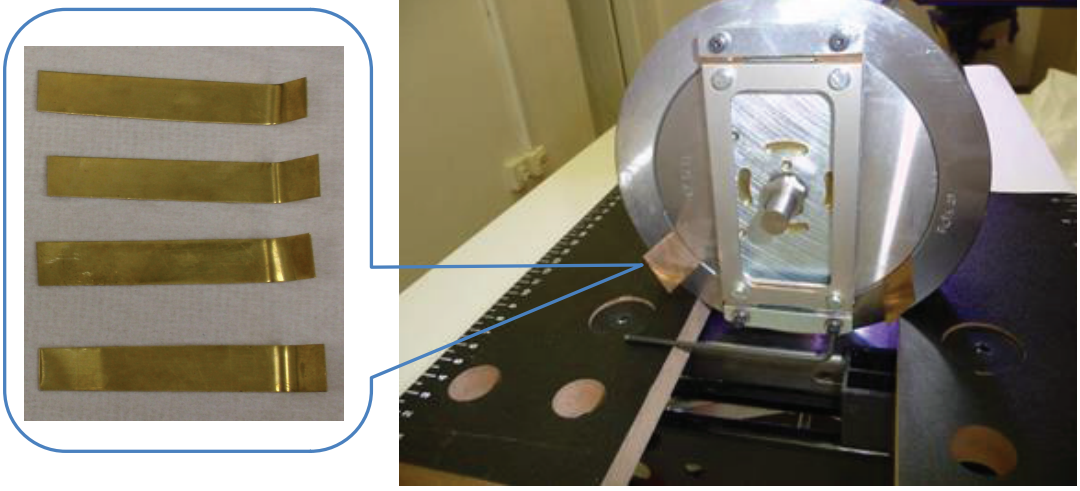
What is the FRS ?

- Furness Rosenberg Standard.
- Non-rotating piston pressure gauge with pressure range (1 Pa – 11 kPa).
- Has the same principle to determine pressure as FPG.
- Mechanical centring used to have no contact between piston-cylinder.

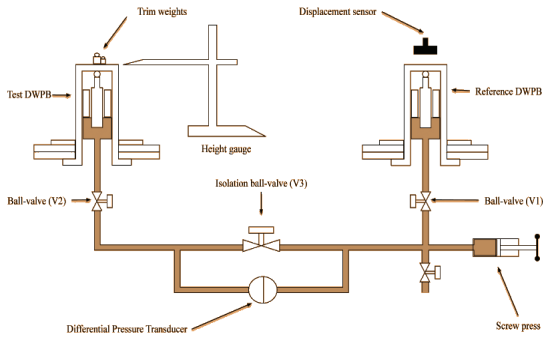


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FRS alignment procedure



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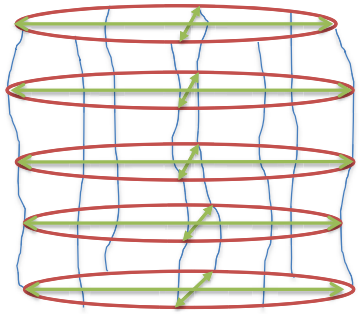
- By using cross float method with another piston gauge as reference

Secondary Standard

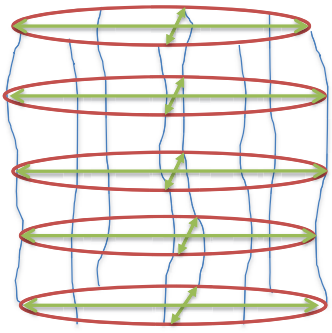
EFFECTIVE AREA DETERMINATION

- From dimensional properties of the PCA analytically.

Primary Standard



Dimensions of piston-cylinder



3D Model

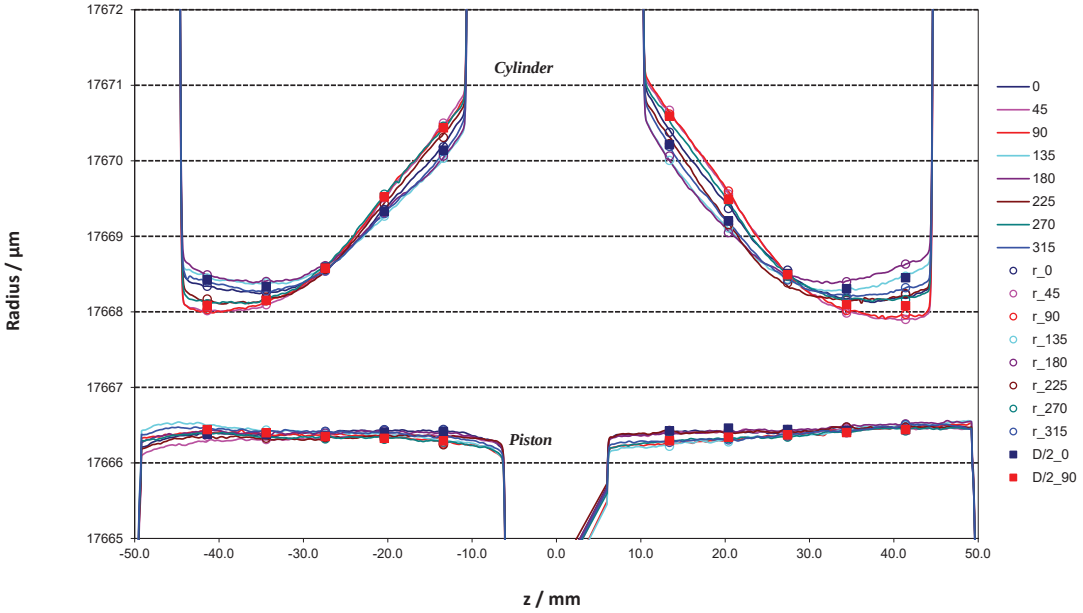
Fit of all available data using Least-Squares method

Minimum discrepancies of 3D-data ⇒ minimum uncertainties

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Dimensions of piston-cylinder

FPG radii results

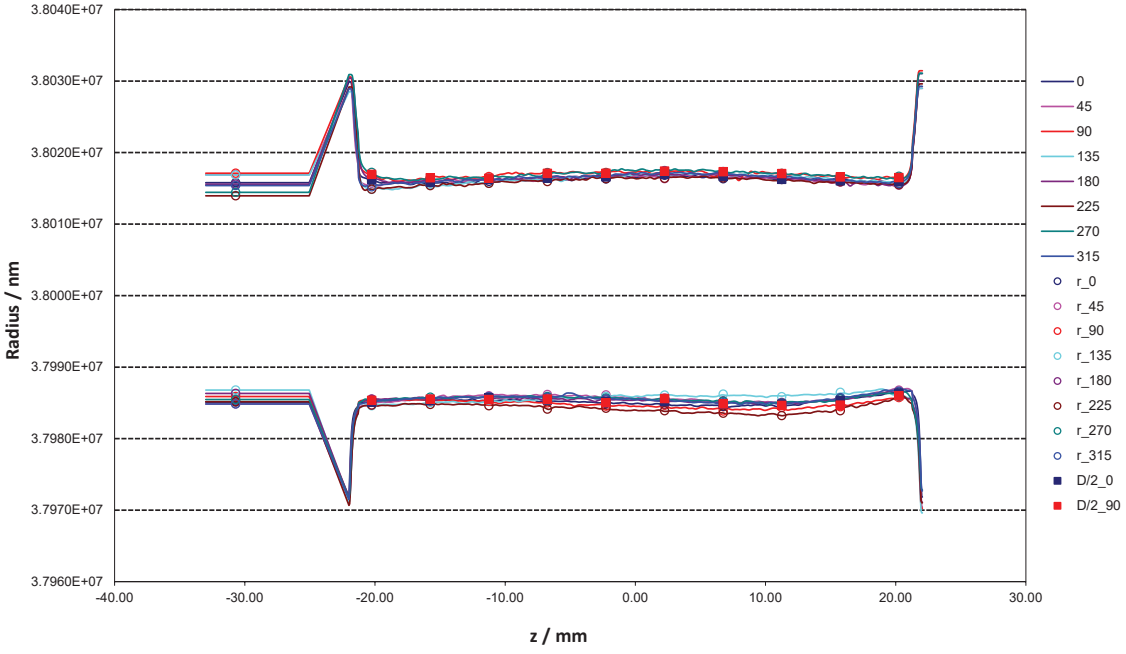


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20.02.2019 11

Dimensions of piston-cylinder

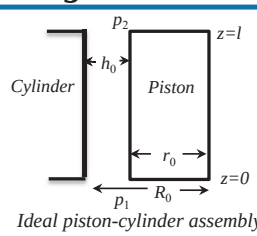
FRS radii results



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20.02.2019 12

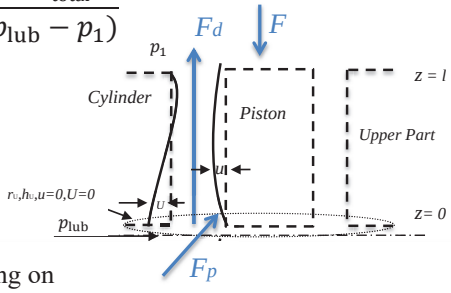
Effective area determination using dimensional properties



Ideal piston-cylinder assembly

$$A_0 = \pi {r_0}^2 \left(1 + \frac{h_0}{r_0}\right), \quad A_U = \frac{\sum F_{total}}{(p_{lub} - p_1)}$$

Dadson's theory



- F : force of measured pressure on piston base
- F_d : drag force by flow in gap
- F_p : vertical force component due to pressure in the gab acting on piston inclined surface.

From dimensional properties of PCA

$$A_U = \pi r_U^2 \left[1 + \frac{h_U}{r_U} + \frac{1}{r_U (p_{lub} - p_1)} \int_0^l (p_U(z) - p_1) \frac{d(u+U)}{dz} dz\right]$$

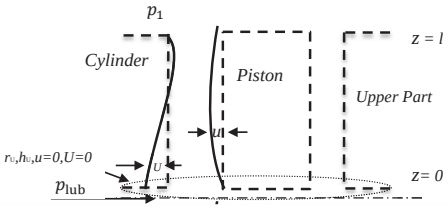
Pressure difference

Viscous flow, ideal gas

$$p_U(z) = \left[p_{lub}^2 - (p_{lub}^2 - p_1^2) \frac{\int_0^z \frac{1}{h_U^3(z)} dz}{\int_0^l \frac{1}{h_U^3(z)} dz}\right]^{1/2}$$

Effective area determination using dimensional properties

- At low pressure and narrow piston-cylinder gap, viscous flow changes to molecular one.
- Rarefied Gas Dynamics methods (e.g. BGK model) are applied.
- Gas flow rate in terms of Poiseuille coefficient (G_p) vs. rarefaction parameter (δ) determined by:

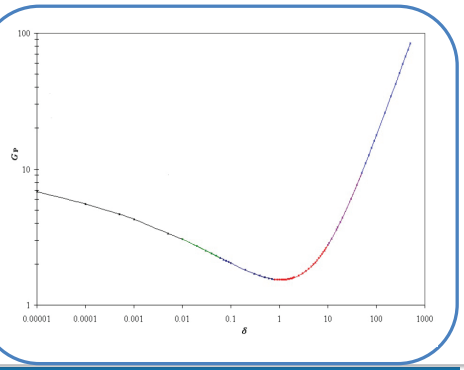


mass flow rate

Boltzmann constant

$$\dot{M} = 2\pi r h^2 (m/2kT)^{0.5} \left(-\frac{dp}{dz}\right) G_p,$$

molecular mass

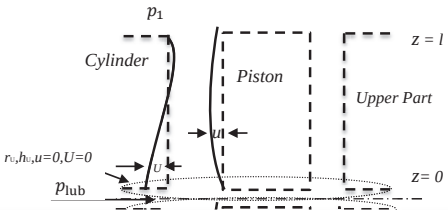


Effective area determination using dimensional properties

From continuity equation, pressure distributions in the gap calculated using:

$$p_{\mathrm{U}}(z) = \left[p_{\mathrm{lub}} - (p_{\mathrm{lub}} - p_1) \frac{\int_0^z \frac{1}{h_{\mathrm{U}}^2 G_{\mathrm{p}}(z)} dz}{\int_0^l \frac{1}{h_{\mathrm{U}}^2 G_{\mathrm{p}}(z)} dz} \right],$$

$$p_{\mathrm{L}}(z) = \left[p_{\mathrm{lub}} - (p_{\mathrm{lub}} - p_2) \frac{\int_0^z \frac{1}{h_{\mathrm{L}}^2 G_{\mathrm{p}}(z)} dz}{\int_0^l \frac{1}{h_{\mathrm{L}}^2 G_{\mathrm{p}}(z)} dz} \right],$$

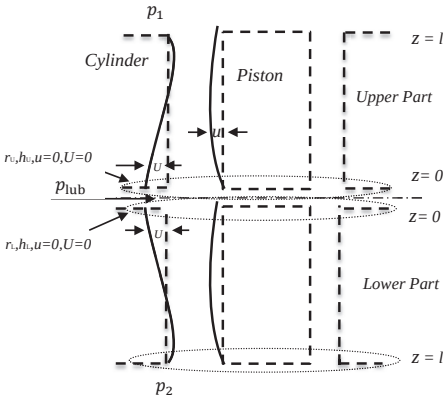


Effective area determination using dimensional properties

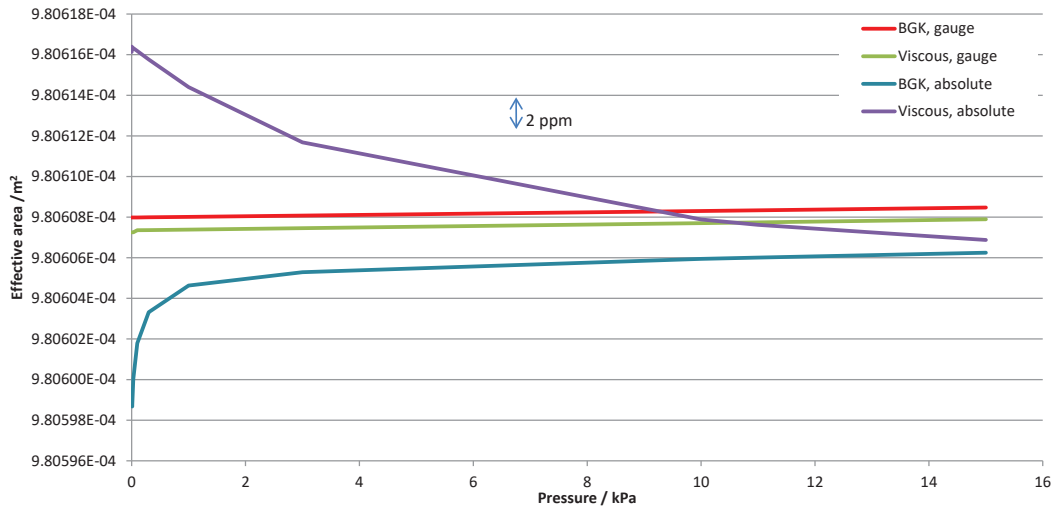
$$A_{\mathrm{U}} = \pi r_{\mathrm{U}}^2 \left[1 + \frac{h_{\mathrm{U}}}{r_{\mathrm{U}}} + \frac{1}{r_{\mathrm{U}}(p_{\mathrm{lub}} - p_1)} \int_0^l (p_{\mathrm{U}}(z) - p_1) \frac{d(u+U)}{dz} dz \right],$$

$$A_{\mathrm{L}} = \pi r_{\mathrm{L}}^2 \left[1 + \frac{h_{\mathrm{L}}}{r_{\mathrm{L}}} + \frac{1}{r_{\mathrm{L}}(p_{\mathrm{lub}} - p_2)} \int_0^l (p_{\mathrm{L}}(z) - p_2) \frac{d(u+U)}{dz} dz \right],$$

$$A = \frac{p_1 A_{\mathrm{U}} - p_2 A_{\mathrm{U0}}}{p_1 - p_2} - \frac{p_{\mathrm{lub}}}{p_1 - p_2} (A_{\mathrm{U}} - A_{\mathrm{U0}}).$$



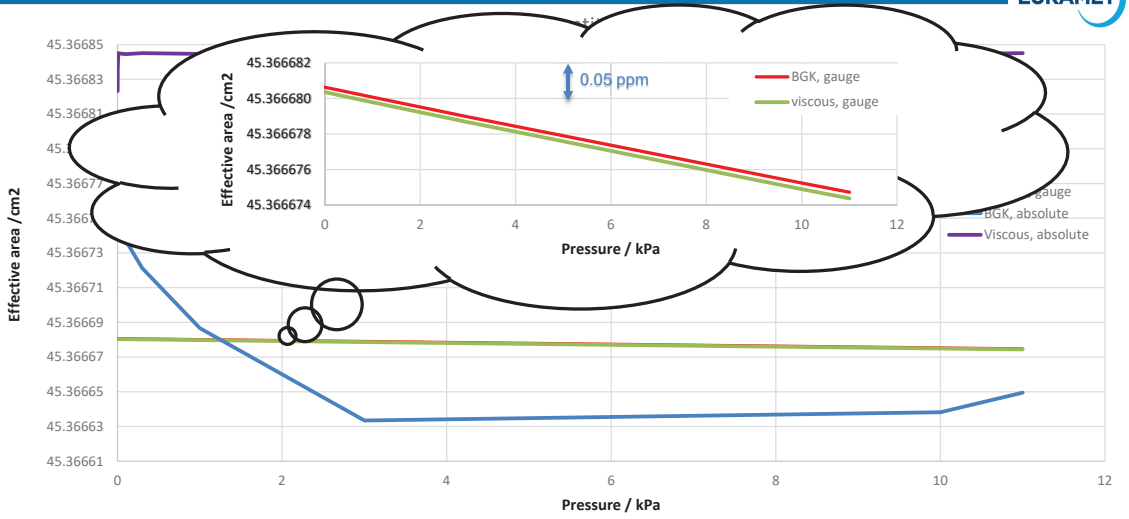
Effective area determination using dimensional properties



The difference in gauge mode between the BGK model and Viscous Flow model found to be lower than 1 ppm.

But the difference in absolute mode found to be 20 ppm at the lower pressure range.

Effective area determination using dimensional properties



The difference in absolute mode found to be 5 ppm.

The difference in gauge mode between the BGK model and Viscous Flow model found to be lower than 0.05 ppm.

Effective area determination using dimensional properties

Big difference between absolute pressure effective areas calculated by viscous and BGK models shows that the viscous flow model is inaccurate in this pressure mode.

Effective areas with their standard uncertainties by BGK are

in absolute mode: $A = 9.8060842 \times (1 \pm 3.2 \cdot 10^{-6})\ \mathrm{cm}^2$

in gauge mode: $A = 9.8060837 \times (1 \pm 3.2 \cdot 10^{-6})\ \mathrm{cm}^2$

Absolute and gauge mode areas are identical within their uncertainties!

2-Dimensional Flow Modelling

Why is 2D modelling required?

- 1D modelling assumes piston-cylinder to be axially symmetric
- 1D modelling is performed separately for each angular direction resulting in mean $<A_0>$ and its standard deviation $s(A_0)$
- FPG and FRS piston-cylinders are dimensionally much more irregular than classical PCAs $\Rightarrow$ $s(A_0)$ is relatively high

Typical uncertainties of 1D modelling ($k = 1$)

Uncertainty source	Contribution to A_0 in ppm
Uncertainty of dimensional data	3.2
Model uncertainty	< 0.2
Typical standard deviation $s(A_0)$	8.8
Combined uncertainty	**9.4**

2D modelling removes the uncertainty contribution of PCA's axial non-symmetry

2-Dimensional Flow Modelling: Theory

Continuity equation in a 2D gap:

$$\frac{\partial}{\partial z}\left(h^3\frac{\rho}{\eta}\frac{\partial p}{\partial z}\right)+\frac{\partial}{\partial t}\left(h^3\frac{\rho}{\eta}\frac{\partial p}{\partial t}\right)=0$$

2D continuity equation for gas medium (ideal gas law) :

$$\frac{\partial^2 p^2}{\partial z^2}+\frac{\partial^2 p^2}{\partial t^2}+\frac{3}{h}\frac{\partial h}{\partial z}\frac{\partial p^2}{\partial z}+\frac{3}{h}\frac{\partial h}{\partial t}\frac{\partial p^2}{\partial t}=0$$

2-Dimensional Flow Modelling: Numerical approach

Finite Difference Method

Derivative	Approximation
$\frac{\partial p^2(z,t)}{\partial z}$	$\frac{p^2(z+\Delta z,t)-p^2(z-\Delta z,t)}{2\Delta z}$
$\frac{\partial p^2(z,t)}{\partial t}$	$\frac{p^2(z,t+\Delta t)-p^2(z,t-\Delta t)}{2\Delta t}$
$\frac{\partial^2 p^2(z,t)}{\partial z^2}$	$\frac{p^2(z+\Delta z,t)-2p^2(z,t)+p^2(z-\Delta z,t)}{\Delta z^2}$
$\frac{\partial^2 p^2(z,t)}{\partial t^2}$	$\frac{p^2(z,t+\Delta t)-2p^2(z,t)+p^2(z,t-\Delta t)}{\Delta t^2}$

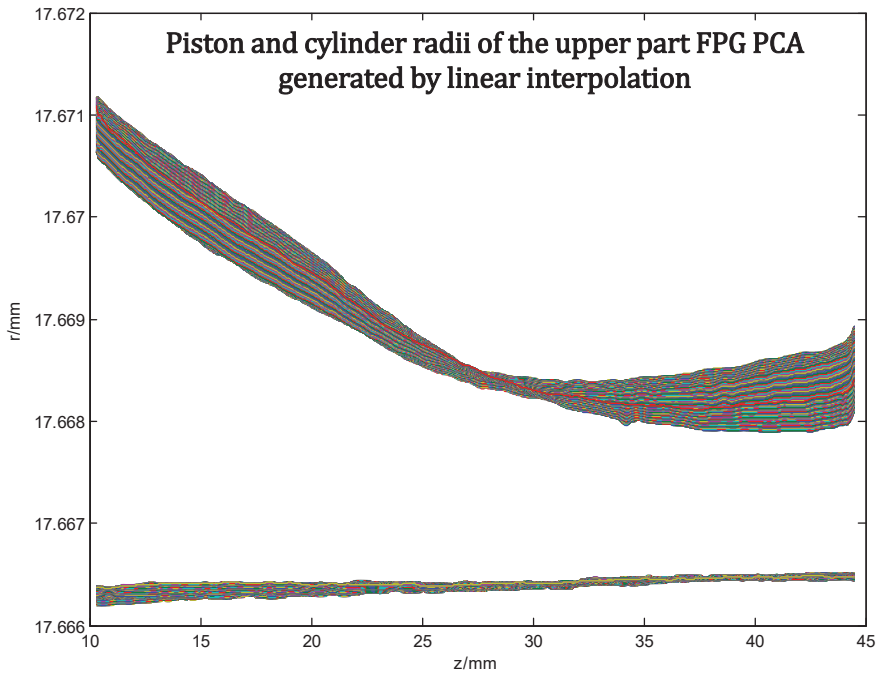
Iterative solution for pressure distribution

$$p(z,t)=\frac{1}{2(1+k^2)}\left[\left(1+\frac{3\Delta z}{2h}\frac{\partial h}{\partial z}\right)\cdot p^2(z+\Delta z,t)+\left(1-\frac{3\Delta z}{2h}\frac{\partial h}{\partial z}\right)\cdot p^2(z-\Delta z,t)\right.$$
$$\left.+k^2\left(1+\frac{3\Delta z}{2kh}\frac{\partial h}{\partial t}\right)\cdot p^2(z,t+\Delta t)+k^2\left(1-\frac{3\Delta z}{2kh}\frac{\partial h}{\partial t}\right)\cdot p^2(z,t-\Delta t)\right]$$

with $k=\Delta z/\Delta t$

2-Dimensional Flow Modelling: Dimensional data processing

2D modelling requires similar density of dimensional data along axial and tangential coordinates ⇒ linear interpolation applied to both straightness and roundness data

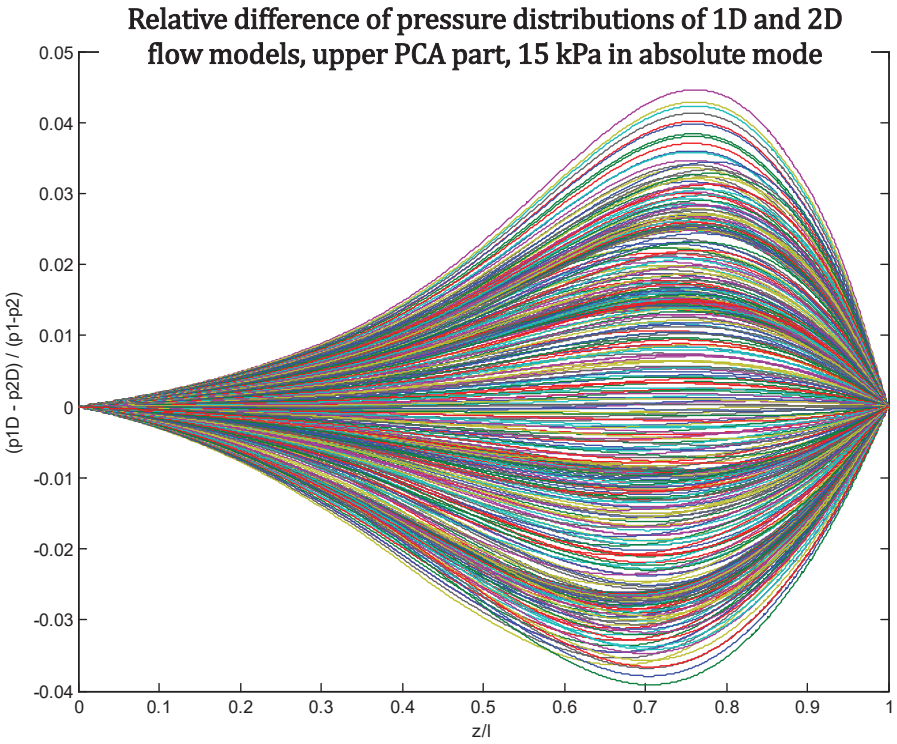


1- and 2-Dimensional Flow Modelling: Main questions to be answered

- Uncertainty due to numerical procedures
- Difference in calculations based on straightness and roundness data
- Difference between absolute and gauge mode effective areas
- Difference for real and ideal gas properties (viscous flow)
- Difference for viscous and RGD models
- Change of effective area with pressure
- Mean and standard deviation of 1D flow calculations performed for different angular directions, for straightness and roundness data
- Difference between mean effective area of 1D flow model and effective area for 2D flow model (viscous flow, ideal gas, straightness and roundness data)

Contribution of PCA's axial non-symmetry

2-Dimensional Flow Modelling: Results



2-Dimensional Flow Modelling: Estimation of effects and uncertainties

Effect or uncertainty source	Contribution to A_0 in ppm
Numerical procedures	0.05
Use of straightness vs. roundness data (included in uncertainty of dim. data)	1.2
Absolute vs. gauge mode	0.76
Real vs. ideal gas properties, viscous flow	0.16
Viscous vs. RGD model	4.2
Change with pressure	0.87
Axial non-symmetry effect of 1D model, max. std. dev.	12
2D model vs. angular mean of 1D model	0.25
Uncertainty of dimensional data	3.2
Combined uncertainty	**3.2**

Conclusion

1) *Calculation of the effective area was performed by viscous, 1D and 2D flow, and BGK models. The viscous flow model was found inaccurate at low pressures in absolute mode*

2) *The effective area was found to be*
$9.8060842 \times (1 \pm 3.2 \cdot 10^{-6})\ cm^2$ *in absolute mode*
$9.8060837 \times (1 \pm 3.2 \cdot 10^{-6})\ cm^2$ *in gauge mode – practically the same in both modes*

3) *Full agreement was found between the experimental and theoretical effective areas calculated using BGK model, in both absolute and gauge modes*

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THANK YOU, ANY QUESTIONS ?

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Gas flow modelling and calculation of the effective area of force-balanced piston gauges FPG and FRS

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GAS FLOW MODELING AND CALCULATION OF THE EFFECTIVE AREA OF FORCE-BALANCED PISTON GAUGES FPG AND FRS

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Presentation contents

- Introduction
- Computation of effective area
  - Effective area of a piston-cylinder assembly
  - Total effective area of a piston gauge
  - Gas flow modeling tools determining the pressure distribution
- Results and discussion (three FPG8601, one FRS4 and one FRS5)
  - Total effective area
  - Theoretical uncertainties
- Concluding remarks

Introduction

- Recently, great efforts are made to establish fundamental standards for pressure measurements in the range of approximately 1 Pa to 15 kPa.
- Promising devices that could be used as primary standards are non-rotating piston gauges, such as the Force-balanced Pressure Gauge (FPG) or the Furness-Rosenberg Standard (FRS).
- These instruments are widely used by National Metrology Institutes (NMIs) and accredited calibration laboratories.
- However, their traceability in Europe is based on calibration against other primary standards above 5 kPa.

Introduction

- In previous years, comparisons between NMIs were mostly targeting at the statement of the equivalence between pressure standards [Krazicek 2014].
- Very few attempts have been made to characterize FPG and FRS gauges as primary pressure standards [Tesar 2007, Hashad 2017].
- In this work, a detailed study for the effective area of three FPG8601, one FRS4 and one FRS5 assemlies, along with their associated theoretical uncertainties is provided.
- Data were provided by PTB, RISE and INRIM for the FPG8601 and by CMI and PTB for the FRS4 and FRS5 respectively.
- The motivation behind this work is to determine whether the FPG and FRS piston-gauges can be used as primary pressure standards with an effective area uncertainty below 15×10^{-6}.

PCA effective area computation

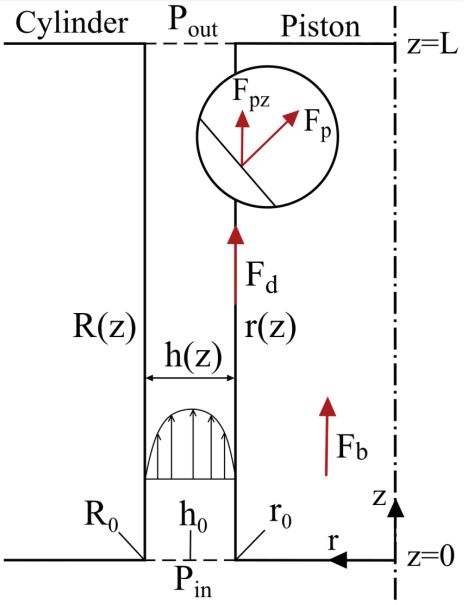


Fig. 1: Schematic representation of a PCA cross-section.

The effective area of a PCA is defined as:

$$A_{eff} = \frac{F}{\Delta P} = \frac{F_b + F_d + F_{pz}}{P_{in} - P_{out}}$$

- F_b: Base force due to pressure difference
- F_d: Drag force due to lubricating gas flow
- F_{pz}: Vertical component of the pressure force acting normal to the piston

The dimensional and operational data of a PCA are:

- P_{in} : Pressure at gap inlet
- P_{out}: Pressure at gap outlet
- $r(z)$: Piston radius
- $R(z)$: Cylinder radius
- $h(z)$: Gap width
- r_0: Inlet piston radius
- R_0: Inlet cylinder radius
- h_0: Inlet gap width

PCA effective area computation

- The investigated piston-gauges consist of two parts. The effective area for a PCA part is given by [Dadson 1982]:
- Top part:

$$A_T = \pi r_T^2 \left(1 + \frac{h_{0,T}}{r_{0,T}} - \frac{1}{r_{0,T}\left(P_{in,T} - P_{out,T}\right)} \int_0^{L_T} \frac{dP_T}{dz_T} \left(\left(R_T - R_{0,T}\right) + \left(r_T - r_{0,T}\right)\right) dz_T\right)$$

- Bottom part:

$$A_B = \pi r_B^2 \left(1 + \frac{h_{0,B}}{r_{0,B}} - \frac{1}{r_{0,B}\left(P_{in,B} - P_{out,B}\right)} \int_0^{L_B} \frac{dP_B}{dz_B} \left(\left(R_B - R_{0,B}\right) + \left(r_B - r_{0,B}\right)\right) dz_B\right)$$

- It is noted that when the dimensional and operational data are known only the pressure distribution is required for the effective area computation.

FPG8601 total effective area

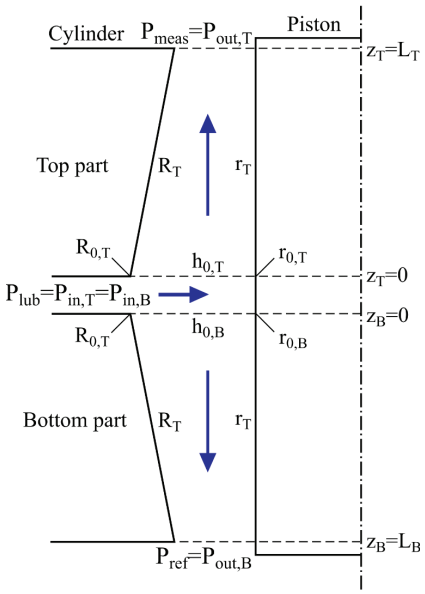


Fig. 2: Schematic representation of an FPG8601 cross-section.

- Total effective area of FPG8601:

$$A_{eff} = \frac{F_B - F_T - F_0}{P_{meas} - P_{ref}}$$

  - F_T: Total force acting on the top part
  - F_B: Total force acting on the bottom part
  - F_0: Remaining force from load cell zeroing

- Introducing the respective effective areas the total effective area reduces to

$$A_{eff} = \frac{P_{meas}A_T - P_{ref}A_{T,0}}{P_{meas} - P_{ref}} - \frac{P_{lub}(A_T - A_{T,0})}{P_{meas} - P_{ref}}$$

- It is noted that the bottom part effective area does not contribute to the overall effective area.

FRS4/5 total effective area

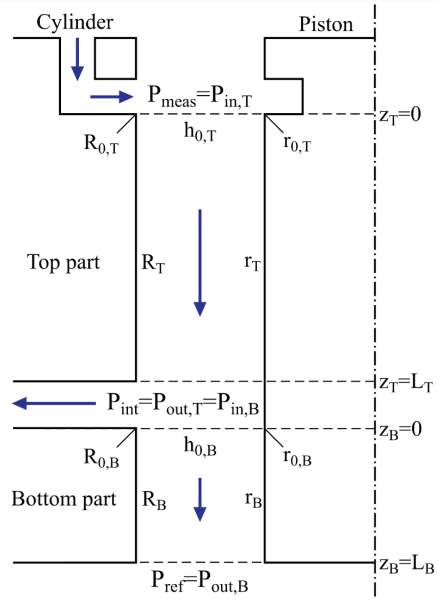


Fig. 3: Schematic representation of an FRS4/5 cross-section.

- Total effective area of FRS4/5:

$$A_{eff} = \frac{F_B + F_T}{P_{meas} - P_{ref}}$$

  - F_T: Total force acting on the top part
  - F_B: Total force acting on the bottom part

- In the same manner the total effective area is expressed as

$$A_{eff} = A_T + \frac{P_{int} - P_{ref}}{P_{meas} - P_{ref}}(A_B - A_T)$$

Pressure distribution determination

To determine the pressure distribution three methodologies have been used:

- 1D axisymmetric **kinetic** approach [Breyiannis 2008]
  - A robust kinetic database for the dimensionless flow rate G has been created.
  - The pressure distribution ordinary differential equation is solved iteratively upon convergence of the outlet pressure:

$$\frac{dP_i}{dz_i} = \frac{\dot{M}_i u_0}{2\pi G h_i (R_i^2 - r_i^2)}$$

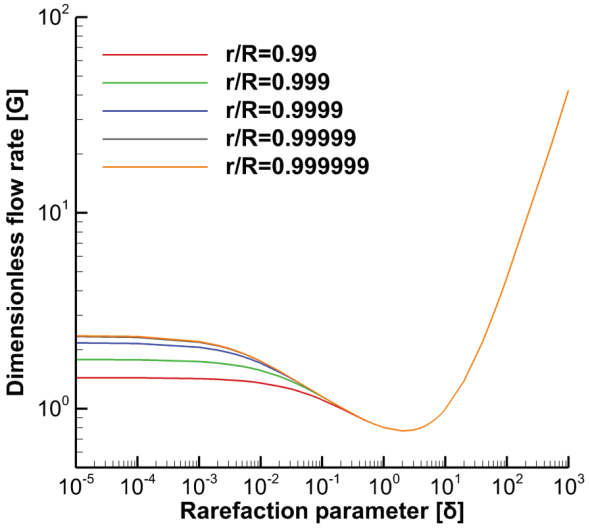


Fig. 4: Flow rate G between coaxial cylinders in terms of δ for various r/R.

Pressure distribution determination

- 1D planar viscous approach [Dadson 1982]
  - The pressure distribution for the compressible Poiseuille flow of an ideal-gas between plates is given in a closed form :

$$P_i(z) = \left[P_{in,i}^2 - (P_{in,i}^2 - P_{out,i}^2) \frac{\int_0^{z_i} h_i^{-3} dz_i}{\int_0^{L_i} h_i^{-3} dz_i} \right]^{1/2}$$

- 2D axisymmetric viscous approach (CFD Software)
  - The mass, momentum and energy conservation equations of a compressible ideal-gas are solved based on the provided dimensional and operational data.
  - The pressure distribution along the piston surface is extracted.
  - The pressure profile is used to compute the effective area.
- It is noted that both viscous approaches are useful only in the continuum regime, while the kinetic approach is valid in the whole range of pressure under investigation.

FPG8601 simulation data

- Gas inside the FPG8601 gap: dry Nitrogen at 20 °C.
- Gauge operation mode simulation pressure:
  - Measured: $100 - 115$ kPa $(\delta{\sim}35 - 40)$
  - Lubrication: 140 kPa $(\delta{\sim}160)$
  - Reference: 100 kPa $(\delta{\sim}40)$
- Absolute operation mode simulation pressure:
  - Measured: 1 Pa $- 15$ kPa $(\delta{\sim}0.0004 - 5)$
  - Lubrication: 40 kPa $(\delta{\sim}45)$
  - Reference: 1 Pa $- 10$ Pa $(\delta{\sim}0.0004 - 0.004)$
- Effective areas are presented with respect to P_{op} for uniformity between the gauge (P_{op}= 100 kPa) and absolute (P_{op} = 0 Pa) modes.

FPG8601 dimensional data

Table 1: Dimensional measurement data provided by PTB, RISE, and INRIM for the FPG6801.

FPG8601 Part	Top			Bottom		
FPG8601 device	PTB	RISE	INRIM	PTB	RISE	INRIM
Measurement angles	8	8	4	8	8	4
No axial Points	343	317	69	339	346	69
Engagement length [mm]	34.2	31.6	34.0	33.8	34.5	34.0
Average gap inlet [µm]	4.646	3.889	4.238	4.414	3.784	4.131
Average gap width [µm]	2.511	2.417	2.474	2.551	2.490	2.486
Average gap outlet [µm]	2.013	1.015	1.087	2.155	1.553	0.922
Average piston area [cm2]	9.80496	9.80431	9.80433	9.80494	9.80433	9.80432

FPG8601 dimensional data

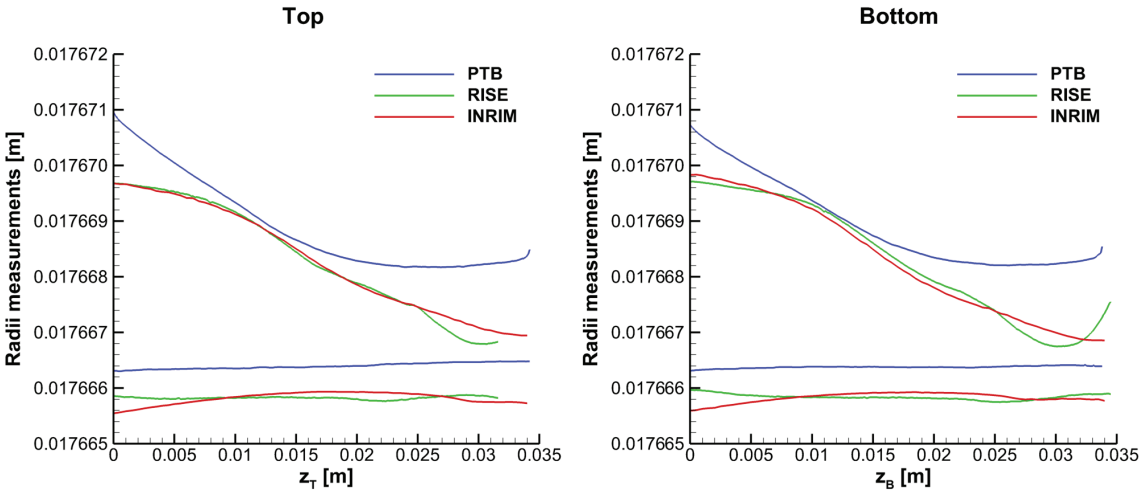


Fig. 5: Piston and cylinder radii measurements provided by PTB, RISE, and INRIM for the FPG8601 averaged over all angles.

FPG8601 effective area (PTB)

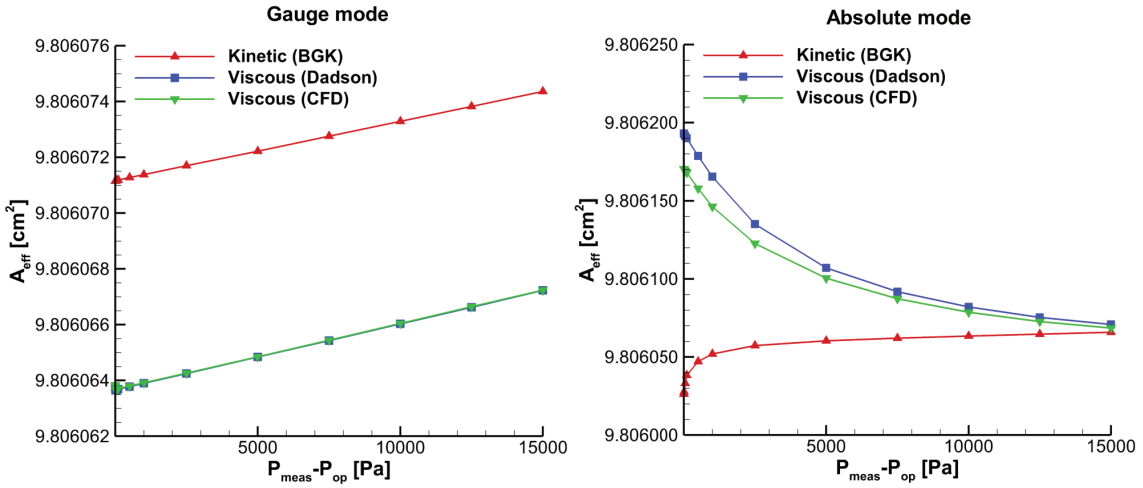


Fig. 6: Computed effective area averaged over all angles for the gauge and absolute modes.

FPG8601 effective area (RISE)

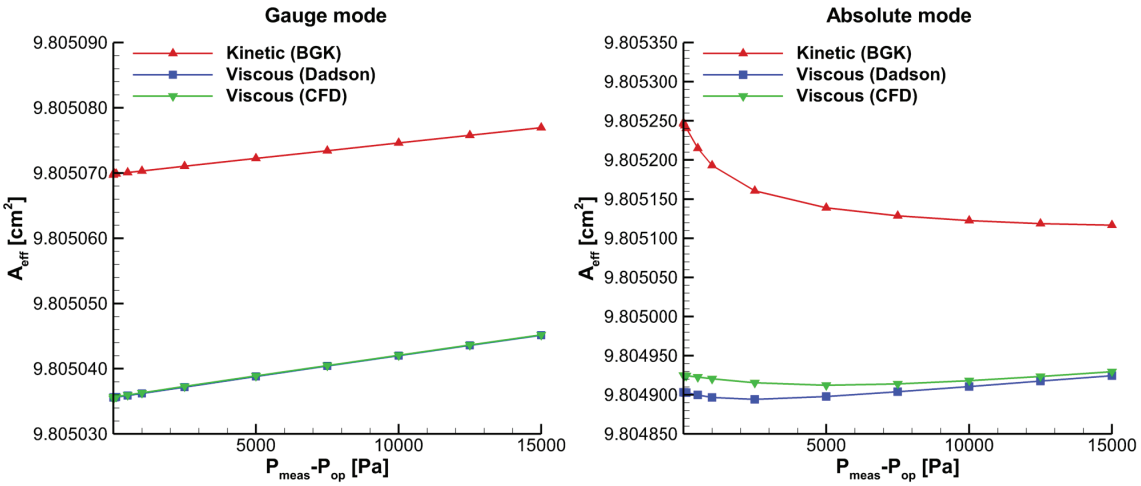


Fig. 7: Computed effective area averaged over all angles for the gauge and absolute modes.

FPG8601 effective area (INRIM)

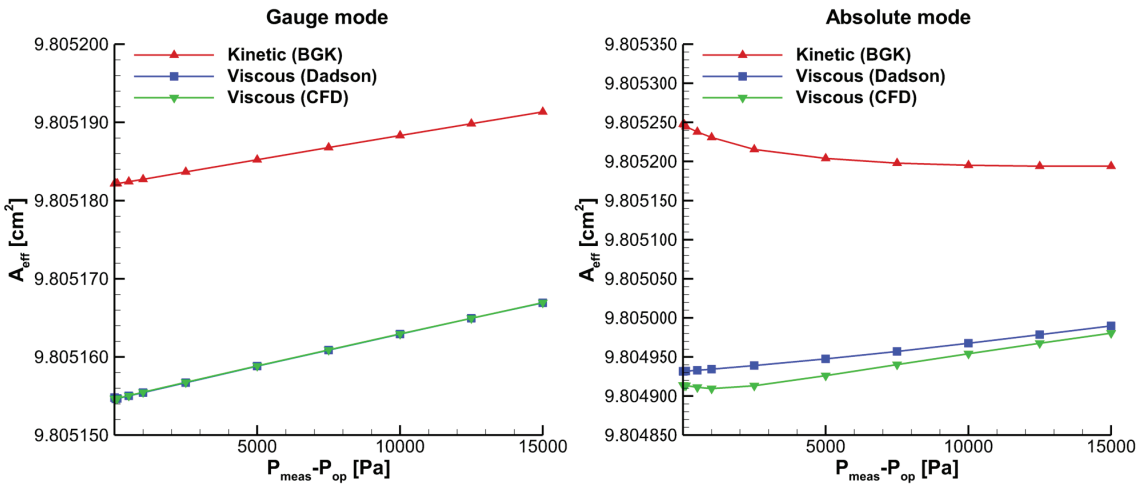


Fig. 8: Computed effective area averaged over all angles for the gauge and absolute modes.

FRS4 simulation data

Gas inside the FRS4 gap: dry Nitrogen at 20 °C.

Gauge operation mode simulation pressure:

- Measured: $100 - 103.2$ kPa $(\delta{\sim}850 - 880)$
- Intermediate: 100 kPa $(\delta{\sim}850)$
- Reference: 100 kPa $(\delta{\sim}850)$

Absolute operation mode simulation pressure:

- Measured: 1 Pa $- 3.2$ kPa $(\delta{\sim}1 \cdot 10^{-2} - 30)$
- Intermediate : 0.01 Pa $(\delta{\sim}1 \cdot 10^{-4})$
- Reference: 0.01 Pa $(\delta{\sim}1 \cdot 10^{-4})$

- Even though the FRS4 owned by CMI operates only on gauge mode, simulations have been performed also for the absolute mode.

FRS4 dimensional data

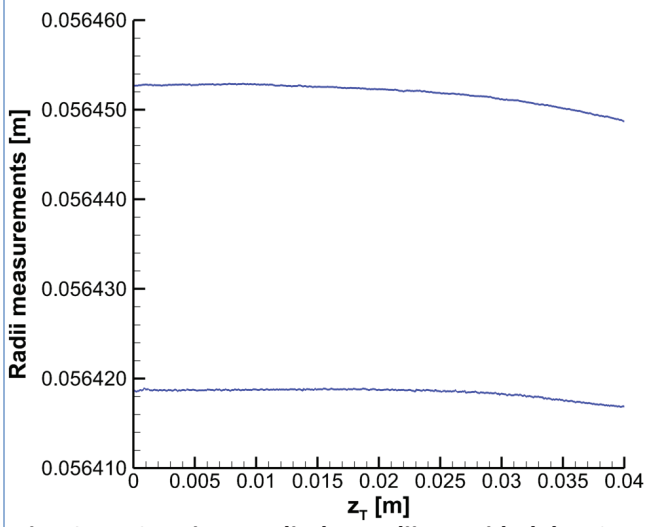


Fig. 9: FRS4 piston-cylinder radii provided by CMI averaged over all angles.

Table 2: FRS4 Dimensional data.

FRS part	Top
Measurement angles	8
No axial points	2000
Engagement length [mm]	40.00
Average gap inlet [μm]	34.038
Average gap width [μm]	33.412
Average gap outlet [μm]	31.825
Average piston area [cm^2]	100.0015

FRS4 effective area (CMI)

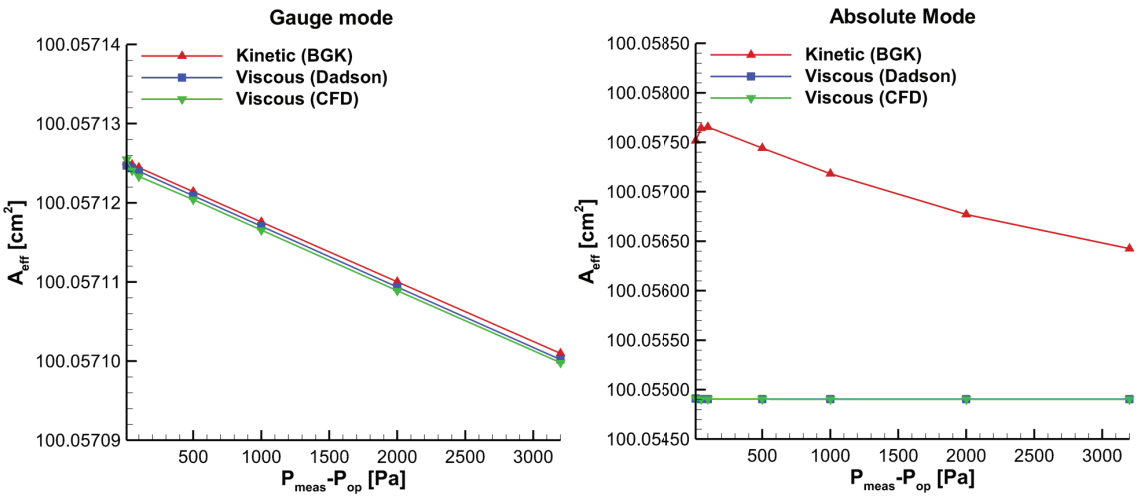


Fig. 10: Computed effective area averaged over all angles for the gauge and absolute modes.

FRS5 simulation data

- Gas inside the FRS5 gap: dry Nitrogen at 20 °C.
- Gauge operation mode simulation pressure:

P_{meas} [kPa]	100-111
P_{int} [kPa]	100
P_{ref} [kPa]	100
δ	850-950

- Absolute operation mode simulation pressure:

P_{meas} [Pa]	10-100	100-1000	1000-10000	10000-11000
P_{int} [Pa]	0.03	0.5	2	20
P_{ref} [Pa]	0.0003	0.005	0.01	0.02
δ	2×10^{-6}-0.85	4×10^{-5}-8.5	9×10^{-5}-85	2×10^{-4}-95

FRS5 dimensional data

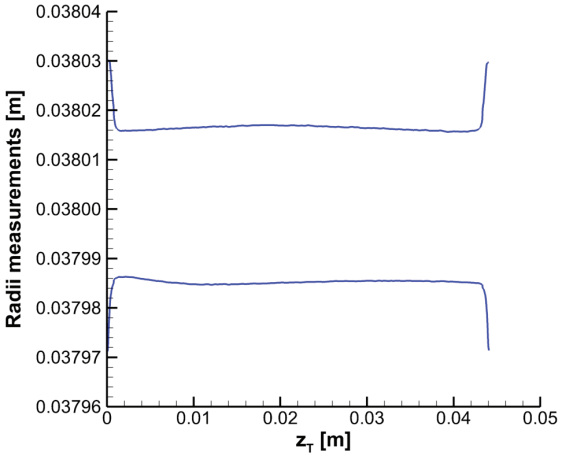


Fig. 11: FRS5 piston-cylinder radii provided by PTB averaged over all angles.

Table 3: FRS5 Dimensional data.

FRS assembly	FRS5	
FRS part	Top	Bottom
Measurement angles	8	8
No axial points	882	1
Engagement length [mm]	44.05	7.95
Average gap inlet [µm]	58.842	30.200
Average gap width [µm]	31.683	30.200
Average gap outlet [µm]	58.287	30.200
Average piston area [cm2]	45.32902	45.33012
Diameter Uncertainty [nm]	25	25

FRS5 effective area (PTB)

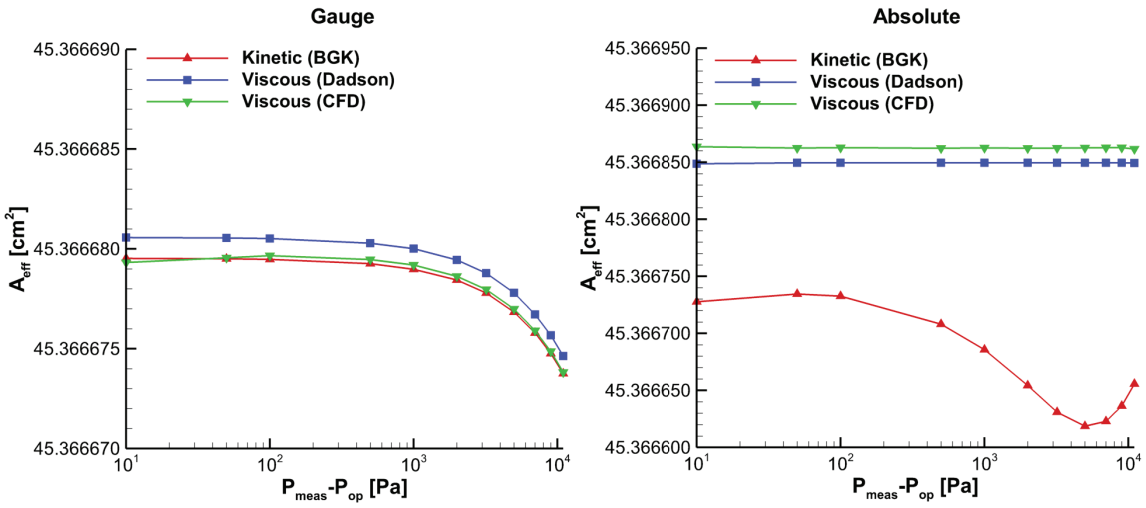


Fig. 12: Computed effective area averaged over all angles for the gauge and absolute modes.

Theoretical uncertainty (dimensional)

- Uncertainty of effective area due to dimensional uncertainties [Sharipov 2016]:

$$\frac{u_d(A_{eff})}{A_{eff}} = \frac{\sqrt{u_p^2 + u_c^2}}{2R_m}$$

u_p : St. abs. piston diameter uncertainty
u_c : St. abs. cylinder diameter uncertainty
R_m : Average piston and cylinder radius

Table 4: Standard absolute dimeter uncertainties provided for all PCAS.

PCA	$u_p\ [nm]$	$u_c\ [nm]$	$R_m\ [mm]$
FPG8601 (PTB)	25	25	17.66765
FPG8601 (RISE)	200	200	17.66708
FPG8601 (INRIM)	26	26	17.66706
FRS4 (CMI)	500	500	56.43512
FRS5 (PTB)	25	25	38.00094

Theoretical uncertainty (accommodation)

- The accommodation coefficient characterizes the gas-surface interaction.
- For typical mechanical surfaces and heavy gases (N_2) it takes values larger than 0.8.
- To estimate the introduced error simulations were performed for different values of α.
- Accommodation coefficient uncertainty:

$$\frac{u_a(A_{eff})}{A_{eff}} = \frac{A_{eff}(a=1) - A_{eff}(a)}{A_{eff}(a=1)}$$

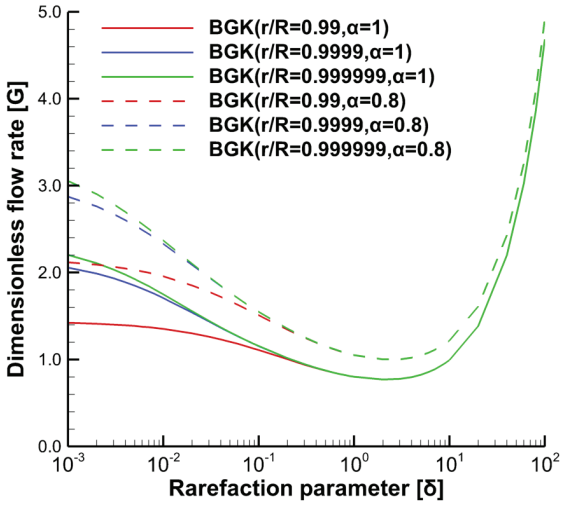


Fig. 13: Flow rate G between coaxial cylinders in terms of δ for various r/R and α.

Theoretical uncertainty (kinetic model)

- Kinetic models such as the BGK replace the original Boltzmann equation collision operator to reduce computation effort.
- To estimate the introduced error simulations were performed based on the LBE.
- Kinetic model uncertainty:

$$\frac{u_a(A_{eff})}{A_{eff}} = \frac{A_{eff}(BGK) - A_{eff}(LBE)}{A_{eff}(BGK)}$$

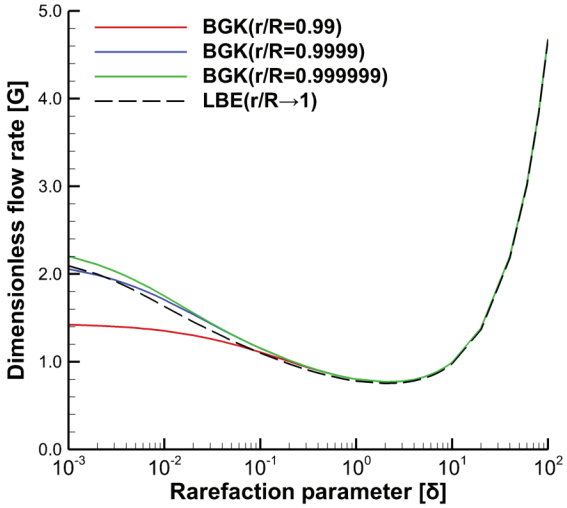


Fig. 14: Flow rate G between coaxial cylinders in terms of δ for various r/R and α=1.

Theoretical uncertainty (temperature, humidity)

- The gas flow is subject to temperature fluctuations affecting the gas properties.
  - Temperature uncertainty: $\frac{u_t(A_{eff})}{A_{eff}} = \frac{A_{eff}(T=20\ ^oC) - A_{eff}(T)}{A_{eff}(T=20\ ^oC)}$
- The gas supply has a relative humidity around 50% which adds to the flow uncertainty.
  - Humidity uncertainty: $\frac{u_h(A_{eff})}{A_{eff}} = \frac{A_{eff}(\text{dry } N_2) - A_{eff}(50\%\ \text{humid } N_2)}{A_{eff}(\text{dry } N_2)}$
- Total uncertainty defined as the Euclidean norm of all investigated uncertainties.

Theoretical uncertainty results

Table 5: Theoretical uncertainty in the gauge mode for all PCAs at maximum operation pressure

PCA	FPG8601 (PTB)	FPG8601 (RISE)	FPG8601 (INRIM)	FRS4 (CMI)	FRS5 (PTB)
Dimensional uncertainty	1.00	8.00	1.04	6.26	0.47
Acc. Coef. (α=0.9)	0.11	0.47	0.38	0.00	0.00
Acc. Coef. (α=0.8)	0.27	1.02	0.81	0.01	0.00
Gas Temperature (±1 °C)	0.00	0.01	0.01	0.00	0.00
Gas Temperature (±2 °C)	0.01	0.02	0.02	0.00	0.00
Model equation (BGK)	0.03	0.11	0.09	0.00	0.00
Relative Humidity (50%)	0.00	0.01	0.01	0.00	0.00
Total uncertainty	1.04	8.07	1.32	6.26	0.47

Theoretical uncertainty results

Table 6: Theoretical uncertainty in the absolute mode for all PCAs at maximum operation pressure

PCA	FPG8601 (PTB)	FPG8601 (RISE)	FPG8601 (INRIM)	FRS4 (CMI)	FRS5 (PTB)
Dimensional uncertainty	1.00	8.00	1.04	6.26	0.47
Acc. Coef. (α=0.9)	0.35	1.20	1.13	0.75	0.17
Acc. Coef. (α=0.8)	0.75	2.44	2.31	1.60	0.30
Gas Temperature (±1 °C)	0.01	0.05	0.05	0.02	0.01
Gas Temperature (±2 °C)	0.03	0.11	0.10	0.04	0.02
Model equation (BGK)	0.05	0.25	0.19	0.17	0.03
Relative Humidity (50%)	0.01	0.04	0.04	0.02	0.01
Total uncertainty	1.25	8.38	2.55	6.47	0.56

Concluding remarks

- The effective area of three FPG8601, one FRS4 and FRS5 has been computed based on kinetic theory; Dadson theory and CFD software have been used as complimentary modeling tools in the viscous regime.

- Computation of effective areas showed:
  - Excellent agreement between UTH and PTB simulations.
  - Dadson, CFD and kinetic results show excellent agreement in the viscous limit.
  - Kinetic and viscous results deviate significantly in the absolute mode.
  - In gauge mode the effective area of all PCAs is almost constant.
  - In absolute mode no trend of the effective area has been observed.

- Investigation of the involved theoretical uncertainties showed:
  - Largest uncertainty contribution due to dimensional measurements
  - Significant uncertainty due to gas-surface interaction.
  - Negligible effect of model equation, gas temperature and humidity.

- The total uncertainty is below the target uncertainty of 15 ppm at maximum operation pressure for all PCAs.

Experimental study of PTB FPG as secondary pressure standard

Wladimir Sabuga[1], Ahmed Hashad[1,2], Sven Ehlers[1]

[1] Physikalisch-Technische Bundesanstalt (PTB), Germany
[2] National Institute for Standards (NIS), Egypt

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Experimental study of PTB FPG as secondary pressure standard

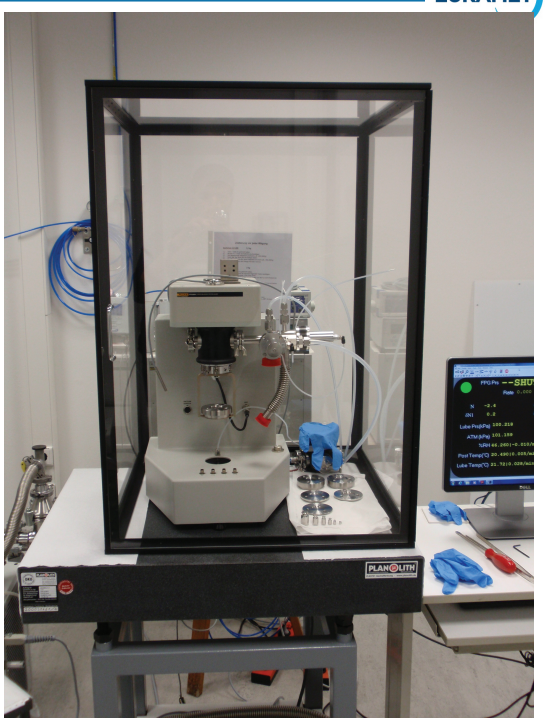
Wladimir Sabuga, Ahmed Hashad, Sven Ehlers

Outline

- Load cell calibration
- Traceability chain
- Calibration against pressure balance
- Benchmark tests in gauge and absolute mode
- Verification against mercury manometer
- Consistency of experiments and theory
- Uncertainties

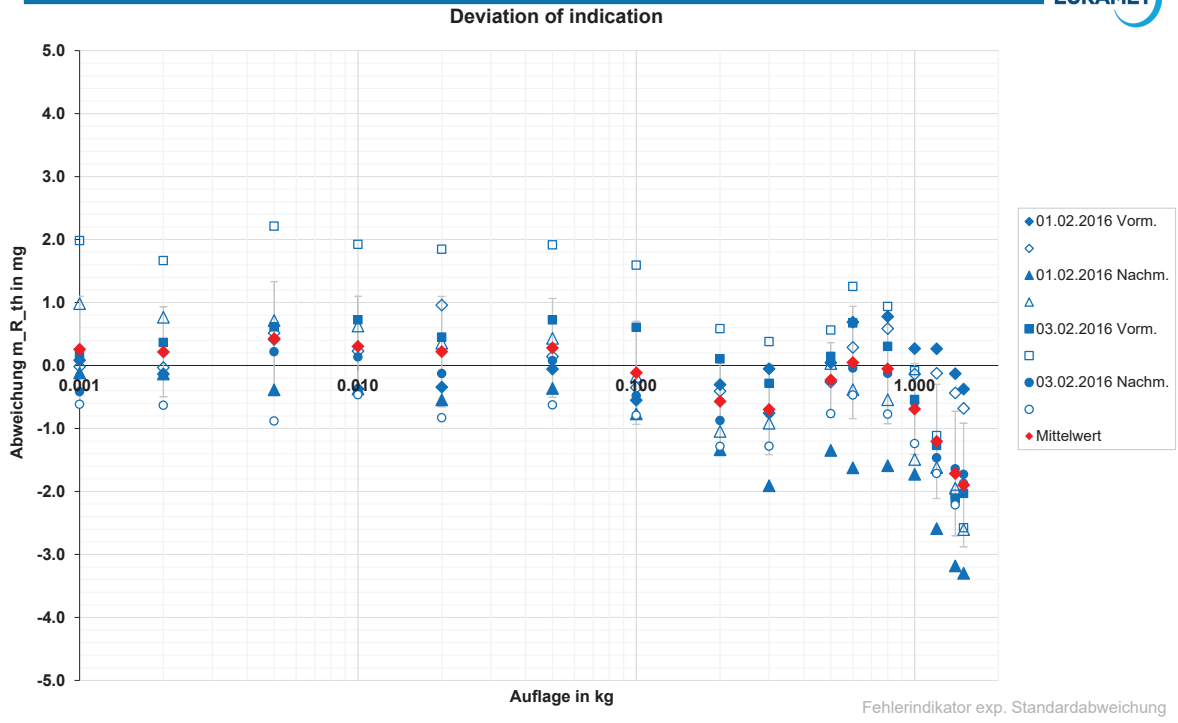
Calibration of components
Load cell

When calibrating the load cell, the FPG was placed inside a protecting cover to avoid the effect of air drafts.



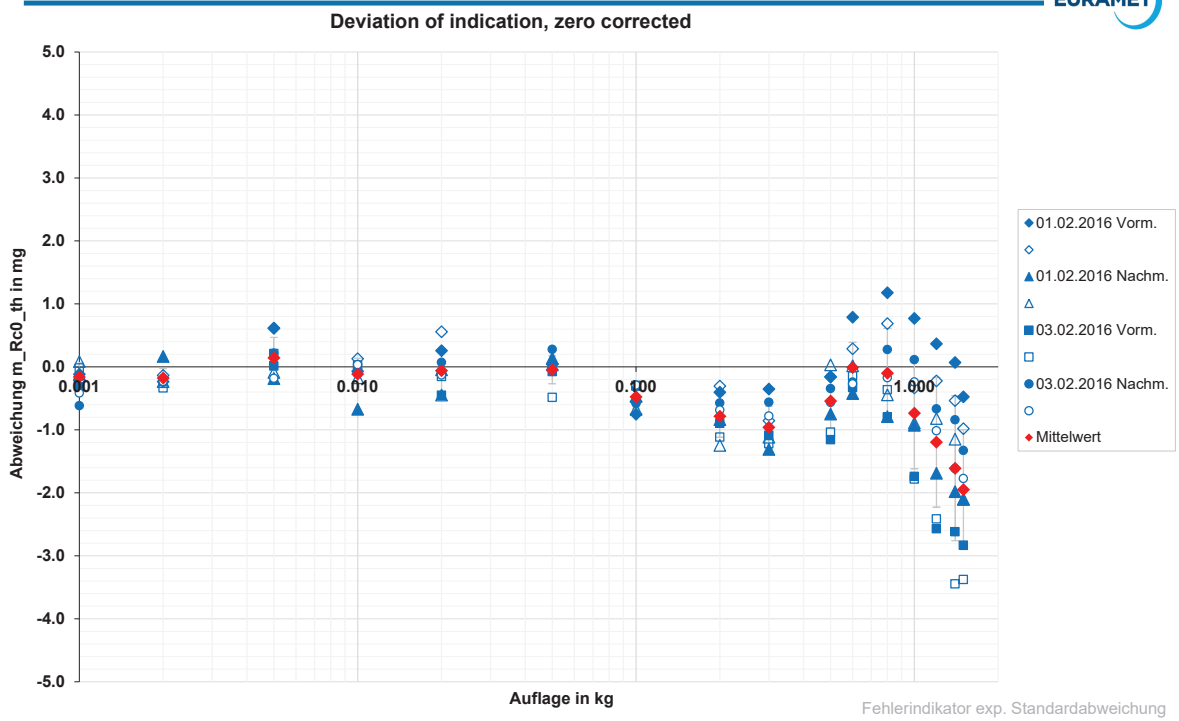
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Calibration of components
Load cell



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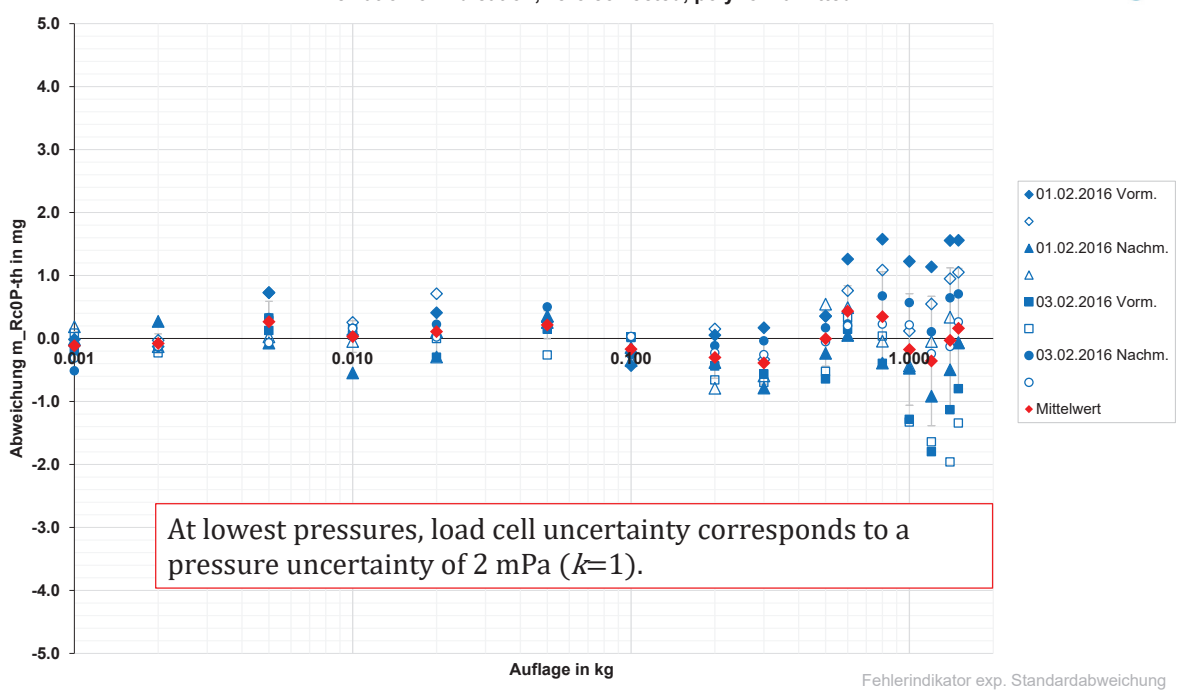
Calibration of components
Load cell



Fehlerindikator exp. Standardabweichung

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Calibration of components
Load cell

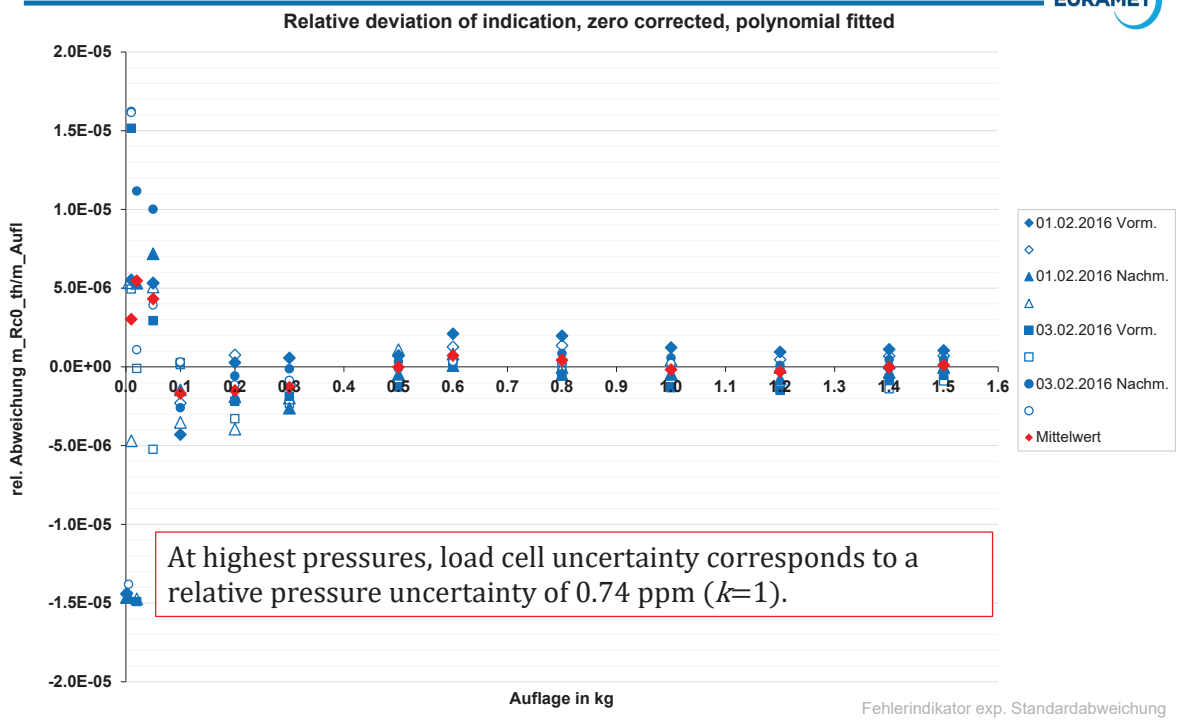


At lowest pressures, load cell uncertainty corresponds to a pressure uncertainty of 2 mPa (k=1).

Fehlerindikator exp. Standardabweichung

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Calibration of components
Load cell



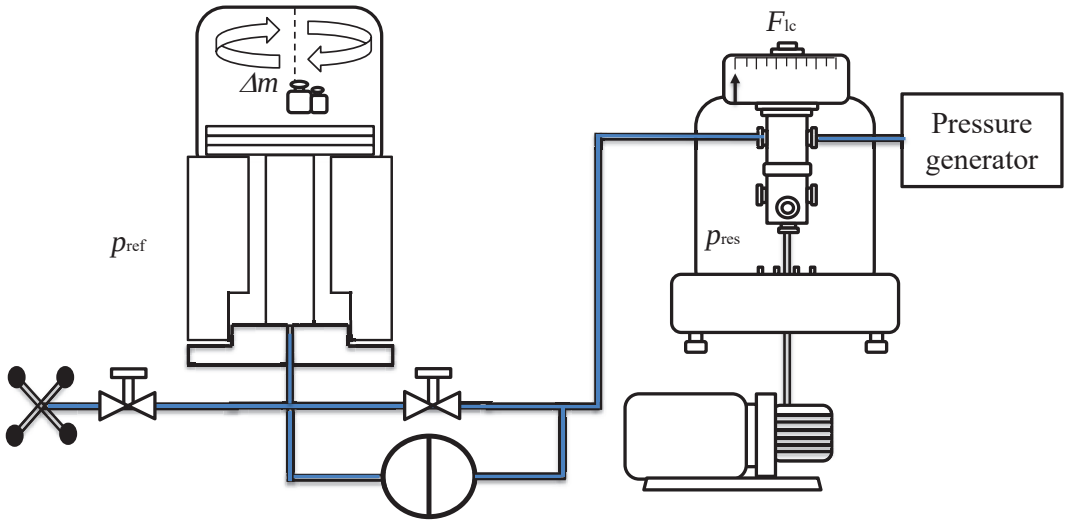
At highest pressures, load cell uncertainty corresponds to a relative pressure uncertainty of 0.74 ppm (k=1).

Fehlerindikator exp. Standardabweichung

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Effective area determination
using cross float method

➢ **Determination of the effective area using reference pressure balance**

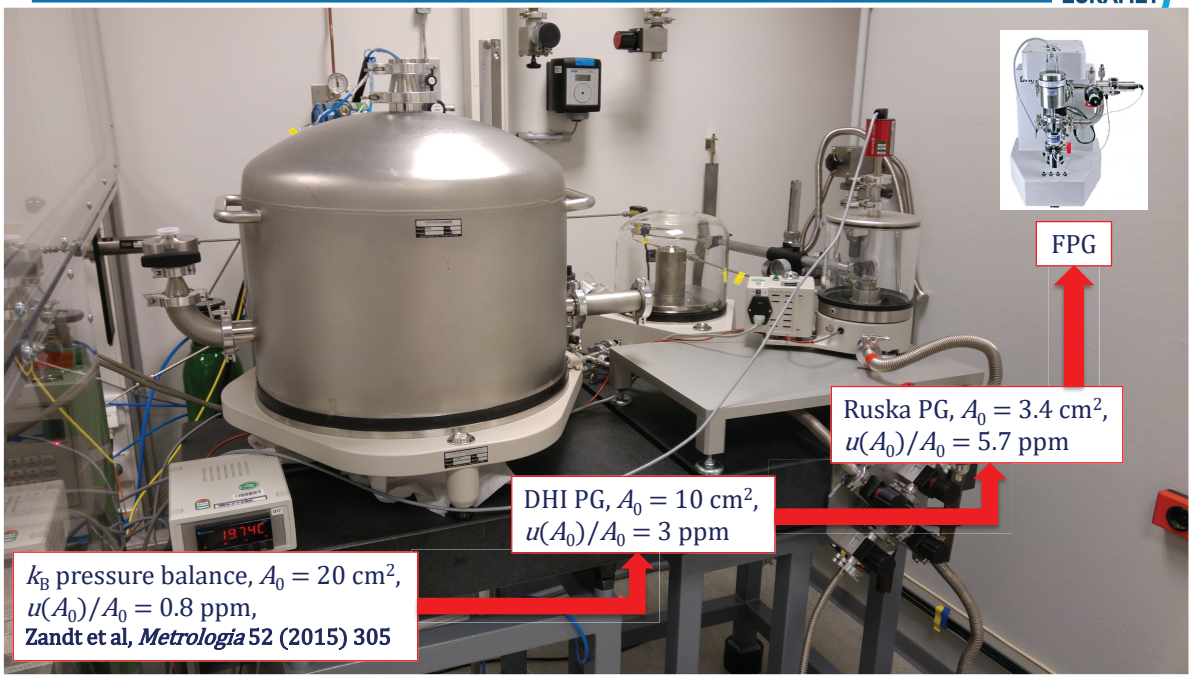


$$A = \frac{F_{lc}}{(p_{ref} - p_{res}) * [1 + (\alpha + \beta) * (t - 20)]}$$

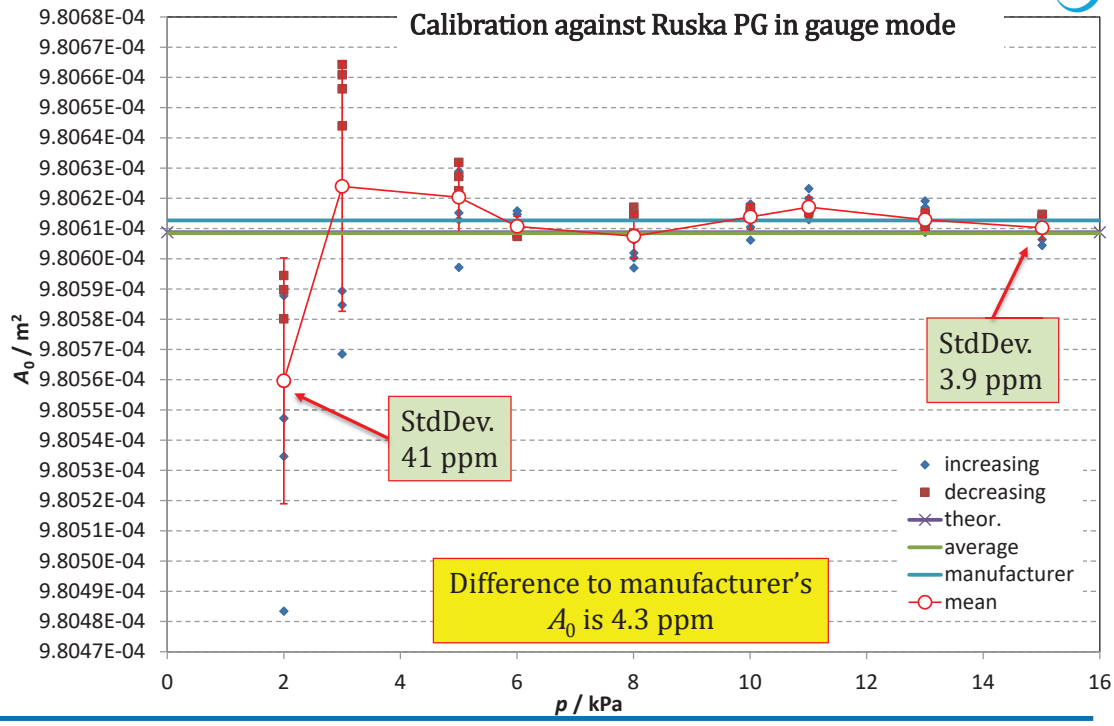
At equilibrium

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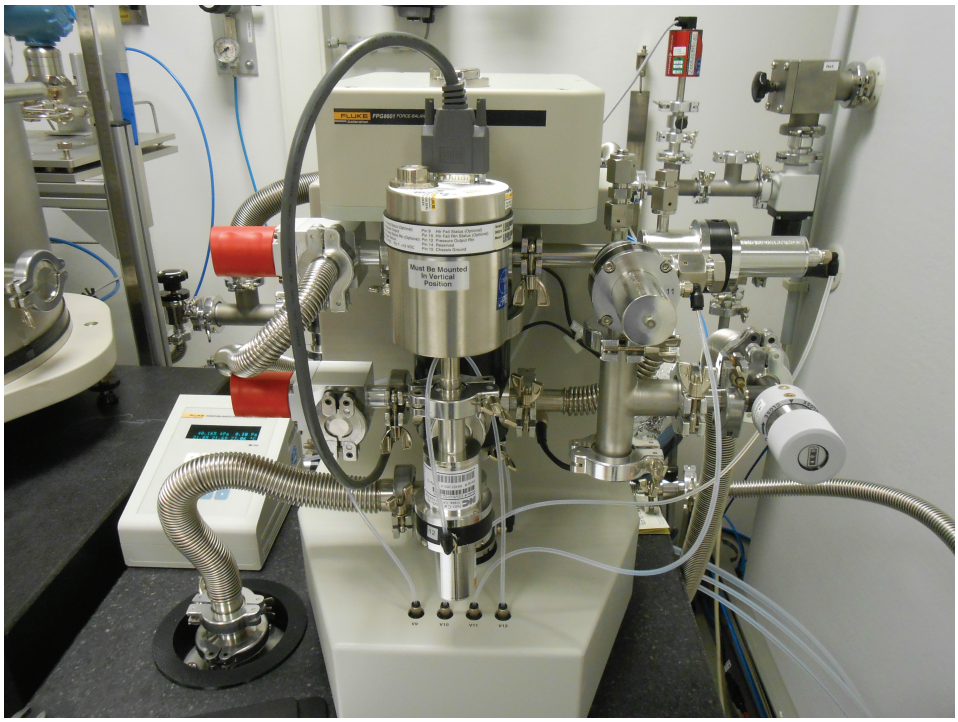
FPG effective area
Traceability chain



FPG effective area
Traceability chain



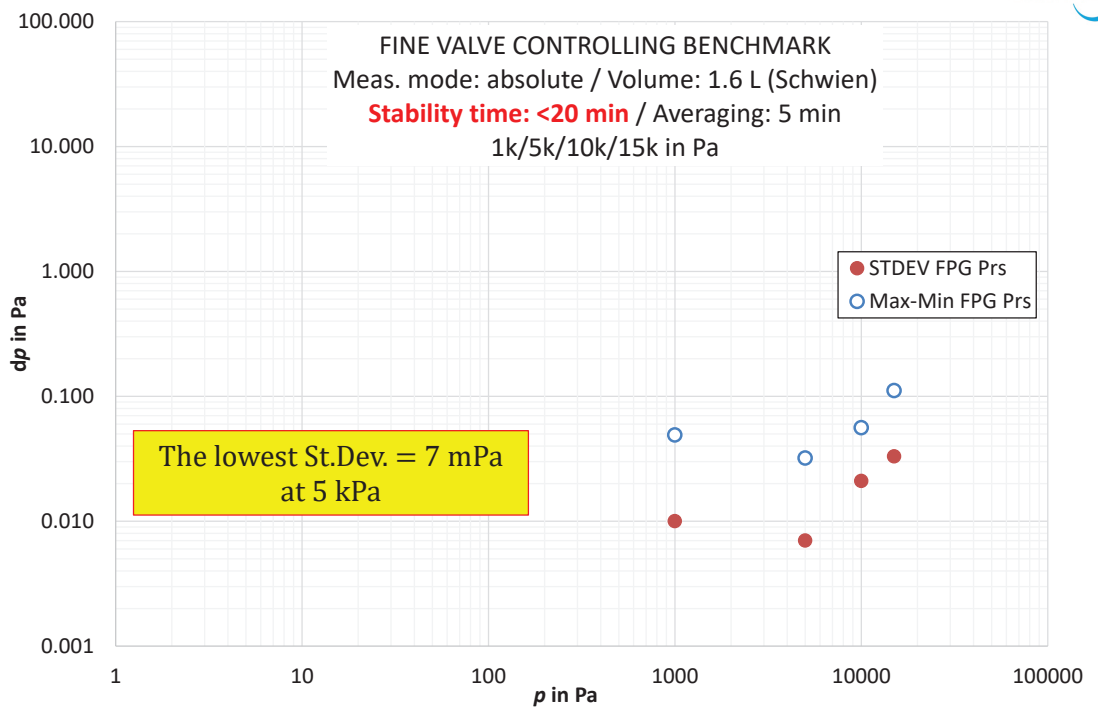
Benchmark tests of FPG with fine leak valve in absolute mode



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20.02.2019 11

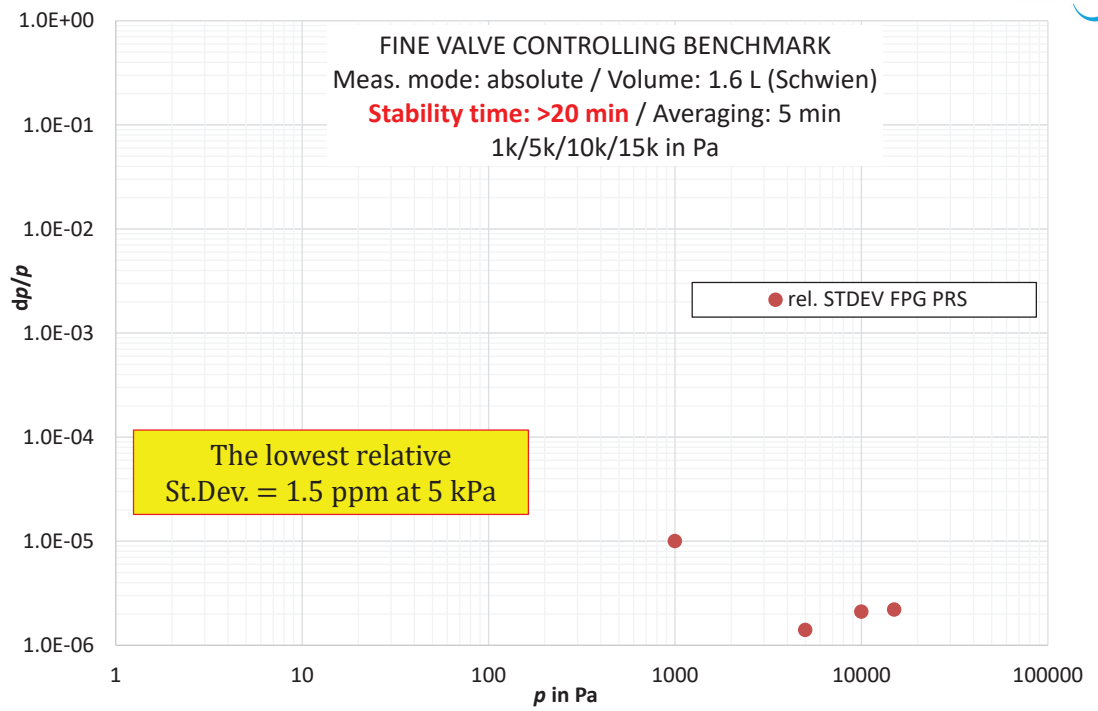
Benchmark tests of FPG with fine leak valve in absolute mode



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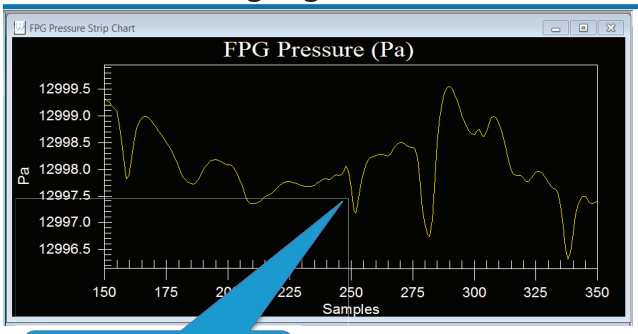
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Benchmark tests of FPG with fine leak valve in absolute mode



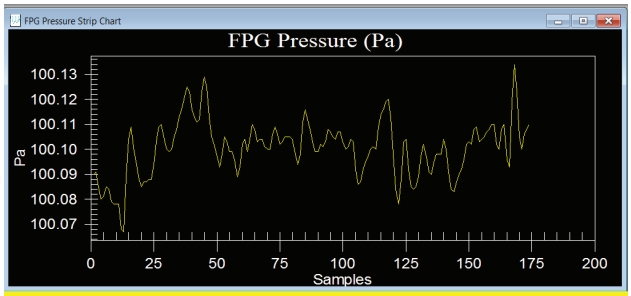
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Benchmark tests of FPG with VLPC in gauge mode



Measurement pressure volume about 1.6 l

Open door effects, typically about 1 Pa

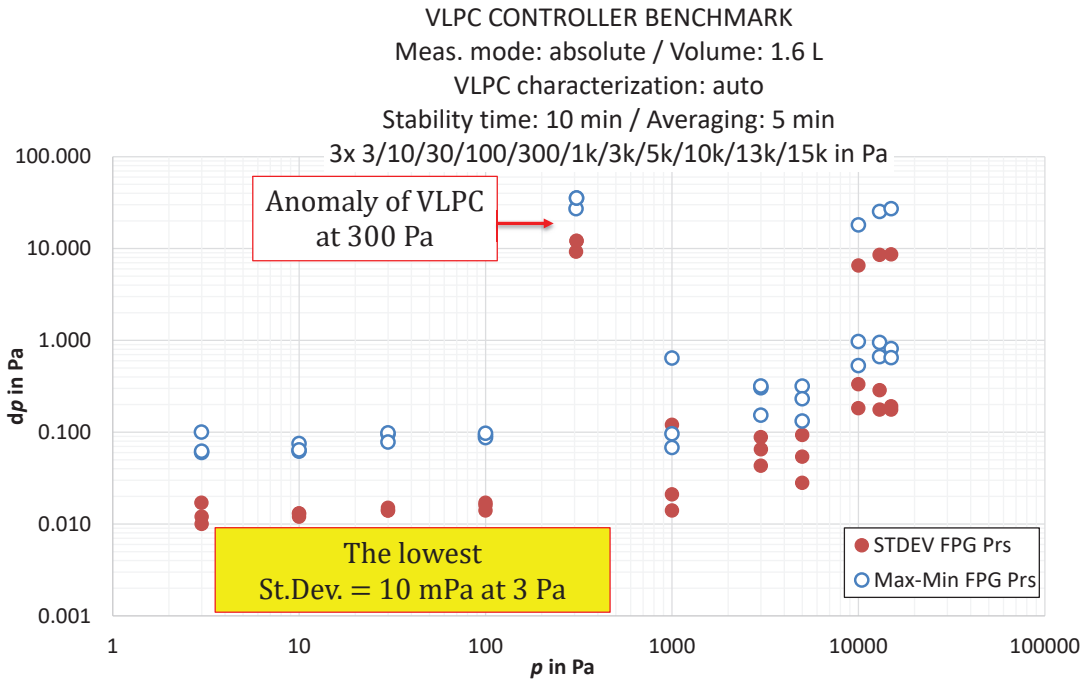


FPG connected with Schwien Hg-manometer

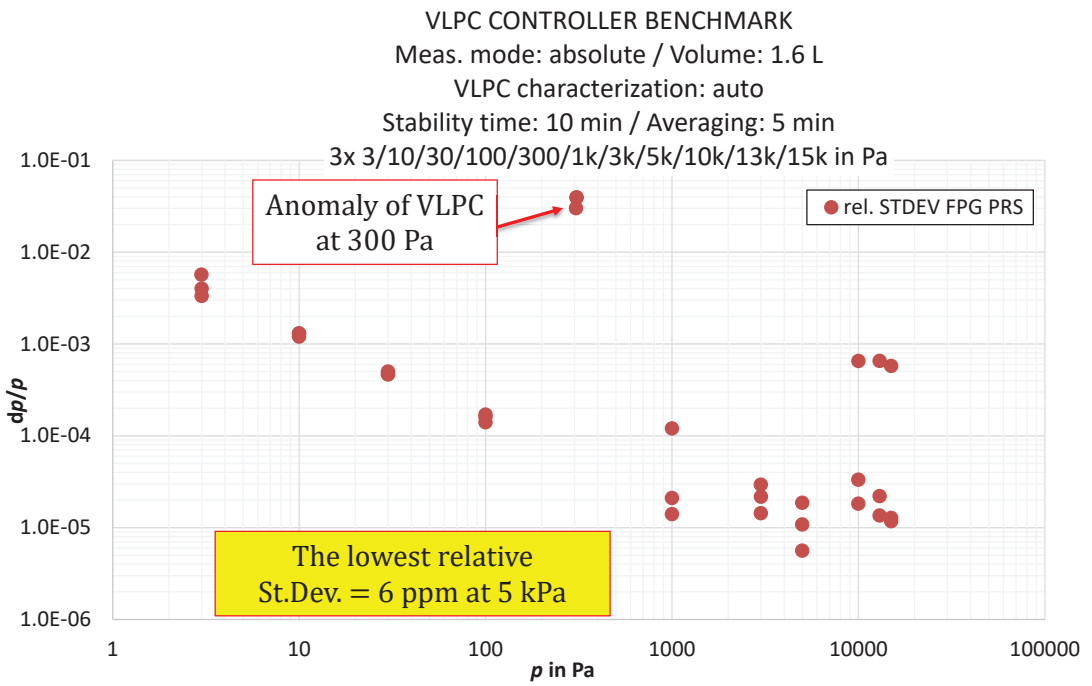
150 Pa VLPC-Range, FPG verbunden mit Schwien-Mess.- und Ref.-Anschluss, Oeffnung zur Umgebung ueber VLPC

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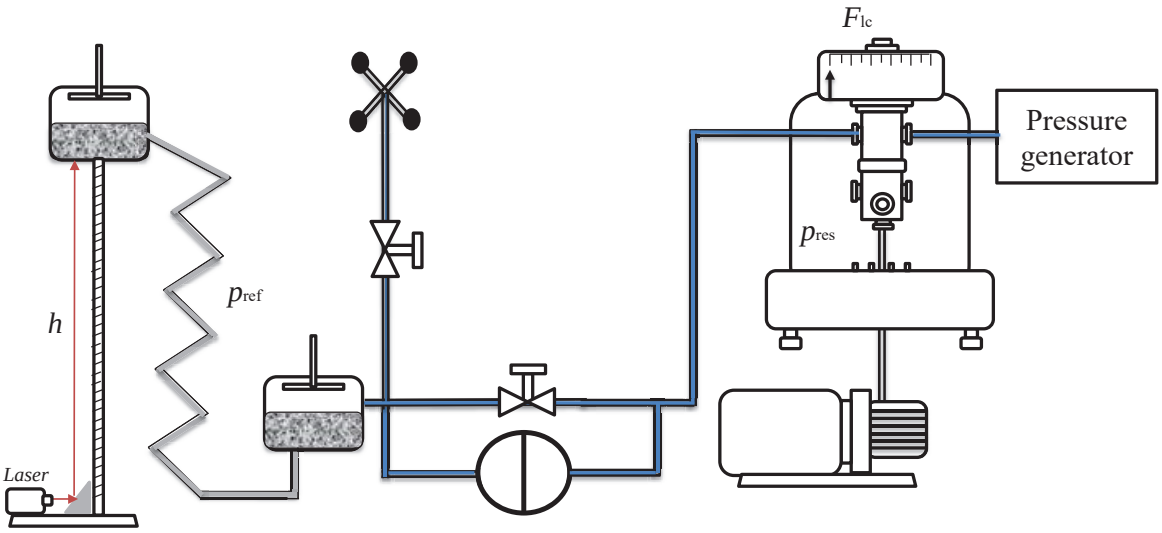
Benchmark tests of FPG with VLPC in absolute mode



Benchmark tests of FPG with VLPC in absolute mode



Verification of FPG using a mercury column

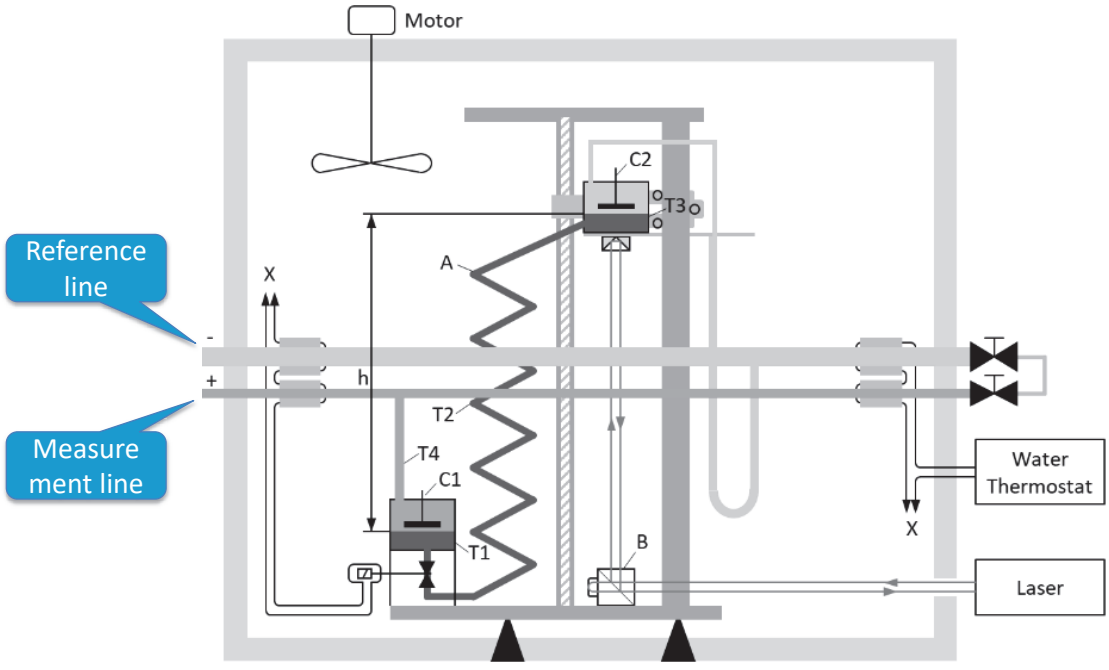


$$A = \frac{F_{lc}}{(p_{ref} - p_{res}) * [1 + (\alpha + \beta) * (t - 20)]}$$

At equilibrium

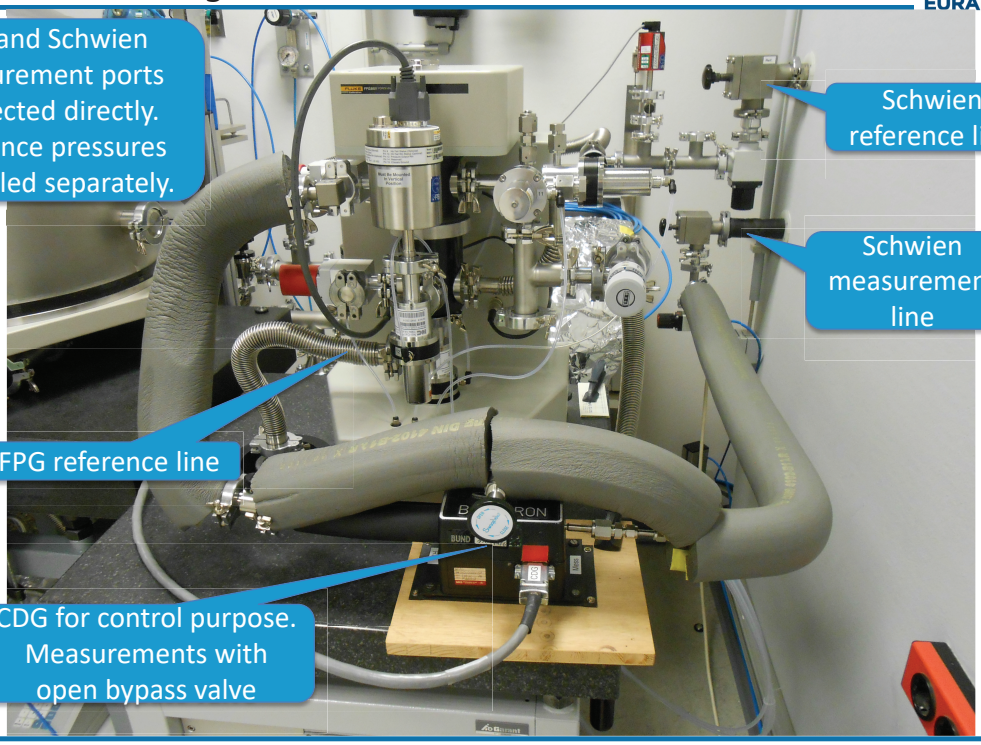
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Verification of FPG against Schwien Hg manometer



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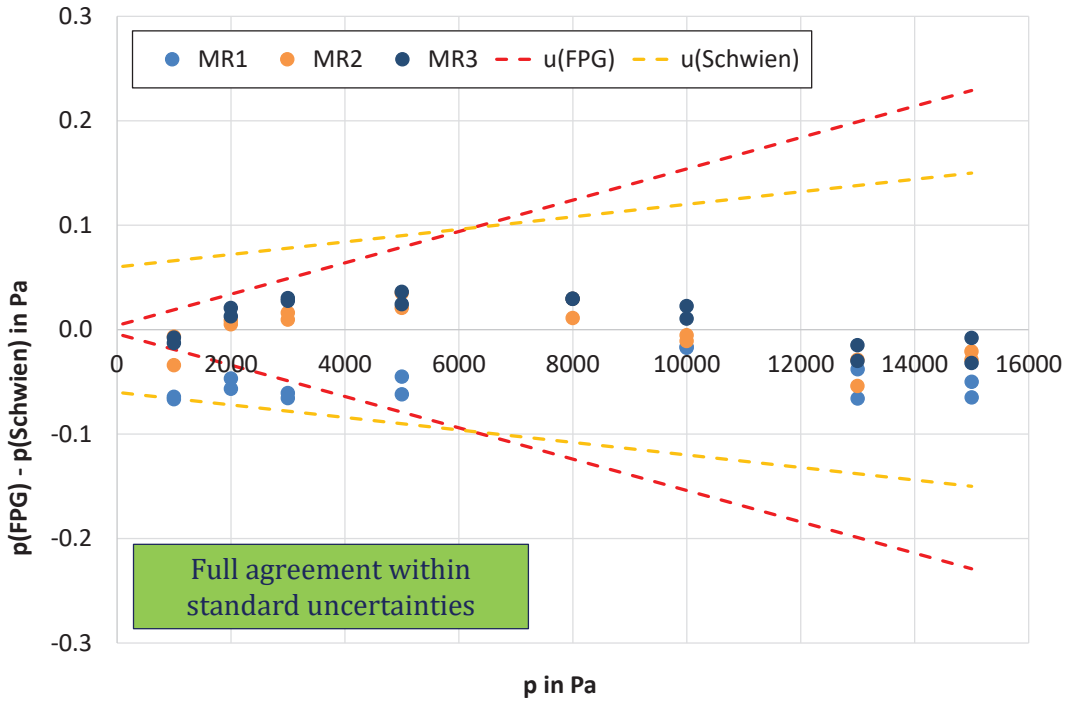
Verification of FPG against Schwien Hg manometer in absolute mode



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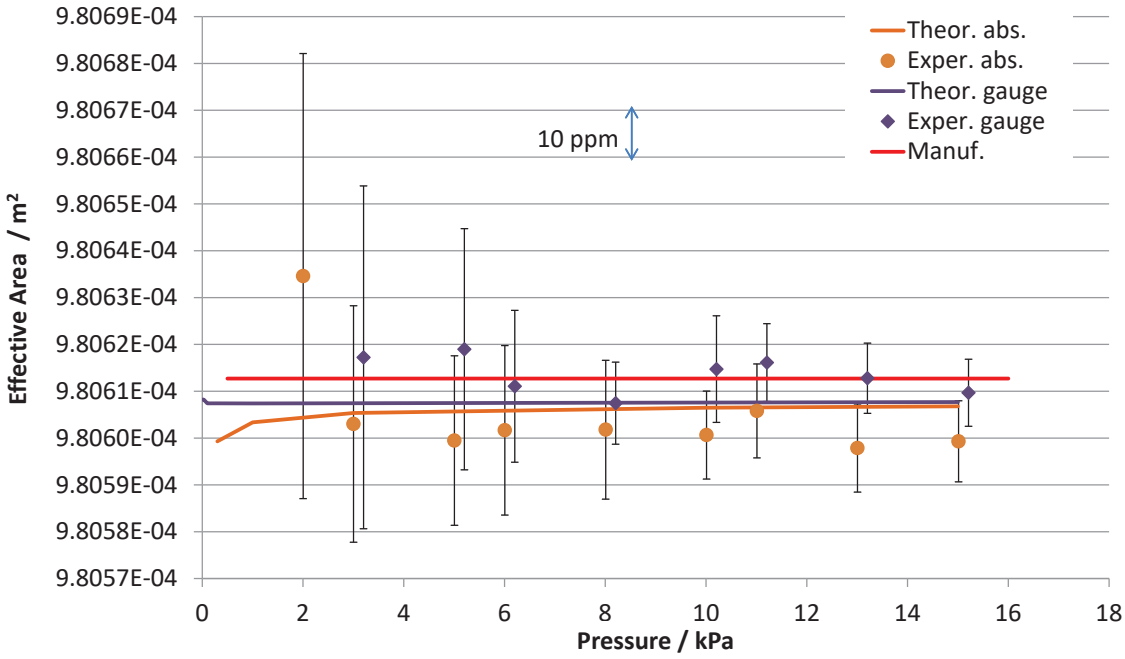
Verification of FPG against Schwien Hg manometer in absolute mode



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Verification of FPG
consistency of experimental and theoretical areas



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Pressure calculation equations FPG

Calibration coefficient of load cell

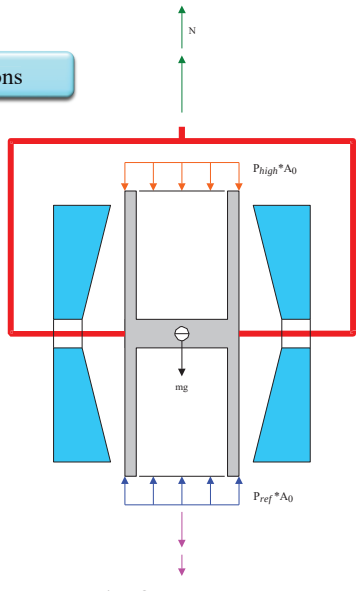
Reading

Corrections

$$\Delta P = \frac{K_{cal} \cdot (N + \delta N_1 + \delta N_2 + \delta N_3)}{A_0 \cdot [1 + (\alpha_p + \alpha_c) \cdot (t - 20)]}$$

Effective area

Thermal expansion coeff. of the piston and cylinder



* FPG 8601 manual

National Metrology Institute

FPG uncertainties

Main type B uncertainties ($k = 1$)

Uncertainty source	Contribution to $u(p)$
Theoretical effective area	3.2 ppm
Experimental effective area	8 ppm
Load cell	2 mPa + 1.2 ppm
Thermal expansion	0.5 ppm
Residual pressure (absolute mode)	2.1 mPa
Combined type B uncertainty	**2.9 mPa + 8.1 ppm**

Type A uncertainty ($k = 1$)

Uncertainty source	Contribution to $u(p)$
Stability of pressure, absolute mode	7 mPa + 2 ppm
Stability of pressure, gauge mode	10 mPa + 6 ppm

Combined experimental uncertainty ($k = 1$)

Operation mode	Theor. A_0, $u(p)$	Experim. A_0, $u(p)$
Absolute	**7.6 mPa + 4 ppm**	**7.6 mPa + 8.3 ppm**
Gauge	**10 mPa + 6.9 ppm**	**10 mPa + 10 ppm**

Conclusions and outlook

- FPG experimentally characterised
- Traceability provided to primary pressure balance and mercury manometer
- Experimental effective areas fully consistent with theoretical
- Stability of measured pressures and therefore increased standard deviation and poor repeatability appear the main uncertainty sources at the moment
- Preliminary, conservative estimation of uncertainty leads to 10 mPa + $10^{-5} \cdot p$ (k = 1), which can be reduced in the future

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Thank you for your attention!

Any questions?

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[1] PTB
[2] Researcher Assistent at NIS, Guest Researcher at PTB

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E-Mail: wladimir.sabuga@ptb.de

Effect of lubrication pressure in FPG

David Herránz, Virginia Marcos, Salustiano Ruiz, Nieves Medina

Centro Español de Metrología (CEM), Spain

GOBIERNO DE ESPAÑA
MINISTERIO DE ECONOMÍA, INDUSTRIA Y COMPETITIVIDAD

Effect of lubrication pressure in FPG

14IND06 pres2vac

EMPIR 2014

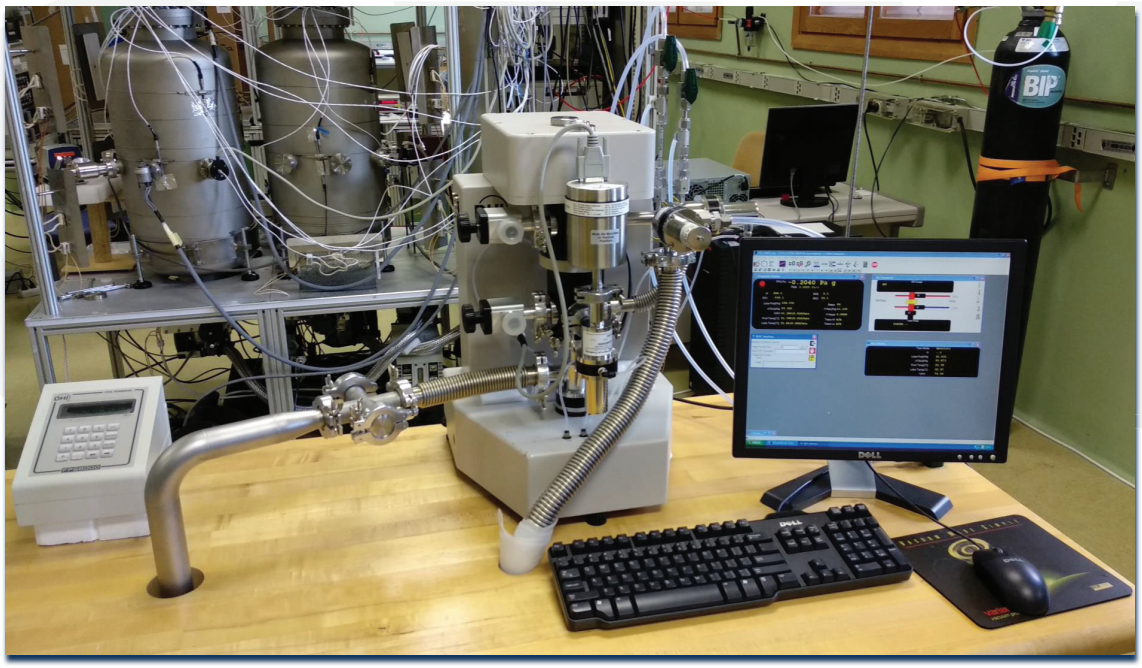
NMI: Centro Español de Metrología (CEM Pressure Laboratory)

D. Herranz, V. Marcos, S. Ruiz and N. Medina

CEM CENTRO ESPAÑOL DE METROLOGÍA

Borås, Sweden 2018-05-16

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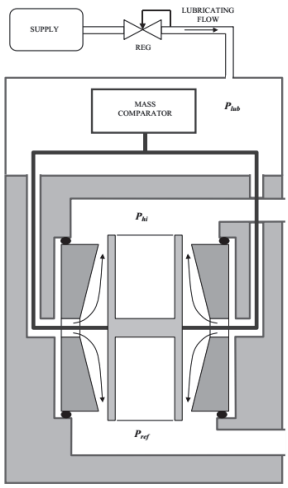
CEM CENTRO ESPAÑOL DE METROLOGÍA

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FPG (Force-Balanced Piston Gauge):

Cylinder has a conical shape on its interior surface to generate a centering force on the piston when a lubrication pressure is applied in the annular gap through the center of the cylinder.

Gas injected between the piston and the cylinder to minimize the effects due to friction, called lubrication gas.



Pressure: $$P = \frac{K_{Cal} \cdot g \cdot (N + \Delta N_1 + \Delta N_2 + \Delta N_3)}{A_{eff(20^oC)} \cdot [1 + \alpha \cdot (t - t_0)]} \cdot \frac{\rho_M - \rho_a}{\rho_M - \rho_0}$$

$$\Delta N_1 = -K_b \cdot (P_{lub} - P_{lub,0})$$

$$\Delta N_2 = K_d \cdot [(P_{lub} - P_{ref}) - (P_{lub,0} - P_{ref,0})]$$

$$\Delta N_3 = V_{ext} \cdot g \cdot \left[\left(\frac{P_{ref}}{T_{ref}} - \frac{P_{ref,0}}{T_{ref,0}} \right) \cdot \frac{M_{gas}}{K_{cal} \cdot Z_{ref,0} \cdot R} \right]$$

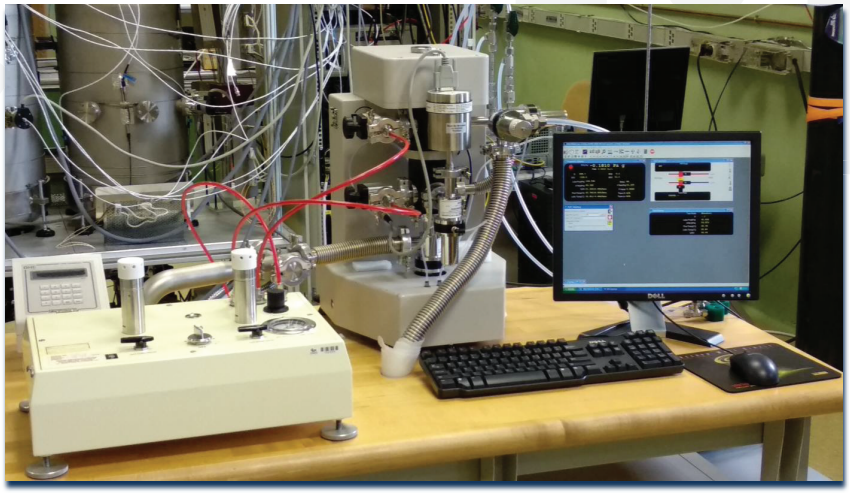
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Aim: Study of the lubrication pressure on the initial conditions of the FPG

Operation mode: gauge mode

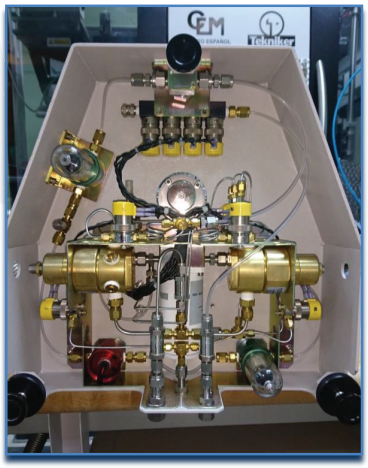
Pressure generator: V1600 piston gauge

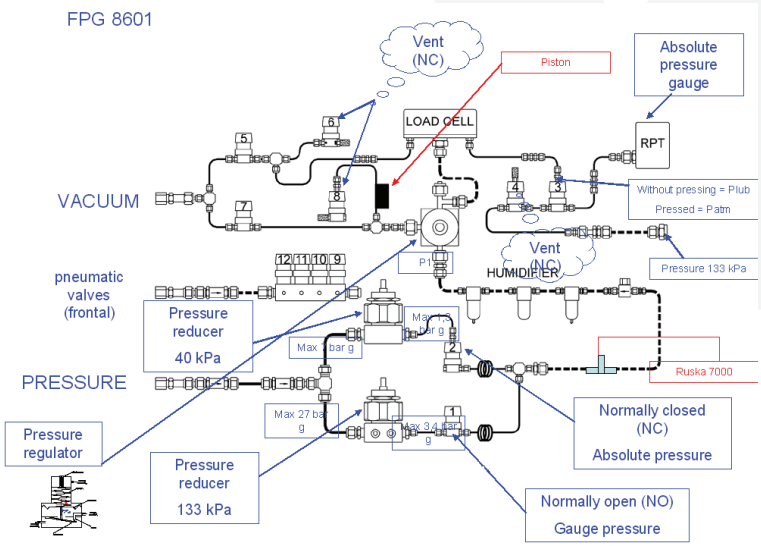


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FPG internal valve configuration:



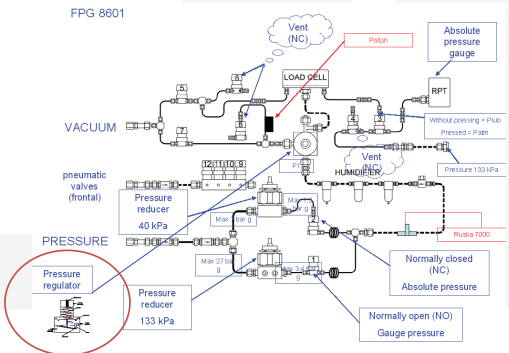


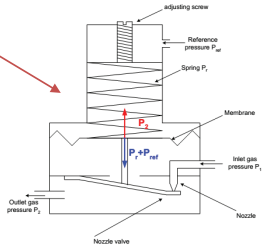
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Method:

Lubrication pressure is regulated by the pressure regulator, which scheme is detailed in the following figure:





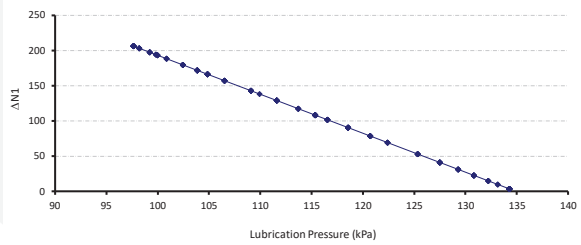
The steps to follow are:

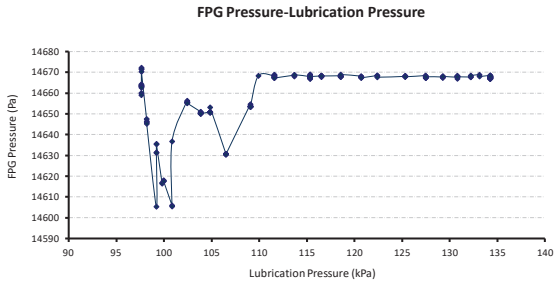
1- Zeroing the FPG
2- Internal calibration is performed
3- Bypass valve (V10) is closed
4- Pressure is generated using the V1600 pressure gauge as a pressure generator and the measurement is recorded every 5 s, until the test is finished.
5- The adjusting screw of the pressure regulator is loosened and then, valve 6 is opened, in order to lubrication pressure decreases until atmospheric pressure.
6- Valve 6 is closed and it is waited until the FPG is stable.
7- The adjusting screw of the pressure regulator is tightened very slowly and waiting in every moment to reach the stability, until the lubrication pressure reaches around 134 kPa.

CEM CENTRO ESPAÑOL DE METROLOGÍA

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CEM results: 15 kPa

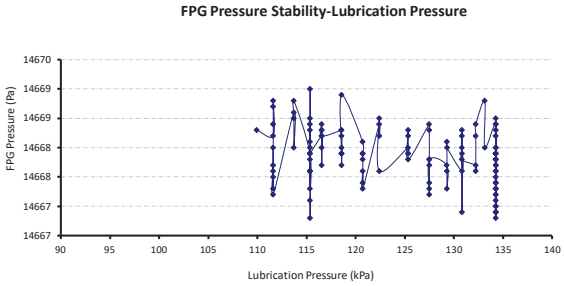




$$\Delta N_1 = -K_b \cdot \left(P_{lub} - P_{lub,0}\right)$$

$$\Delta N_2 = K_d \cdot \left[\left(P_{lub} - P_{ref}\right) - \left(P_{lub,0} - P_{ref,0}\right)\right]$$

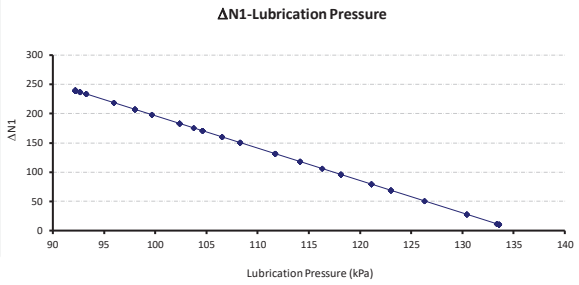
$$\Delta N_3 = V_{ext} \cdot g \cdot \left[\left(\frac{P_{ref}}{T_{ref}} - \frac{P_{ref,0}}{T_{ref,0}}\right) \cdot \frac{M_{gas}}{K_{cal} \cdot Z_{ref,0} \cdot R}\right]$$

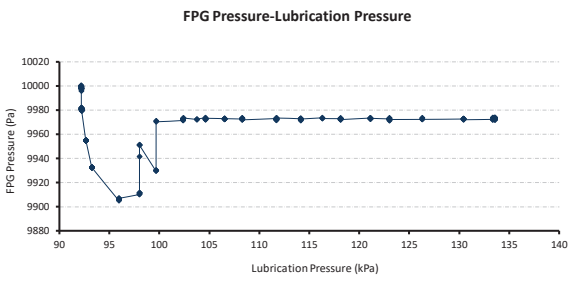


CEM CENTRO ESPAÑOL DE METROLOGÍA

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CEM results: 10 kPa

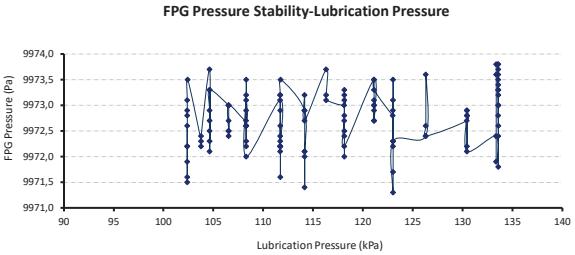




$$\Delta N_1 = -K_b \cdot \left(P_{lub} - P_{lub,0}\right)$$

$$\Delta N_2 = K_d \cdot \left[\left(P_{lub} - P_{ref}\right) - \left(P_{lub,0} - P_{ref,0}\right)\right]$$

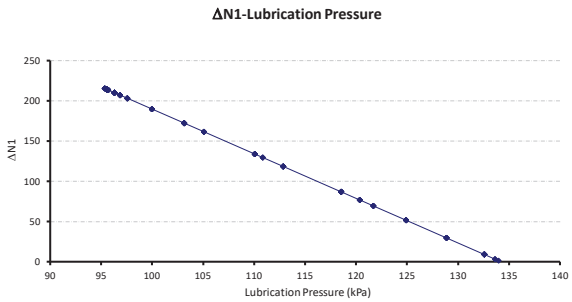
$$\Delta N_3 = V_{ext} \cdot g \cdot \left[\left(\frac{P_{ref}}{T_{ref}} - \frac{P_{ref,0}}{T_{ref,0}}\right) \cdot \frac{M_{gas}}{K_{cal} \cdot Z_{ref,0} \cdot R}\right]$$

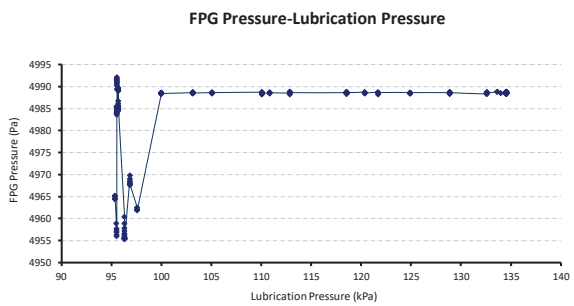


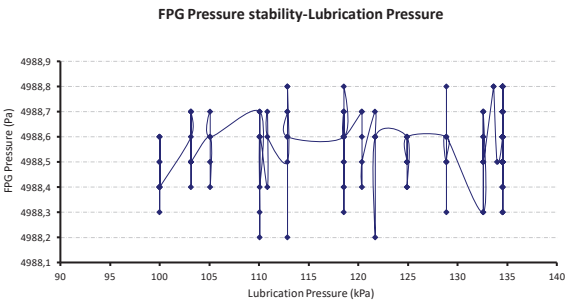
CEM CENTRO ESPAÑOL DE METROLOGÍA

GOBIERNO DE ESPAÑA
MINISTERIO DE ECONOMÍA, INDUSTRIA Y COMPETITIVIDAD

CEM results: 5 kPa







$$\Delta N_1 = -K_b \cdot \left(P_{lub} - P_{lub,0}\right)$$

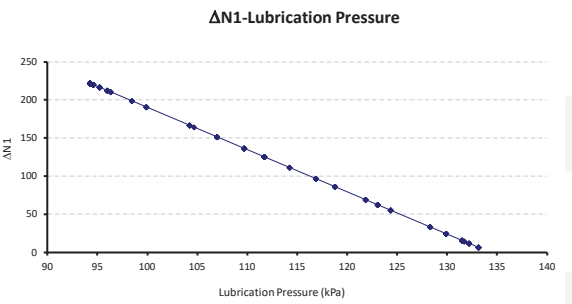
$$\Delta N_2 = K_d \cdot \left[\left(P_{lub} - P_{ref}\right) - \left(P_{lub,0} - P_{ref,0}\right)\right]$$

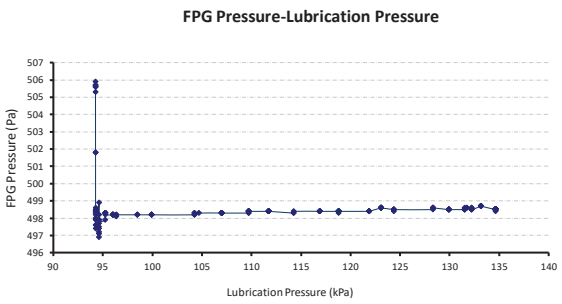
$$\Delta N_3 = V_{ext} \cdot g \cdot \left[\left(\frac{P_{ref}}{T_{ref}} - \frac{P_{ref,0}}{T_{ref,0}}\right) \cdot \frac{M_{gas}}{K_{cal} \cdot Z_{ref,0} \cdot R}\right]$$

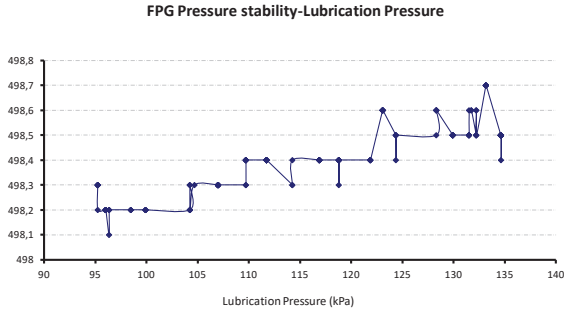
CEM CENTRO ESPAÑOL DE METROLOGÍA

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MINISTERIO DE ECONOMÍA, INDUSTRIA Y COMPETITIVIDAD

CEM results: 500 Pa





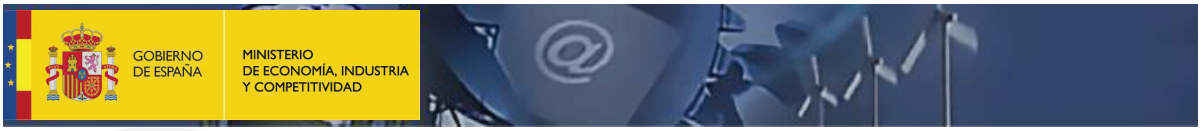


$$\Delta N_1 = -K_b \cdot \left(P_{lub} - P_{lub,0}\right)$$

$$\Delta N_2 = K_d \cdot \left[\left(P_{lub} - P_{ref}\right) - \left(P_{lub,0} - P_{ref,0}\right)\right]$$

$$\Delta N_3 = V_{ext} \cdot g \cdot \left[\left(\frac{P_{ref}}{T_{ref}} - \frac{P_{ref,0}}{T_{ref,0}}\right) \cdot \frac{M_{gas}}{K_{cal} \cdot Z_{ref,0} \cdot R}\right]$$

CEM CENTRO ESPAÑOL DE METROLOGÍA



Thank you for your attention

CEM CENTRO ESPAÑOL DE METROLOGÍA

Calibration techniques for the intermediate pressure-to-vacuum range – Introduction

Mark Plimmer[1], Djilali Bentouati[2]

[1] Conservatoire national des arts et metiers (CNAM), France
[2] Laboratoire national de métrologie et d'essais (LNE), France

Workpackage 3: Calibration techniques for the intermediate pressure-to-vacuum range

INTRODUCTION

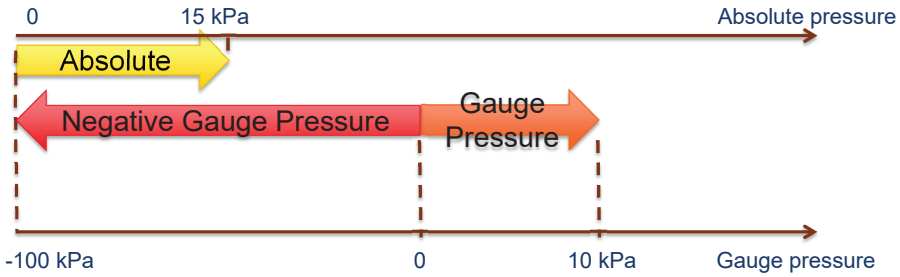
Mark Plimmer

Djilali Bentouati

Pres2Vac - Final Workshop 16th – 17th May 2018, RISE – Borås, Sweden

Pressure ranges

- Absolute pressure from 10 Pa to 15 kPa
- Positive gauge pressure from 0 to 10 kPa
- Negative gauge pressure from -100 kPa to 0 kPa



3

Tasks

- Task 3.1: Methods for accurate, **weather independent** calibration of low gauge pressure measuring instruments.
- Task 3.2: Methods for negative gauge pressure calibration based on **primary standards** :
  - Absolute and gauge pressure balances.
  - Force Piston Gauge (FPG).
  - Liquid column manometers.
- Task 3.3: Traceable methods for FPG calibration
  - Absolute mode.
  - Gauge mode.
- Task 3.4: Calibrations of positive and negative gauge pressure devices from -100 kPa to +10 kPa
  - Secondary laboratories.
  - Industry.

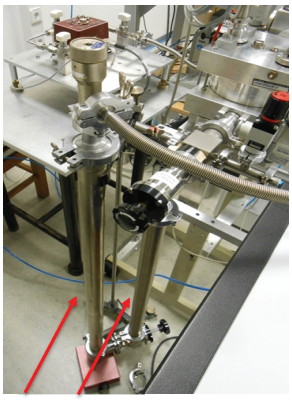
4

Task 3.1: Methods for accurate, **weather independent** calibration of low gauge pressure measuring instruments.

- Measurement range depends on how big atmospheric pressure is.
- Changes in ambient conditions have the greatest relative impact for low gauge pressure measurement (±15 kPa).
- Need to limit their effect.



Hermetic chamber (CMI) :
Standard and device under test (DUT) both enclosed at desired pressure.

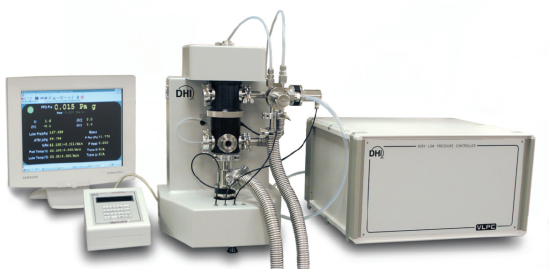


Variable volume reservoir (PTB)
Standard and device under test (DUT) both connected to the volume.

5

Task 3.3: Traceable methods for FPG calibration

- 0 to 15 kPa gauge mode and 1 Pa to 15 kPa absolute mode
- FPGs widely used in primary and secondary labs and some industrial ones.
- Static piston cylinder assemblies ≠ rotating dead-weight balances
- Need to develop and validate traceable methods to calibrate them.



6

Tasks 3.2 and 3.4: Calibration of positive and negative gauge pressure measuring intruments

- Very few primary standards have been developed for the range -100 kPa to +10 kPa.
- Need to extend methods initially developed for positive gauge pressure to negative gauge pressure.
- Target accuracies in industry have increased in recent years:
  3 Pa + 200 ppm industry
  1 Pa + 30 ppm secondary laboratories
- Primary laboratories have had to improve their measurement capabilities by using pressure balances to calibrate instruments for secondary labs and industry.

7

First session:

- 5 min Calibration Techniques- Introduction
  Mark PLIMMER (LNE/CNAM)

- 30 min Methods and instruments for accurate, weather-independent calibration of low gauge pressure instruments
  Dominik PRAŽÁK (CMI), Wladimir SABUGA (PTB)

- 20 min Methods for the calibration of Force balanced Piston Gauge FPG
  Djilali BENTOUATI (LNE/CNAM)

Second session:

- 20 min Measurement of positive and negative gauge pressure using an absolute pressure balance
  Mark PLIMMER (LNE/CNAM)

- 15 min Negative gauge pressure calibration by means of a mercury manometer and conventional gauge pressure piston gauges
  Sven EHLERS (PTB)

- 10 min Examples: Calibration of two digital manometers for negative and positive gauge pressure
  Djilali BENTOUATI (LNE/CNAM)

- 8 min Conclusion
  Mark PLIMMER (LNE/CNAM)

Weather-independent low pressure measurements - I

Zdeněk Krajíček, Martin Vičar, Dominik Pražák,

Český metrologický institute (CMI), Czech Republic

Weather-independent low pressure measurements - I

Zdeněk Krajíček, Martin Vičar, Dominik Pražák

Czech Metrology Institute



pres2vac

-A metrology research project within the European Union Horizon 2020 framework.

Introduction

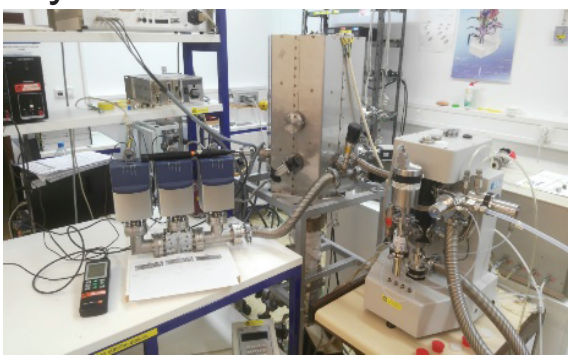
- The calibration uncertainty of the very good low gauge pressure measuring instruments can often be much larger than the accuracy of the instruments themselves, only because the atmospheric pressure is unstable.
- Often the calibrating laboratories have to wait for stable weather and/or perform their calibrations at weekends to reduce the effects of weather and/or activities in laboratory buildings.
- Our task was to study the calibration methods for accurate, weather-independent calibrations of low pressure gauges, particularly important for the range below 1 kPa.
- Target uncertainty (in primary labs) ≤ 0.1 Pa + $3 \cdot 10^{-5} \cdot p$. (The typical atmospheric disturbances can be higher by few orders of magnitude).
- Target uncertainty (in industrial conditions) ≤ 3 Pa + $2 \cdot 10^{-4} \cdot p$.

Humidity influence

- It is another important factor having effect on the pressure sensors and the electronics.
- A study of the influence of the surrounding air humidity to the capacitance diaphragm gauges indication was performed.
- Nine gauges (INFICON, type CDGsci) were used, three of 100 Torr full scale, three of 10 Torr and three of 1 Torr. They are hermetically sealed and backfilled with pure nitrogen to prevent penetration of the water from the outside atmosphere inside the gauge body.
- Three humidity values were used for the experiment near the ideal points (0, 50 and 100) % relative humidity. The humidity level was established by the dry pure air (lower humidity), the natural ambient air in the laboratory (near 50 %) and using the humid air (for high humidity).

Overall view of the calibration system

Because the relationships between the humidity and the characteristics of the sensors were expected to be weak, FPG8601 was used for the calibrations.



The gauges in enclosure with controlled relative humidity

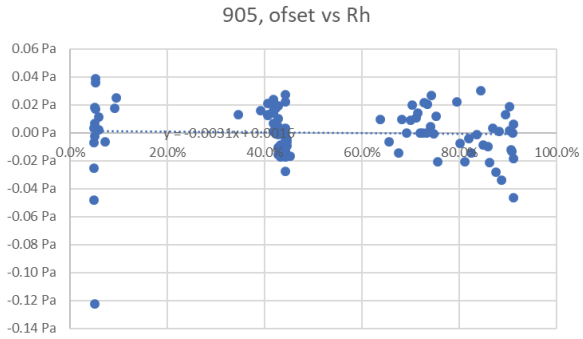
For the humidity measurements, a temperature-humidity meter Testo 645 was used. It had no electronic communication, so the recording was done by the periodic snapshots of a USB camera.

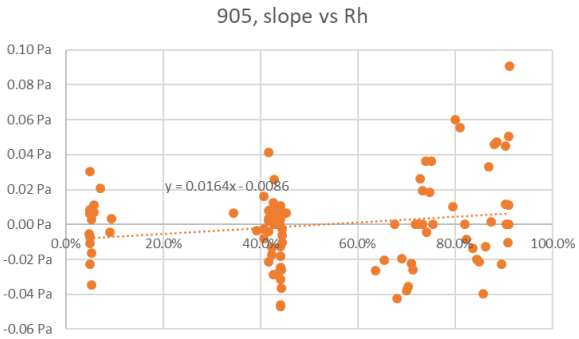


100 Torr gauges

Example: S.No. 905 offset and slope versus relative humidity.

All three gauges showed the changes of the offset and the slope below 0.02 Pa, lower than the observed dispersion of the indication values and lower than the uncertainty of the calibration system itself (0.03 Pa).

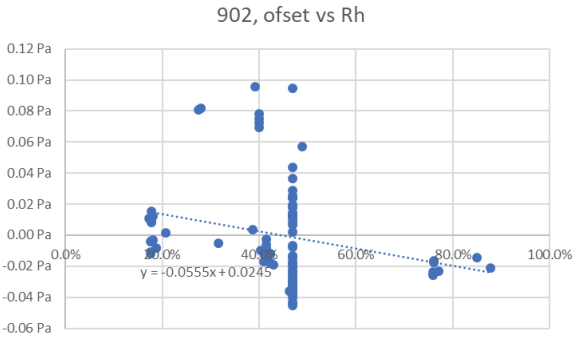


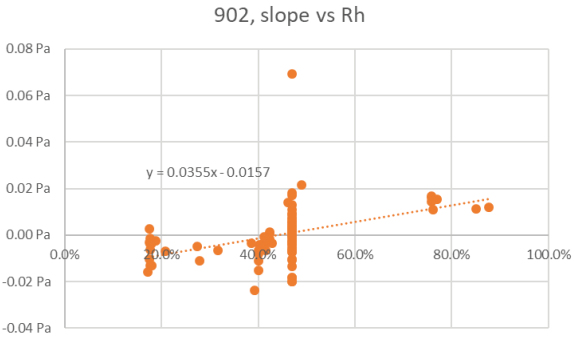


10 Torr gauges

Example: S.No. 902 offset and slope versus relative humidity.

The gauges showed a potential shift of their offsets, approximately −0.04 Pa. However, comparing it to the dispersion of the values at 50 % of the relative humidity level, the correlation between relative humidity and offset is more or less questionable.

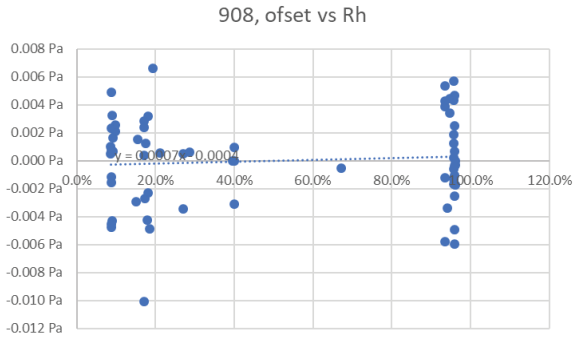


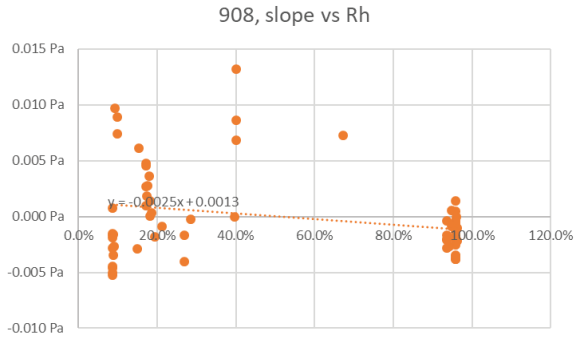


1 Torr gauges

Example: S.No. 908 offset and slope versus relative humidity.

Although these gauges were expected to be relatively more sensitive to the humidity due to their lowest range, the offset and span relationships versus the relative humidity of the surrounding air is evidently negligible. The maximum change of the slope (0.002 Pa) is 10× smaller than the uncertainty level at 133 Pa of absolute pressure for the used standard.





Conclusion

- The data did not show any significant correlation between the relative humidity of the surrounding air and the indications of the gauges.
- The gauges proved their insensibility to the atmospheric humidity.



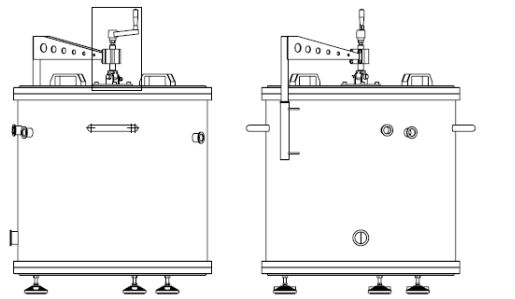
Atm. pressure fluctuations

- 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:

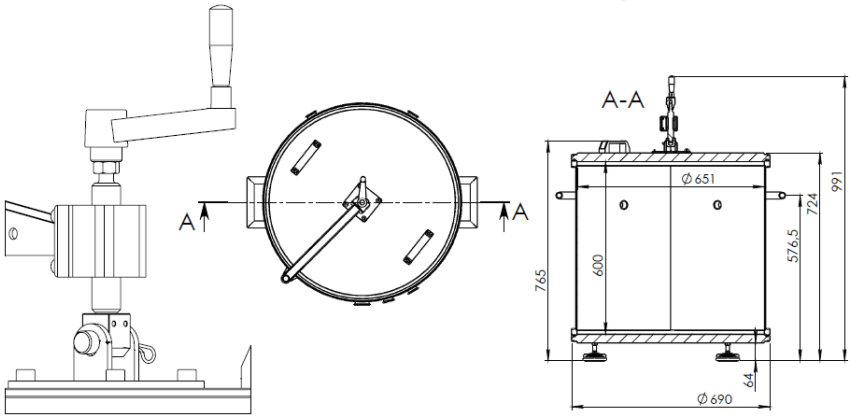
1. Utilisation of an additional volume connected to the reference ports of the LS and the DUT.
2. Use of a hermetic chamber large enough to enclose both the LS and the DUT (*interesting especially for the low gauge pressure devices with only one port*).

Hermetic chamber

It consists of a cylindrical steel body and two aluminium covers.



A vacuum compatible construction was chosen to be able to widen its utilization in the future.

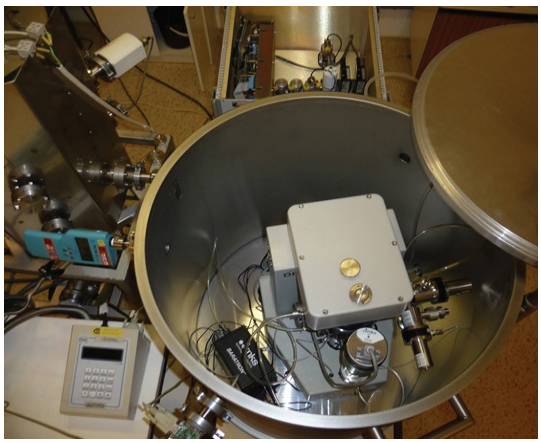


FPG8601 serves as laboratory standard (LS) for this range. It was decided to adjust the internal dimensions to this instrument, plus some extra space for a device under test (DUT).



Three leaktight electrical feedthroughs for an FPG and one for a DUT and four leaktight pressure control feedthroughs plus one spare for a vacuum pump.





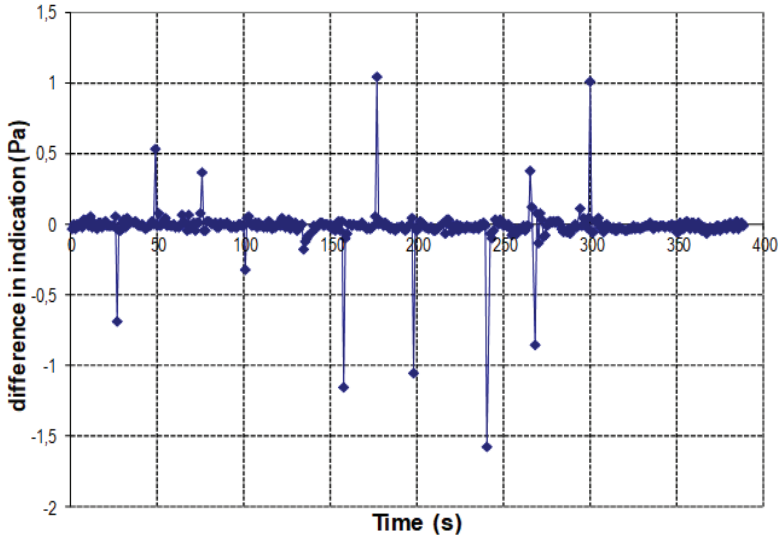
We also placed there a 1 Torr differential MKS Baratron as DUT (heating turned off).



A hermeticity of the entire system was checked by filling the chamber by formation gas (95 % N_2, 5 % H_2) and consequent sniffing by Adixen ASM 340.

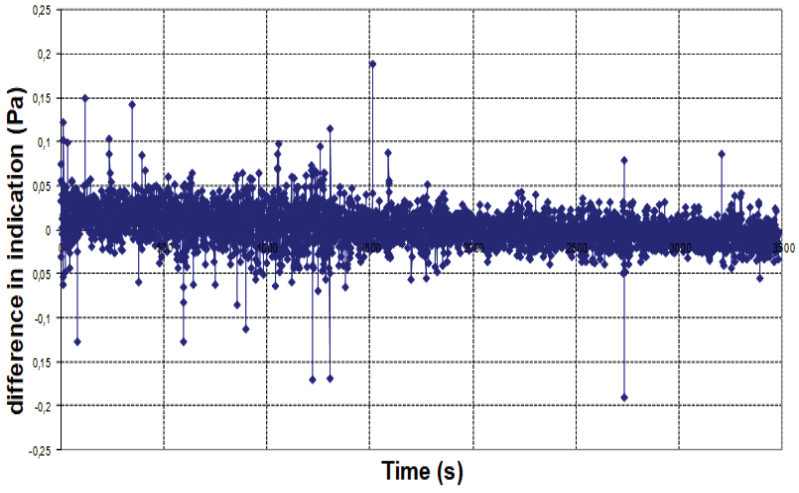
Unstable environment

- The chamber was open during the measurements in a bad, unstable weather and door closings and openings.
- The measured differences between FPG and Baratron indications at 5 Pa had standard deviation 0.153 Pa, i.e. 3 %.



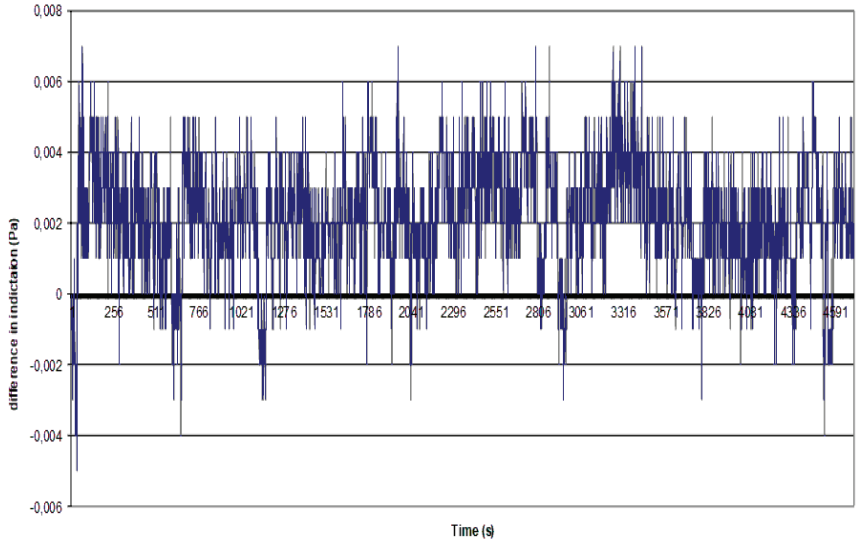
Usual stable environment

- The chamber was open during the measurements in a “normal” weather and without “door disturbances”.
- The measured differences between FPG and Baratron indications at 5 Pa have standard deviation 0.053 Pa, i.e. 1 %.



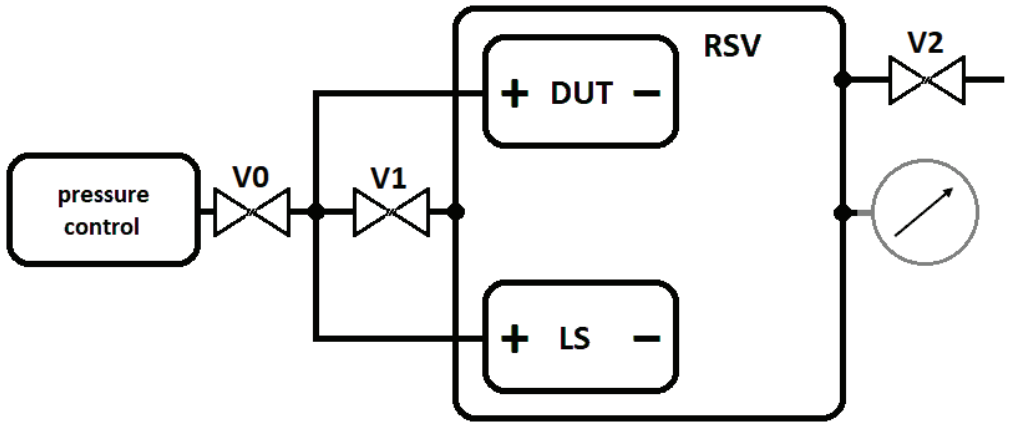
Hermetic environment

- The chamber was closed during the measurements.
- The measured differences between FPG and Baratron indications at 5 Pa have standard deviation 0.002 Pa, i.e. 0.04 %.

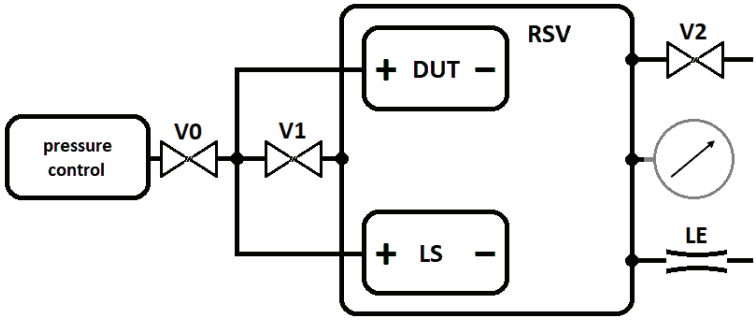


- As the experiments have demonstrated, the method allows to reach the calibration uncertainties not even for the industrial conditions but also for the primary laboratories.
- Both the LS and the DUT must enable electronic communication, and the relevant electrical feedthroughs into the reference stabilisation volume (RSV) must be ensured, or a window in the RSV to read the indications of the LS and DUT must be present.
- A measurement of the pressure inside the RSV by a barometer is non-mandatory, however stability of this pressure must be ensured.

Hermetic chamber set-up



Damped chamber variant (was not tested).



- The RSV can be completely isolated from the external atmosphere by valve V2 in the first set-up or there can be always present a connection between the RSV and the external atmosphere by a small leak damping the atmospheric fluctuations in the second set-up.
- A thermally insulated RSV can be used to provide a stability of the ambient pressure better than that of the bare laboratory, but only in the case when the LS and the DUT do not produce too much heat.
- The zeroing of the LS and the DUT can be done by opening valve V1.
- Considering the mentioned uncertainty limits, the *Comprehensive calibration procedure* according to [EURAMET cg 17 Guidelines on the Calibration of Electromechanical and Mechanical Manometers] should be used. (Calibrations at 11 pressure points in three measuring series in loading and unloading. Repeatability for each pressure point from the three calibration cycles.)



The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

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to be continued …

Weather-independent low pressure measurements - II

Wladimir Sabuga[1], Thomas Konczak[1], Sven Ehlers[1], Ahmed Hashad[1,2]

[1] Physikalisch-Technische Bundesanstalt (PTB), Germany
[2] National Institute for Standards (NIS), Egypt

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

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continued …

Weather-independent low pressure measurements - II

… using a variable volume chamber

Wladimir Sabuga, Thomas Konczak,
Sven Ehlers, Ahmed Hashad

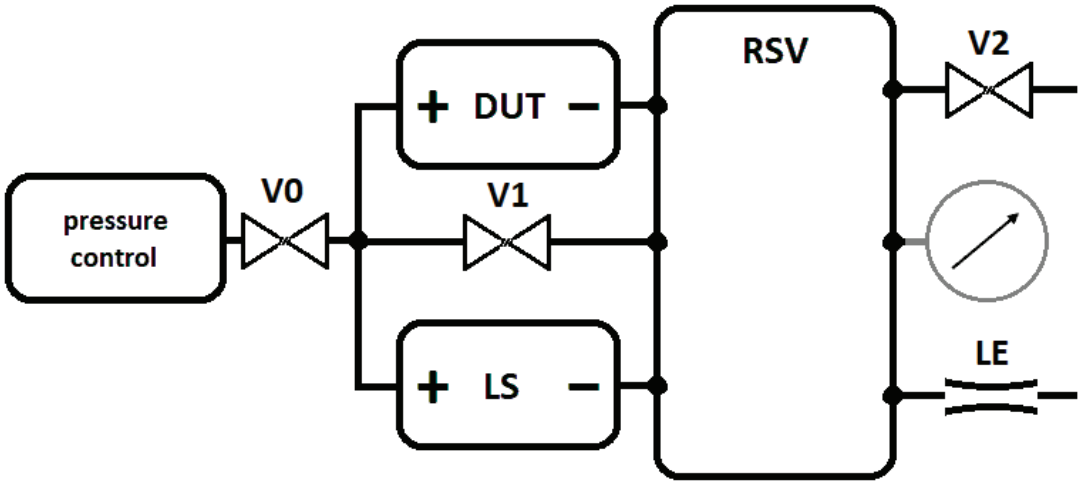
Task

PTB pres2vac EURAMET

JRP Protocol:

- "PTB will carry out tests with a variable volume chamber that is open to the atmosphere or controlled by a pressure controller and used a as a source of ambient reference pressure
- They will use the variable volume chamber to calibrate a precision gauge pressure measuring instrument against an FRS piston gauge
- The calibration results as well as the pressure fluctuation records will be compared by PTB with those obtained under normal/ambient laboratory conditions."

Principle of method



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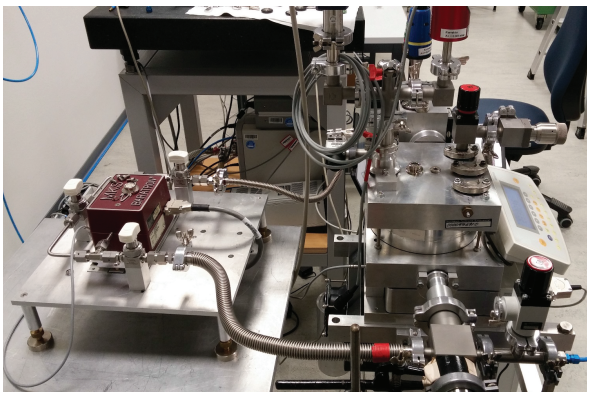
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Experimental arrangement and measurement conditions

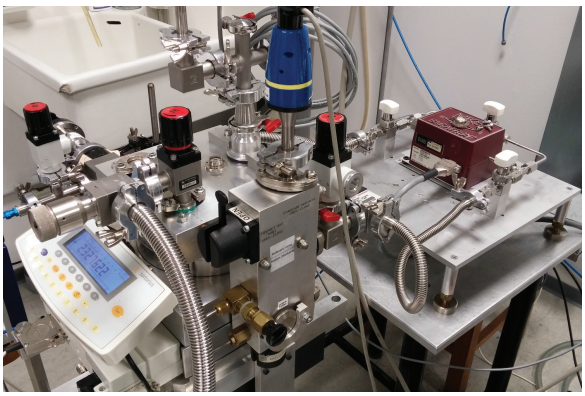
- Low gauge pressure calibrations at 100 Pa, 5 kPa and 10 kPa
- FRS5 of 11 kPa FS was used as reference standard (LS)
- MKS Baratron differential CDG of 13 kPa FS was used as DUT

Connections of FRS5 with MKS Baratron

Reference connection side



Measurement connection side



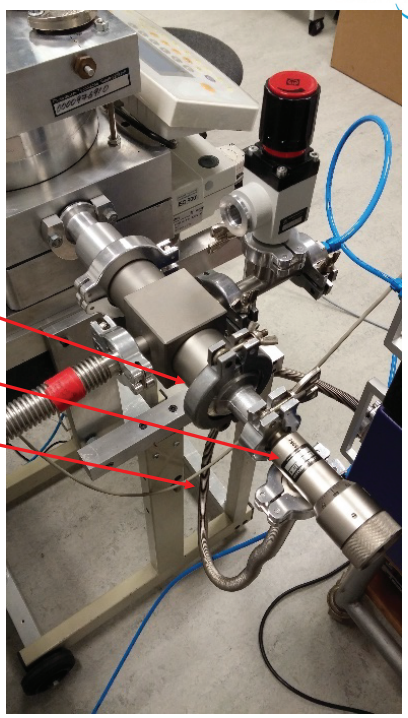
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Measurement condition 1: No additional volume

4 modes were tested:

- Flange being removed, reference pressure connection open to atmosphere
- Reference pressure controlled by pressure controller at 100 kPa through pipe and with fine leak valve completely open
- Pressure in pipe controlled by pressure controller at 10 kPa using fine leak valve to keep pressure in the reference connection at about 100 kPa
- Pressure in pipe controlled at about 100 kPa using fine leak valve and diaphragm pump pumping down to about 1 kPa

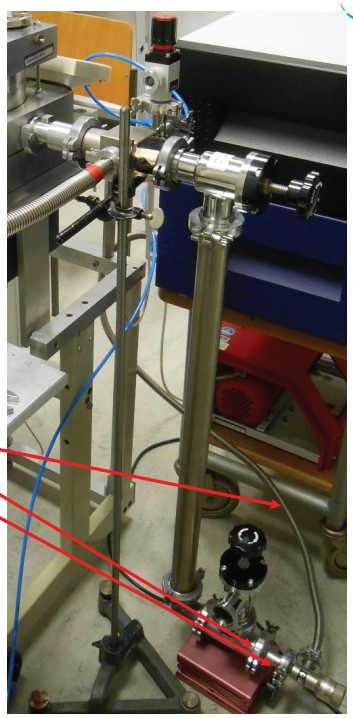


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Measurement condition 2: With additional volume of 1.2 l

3 modes were tested:

- Flange being removed, reference pressure connection open to atmosphere
- Reference pressure controlled by pressure controller at 100 kPa through pipe and with fine leak valve completely open
- Pressure in pipe controlled by pressure controller at 10 kPa using fine leak valve to keep pressure in the reference connection at about 100 kPa.

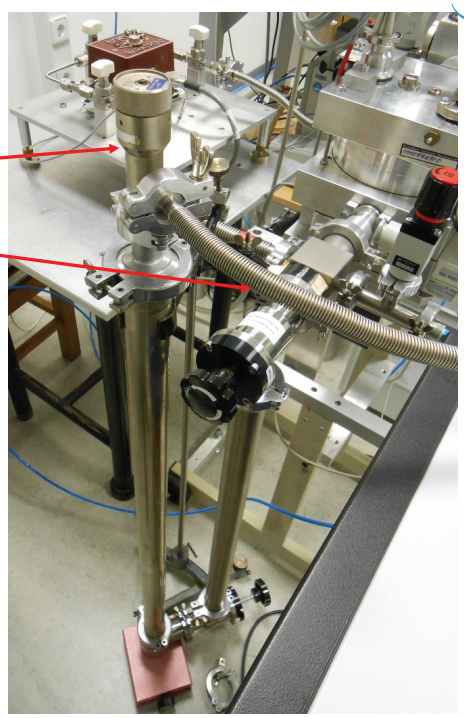


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Measurement condition 3: With additional volume of 2.7 l

2 modes were tested:

- Reference pressure controlled by pressure controller at 100 kPa through pipe and with fine leak valve completely open
- Pressure in pipe controlled by pressure controller at 10 kPa using fine leak valve to keep pressure in the reference connection at about 100 kPa.

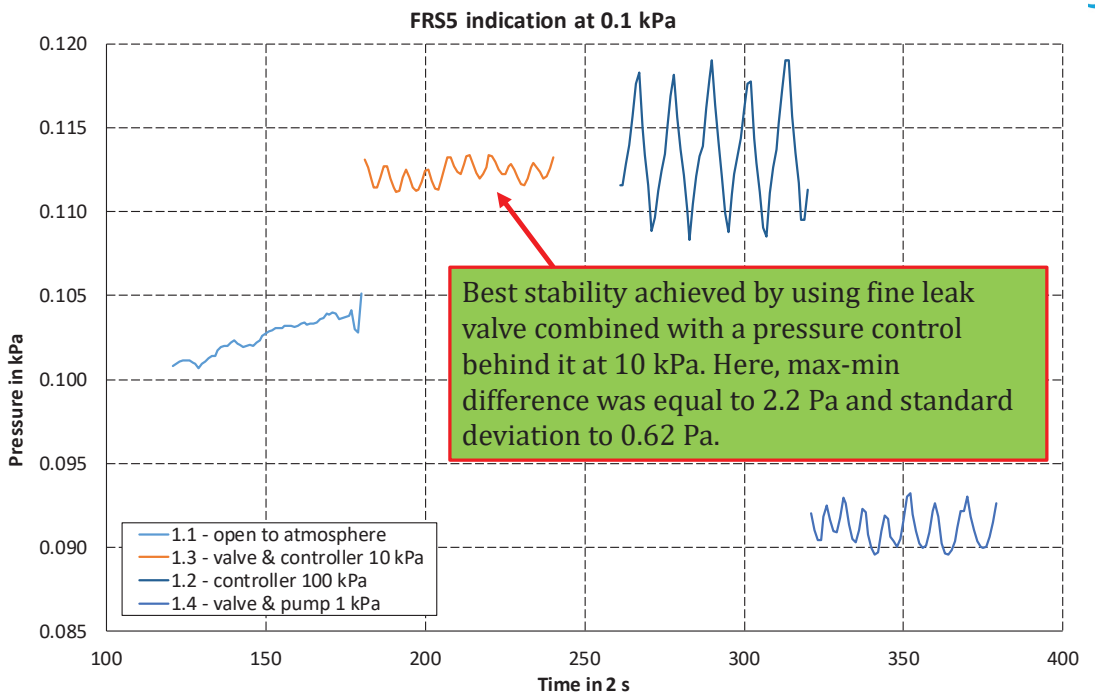


Reference pressure was monitored through by a pressure controller Mensor CPC 8000. Pressure of 100 kPa or 10 kPa in pipe was controlled by pressure controller WIKA CPC 8000.

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20.02.2019 7

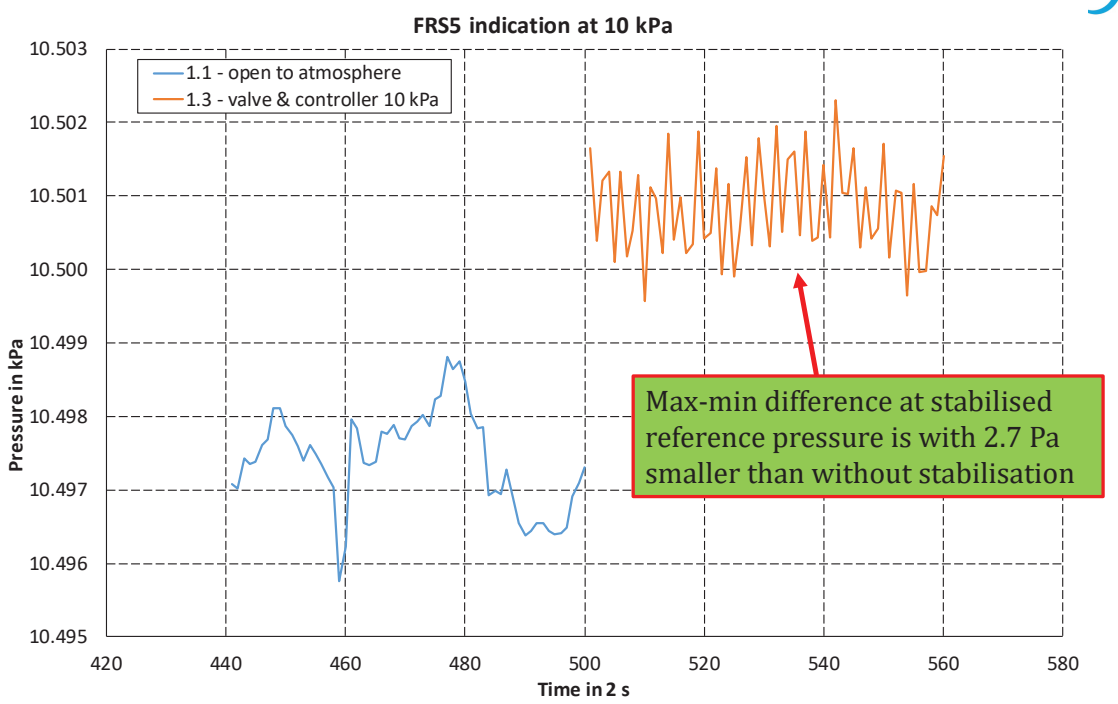
No additional volume: FRS5 readings at 0.1 kPa



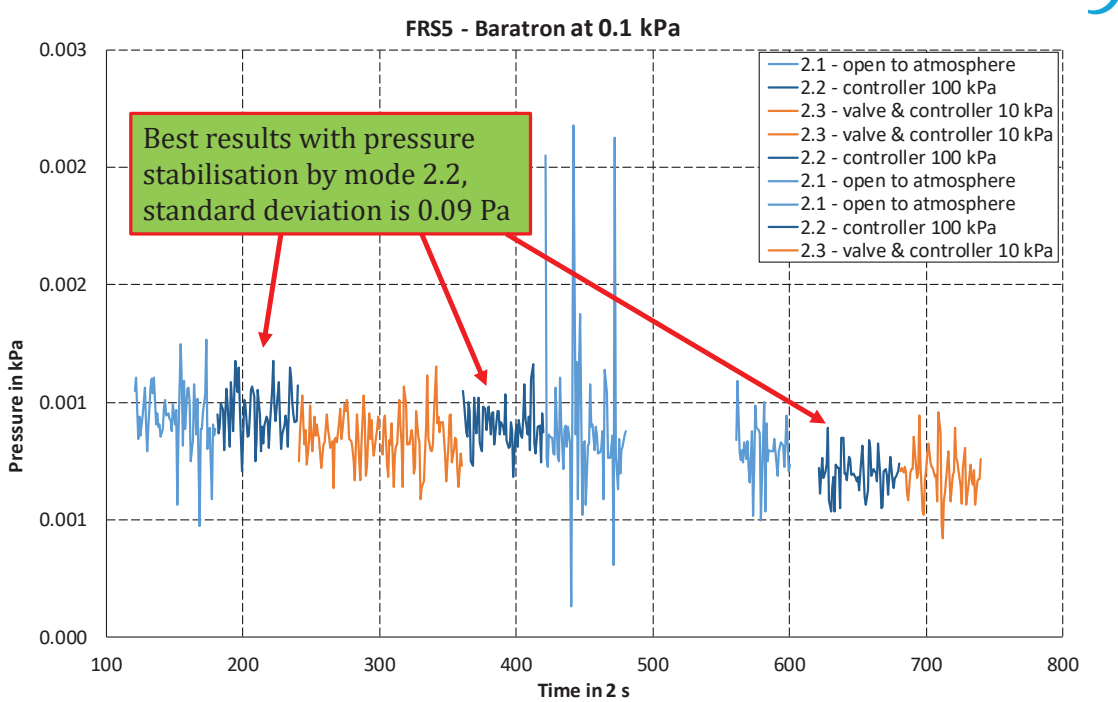
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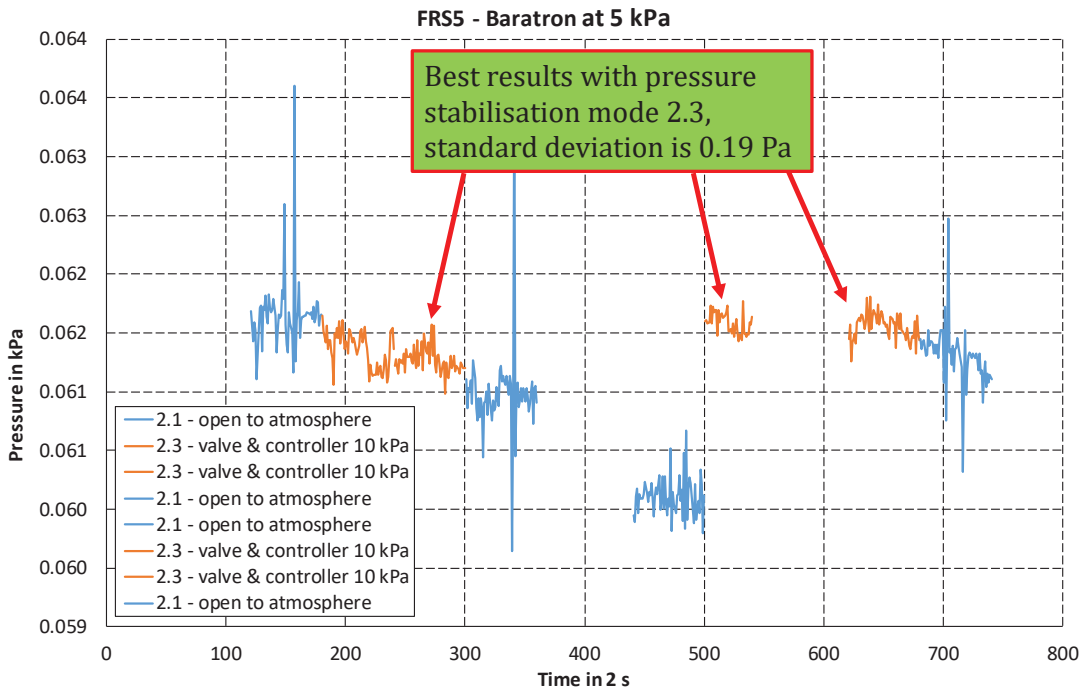
No additional volume: FRS5 readings at 10 kPa



Additional volume of 1.2 l: Baratron calibration at 0.1 kPa



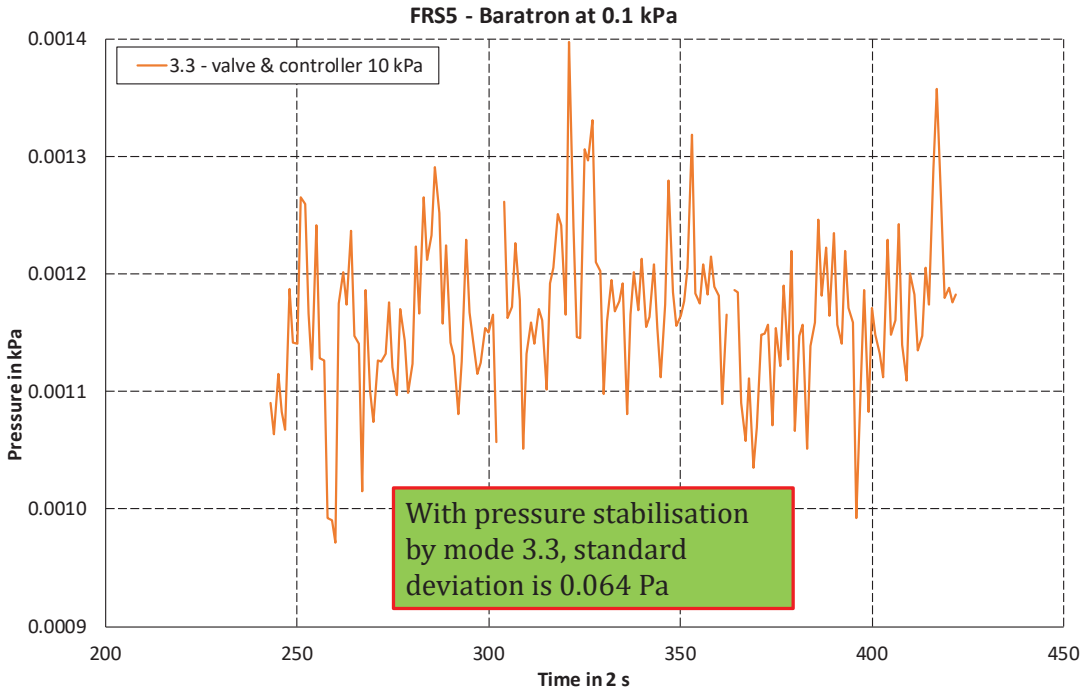
Additional volume of 1.2 l: Baratron calibration at 5 kPa



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20.02.2019 11

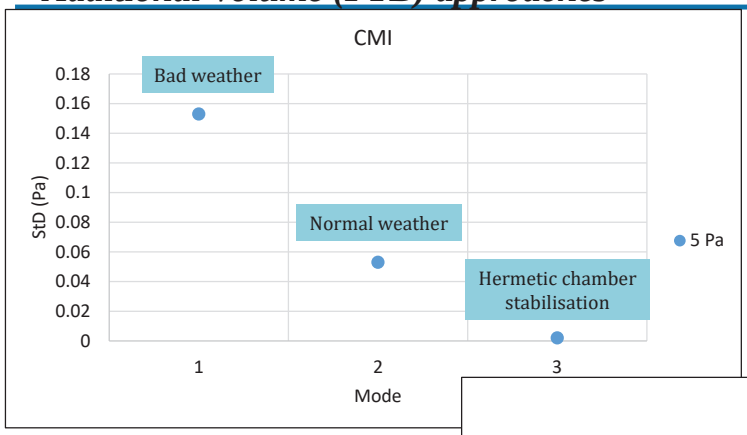
Additional volume of 2.7 l: Baratron calibration at 0.1 kPa

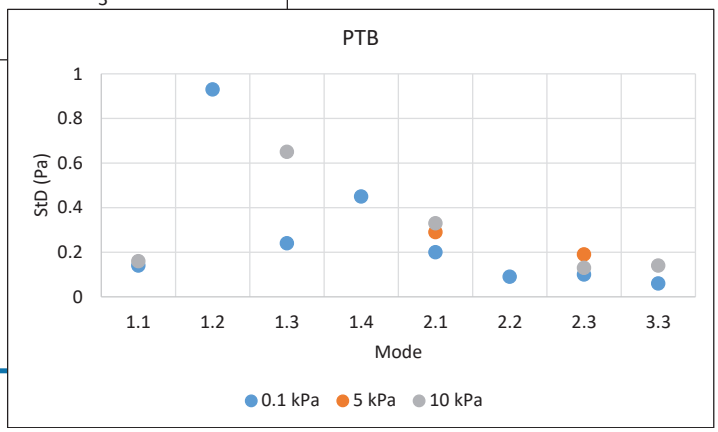


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20.02.2019 12

Comparison of Hermetic Chamber (CMI) and Additional Volume (PTB) approaches





Comparison of Hermetic Chamber (CMI) and Additional Volume (PTB) approaches - summary

Labora-tory	*p* / Pa	*u*(*p*) / Pa (k=1)	StD / Pa (without control of ambient pressure)	StD / Pa (with control of ambient pressure)
CMI	5	0.020	0.15	0.002
PTB	100	0.047	0.14	0.06 (method 3.3)
	5000	0.10		0.19 (method 2.3)
	10000	0.16	0.16	0.13 (method 2.3)

Conclusions and outlook

- Methods using hermetic chamber or pressure-controlled additional volume were tested to achieve atmospheric-fluctuations-free calibrations of the low gauge pressures.
- With both methods, calibration uncertainties below 3 Pa + $2 \cdot 10^{-4} \cdot p$ are always achievable even at unstable weather conditions and in industrial conditions.
- A possibility has also been demonstrated to calibrate FPG in gauge mode independently of ambient pressure.
- The methods are applicable to mechanical and electronic low gauge pressure measuring instruments. Preliminary, an estimation shows that uncertainties as low as 10 mPa + $10^{-5} \cdot p$ (k = 1) can be achieved in the future.

EMPIR
EURAMET
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

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Thank you for your attention!

Any questions?

Traceable methods for calibration of Force balanced Piston Gauges (FPGs) in absolute and gauge modes

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WP3: Task3.3

Traceable methods for calibration of Force balanced Piston Gauges (FPGs) in absolute and gauge modes

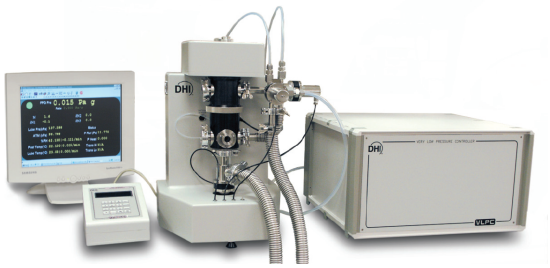
Djilali Bentouati

Mark Plimmer

Pres2Vac - Final Workshop 16th – 17th May 2018, RISE – Borås, Sweden

Traceable methods for calibration of Force balanced Piston Gauges (FPGs) in absolute and gauge modes

Activities A3.3.1 to A3.3.4



- Range 0 to +15 kPa
- Target uncertainties (k=2)
  - U_p = 10 mPa + 3×10^{-5} × p (gauge)
  - U_p = 15 mPa + 3×10^{-5} × p (absolute)

The extra 5 mPa is due to the residual vacuum uncertainty

To reach these levels, you need *primary* standards

Dead-weight balance

Mercury column manometer

3

Methods

- Gauge mode
  - Direct comparison with the standard
  - Comparison using capacitance diaphragm gauge (CDG) as a zero indicator
- Absolute mode
  - Very difficult to perform a direct comparison (see later)
  - Comparison using CDG as a zero indicator

Measurements performed by :

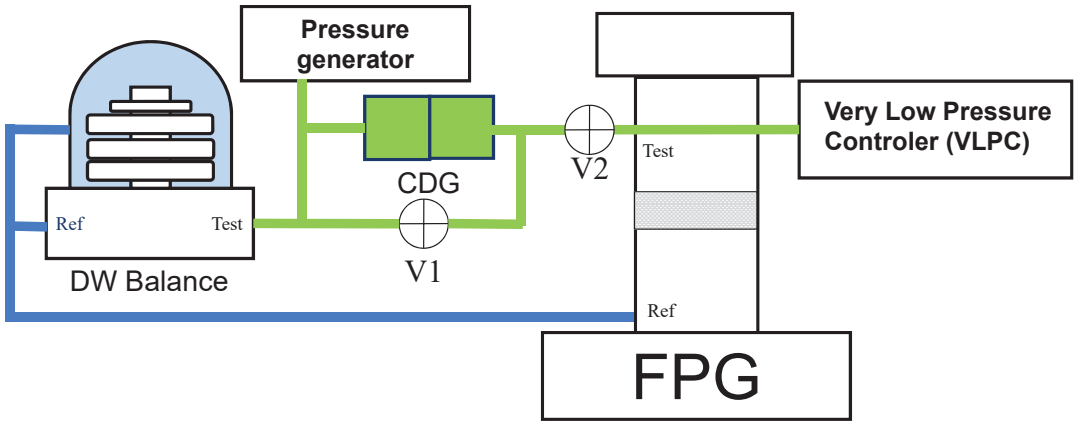
4

Pre-calibration steps

- Calibrate
  - Pressure balance masses (1.5×10^{-6} M) k=2
  - Internal calibration mass of FPG (3 mg)
  - CDGs (0.1%)
- Check
  - Thermometers (0.1 K) k=2
  - Linearity of load cell (< 10^{-6})
- Perform the internal calibration of the FPG
- Zero the FPG

5

Comparison using CDG as a zero indicator (gauge mode)

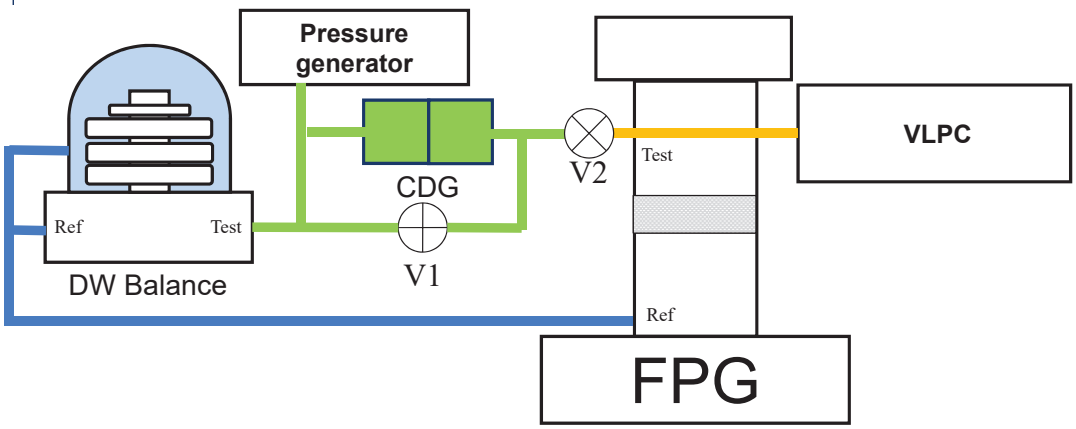


Conflict of pressure regulation by two different instruments ⟹ Need CDG

⚠ Reference ports must be connected together to limit the effect of atmospheric pressure fluctuations on the FPG reference chamber.
This advantage outweighs the perturbation of the DW balance caused by the link.

6

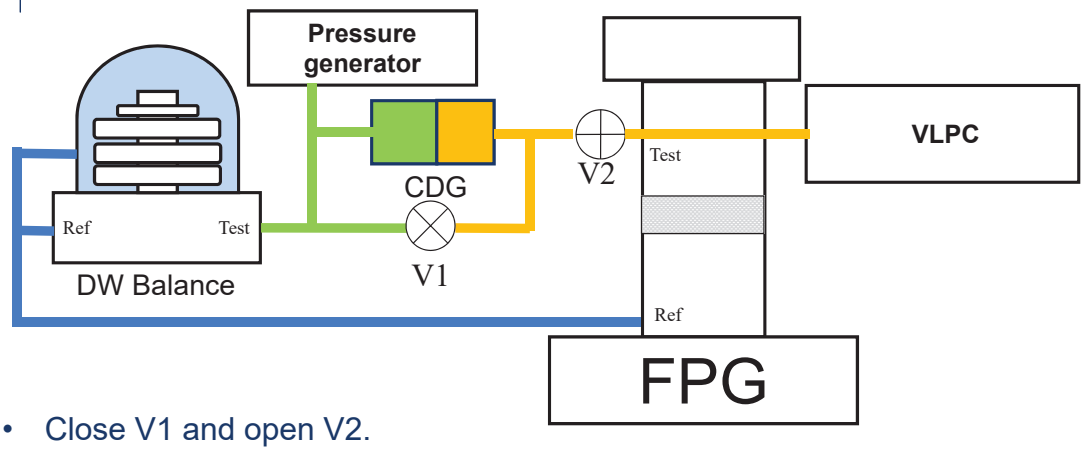
Step 1 : Determination of the zero of the CDG



- Close V2 and open V1.
- In this configuration, the pressure is the same on both sides of the gauge diaphragm.
- Now apply the desired pressure to both the standard and the FPG.
- Wait 1 minute for the DW balance to stabilize then note the CDG reading $p_{CDG\,0}$.

7

Step 2 : Pressure differential between both instruments



- Close V1 and open V2.
- Each instrument generates a pressure on its own side of the diaphragm.
- The CDG measures the pressure differential between them.
- This should be < 1 Pa for the uncertainty due to the CDG to be negligible (< 1 mPa).
- Note the CDG reading p_{CDG}, *the temperature of the dead-weight balance, the FPG reading and all its associated parameters (N,δN1, δN2, δN3 and temperature).*

8

Step 3 : Calculation of the reference pressure

- The reference pressure is calculated with the pressure delivered by the standard and the indications of the CDG

$$p_{ref} = p_{bal} + (p_{CDG} - p_{CDG\,0})$$

- All the steps are repeated for the ensemble of pressure points.

Remarks:

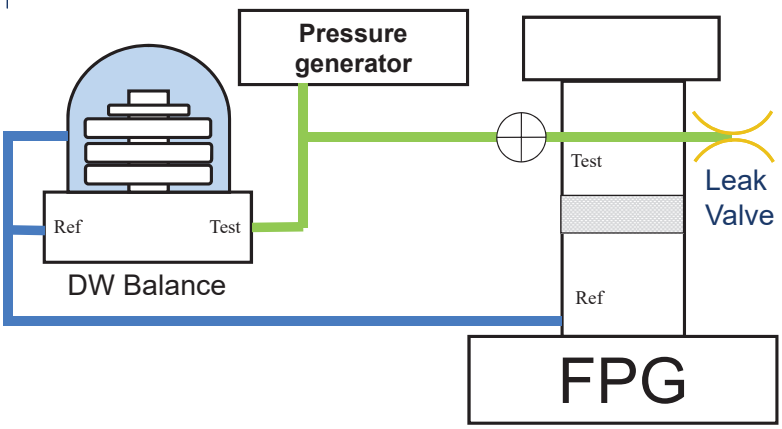
The stabilization time needed to obtain good measurements is > 60 s.

At least 30 s averaging time is needed to compensate the VLPC regulation cycle.

During this averaging time, the pressure stability of the FPG is ≈ 200 mPa.

9

Direct Comparison



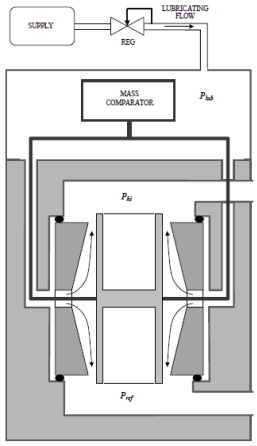


Figure taken from Fluke FPG8601 Operation Manual

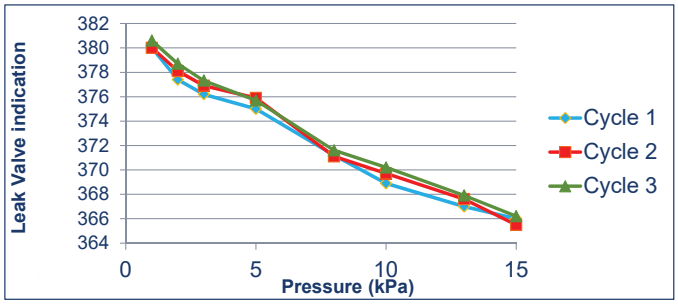
- Pressure regulated only by dead-weight balance.
- VLPC replaced by manually adjustable leak valve.
- This leak allows one to compensate the flow due to lubricating pressure.

10

Direct calibration procedure

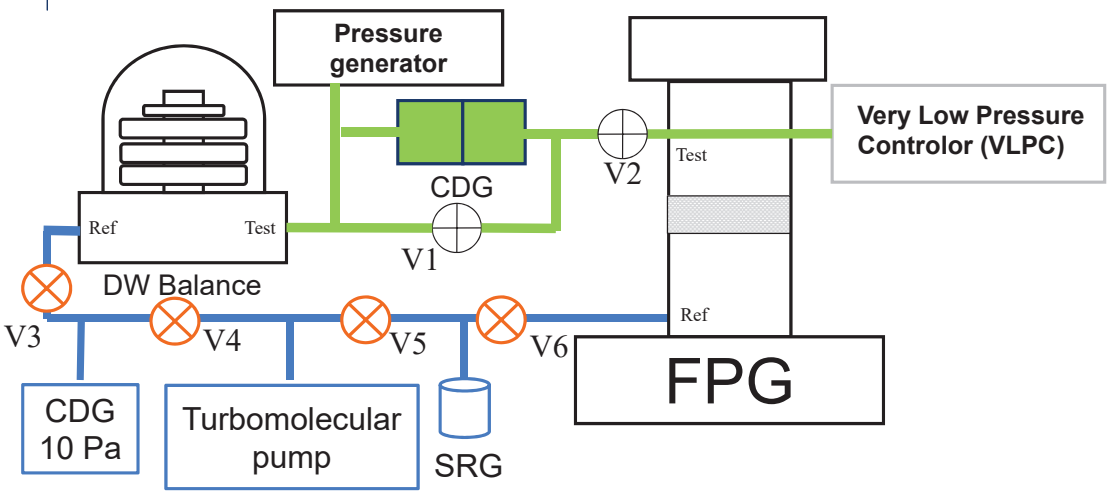
- Isolate the measurement and reference chambers of the FPG.
- Apply chosen pressure with the dead-weight balance.
- Close the valve and adjust leak valve to obtain a stable FPG reading.
- Open the valve and wait for stabilization.
- Note FPG reading and parameters.
- Repeat the operation for each desired pressure.
- It helps save time if one uses a leak valve with a digital position display.





11

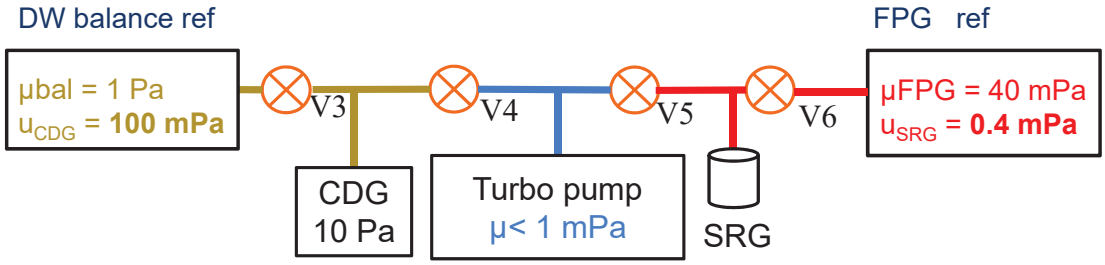
Calibration in absolute mode



- Between the test ports the set-up is the same as for gauge mode.
- In absolute mode, the reference ports cannot be connected.
  Residual pressures : 40 mPa for FPG and 1 Pa for the DW balance
- Uncertainty component due to residual pressures can be eliminated.

12

How to reduce the contribution of residual pressure measurements



- When an SRG is used to measure the FPG residual pressure, its contribution to the uncertainty budget is negligible.
  - Neverless the zero of the SRG must be checked.

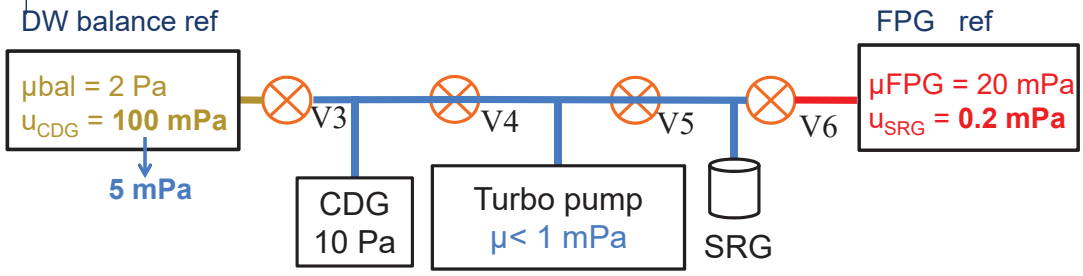
- Uncertainty due to the residual pressure of the balance 100 mPa.

  To reduce this contribution, drifts of the CDG have to be addressed:

  - Drift of the sensitivity by calibration before and after measurement.
  - Drift of zero checked before each measurement cycle.

13

Method to check the zero



Before and after each measurement cycle:

- Isolate CDG and SRG from instruments by closing V3 and V6.
- Open the valve V4 to connect the CDG to the pump.
- Open the valve V5 to connect the SRG to the pump.
- Ensure the zero of SRG is lower than 1 mPa.
- Note the value indicated by the CDG.
- Correction applied to CDG is mean of zeros before and after.

14

Calculation of effective piston cylinder area A_0

Full calculation

Compulsory for very first determination

- $$A_0 = \frac{K_{cal}\cdot(N_m+\delta N1+\delta N2+\delta N3)}{p_{ref}\cdot\left[1+(\alpha_p+\alpha_c)\cdot(t-20)\right]}$$

Simplified calculation

Used for 2nd and subsequent determinations

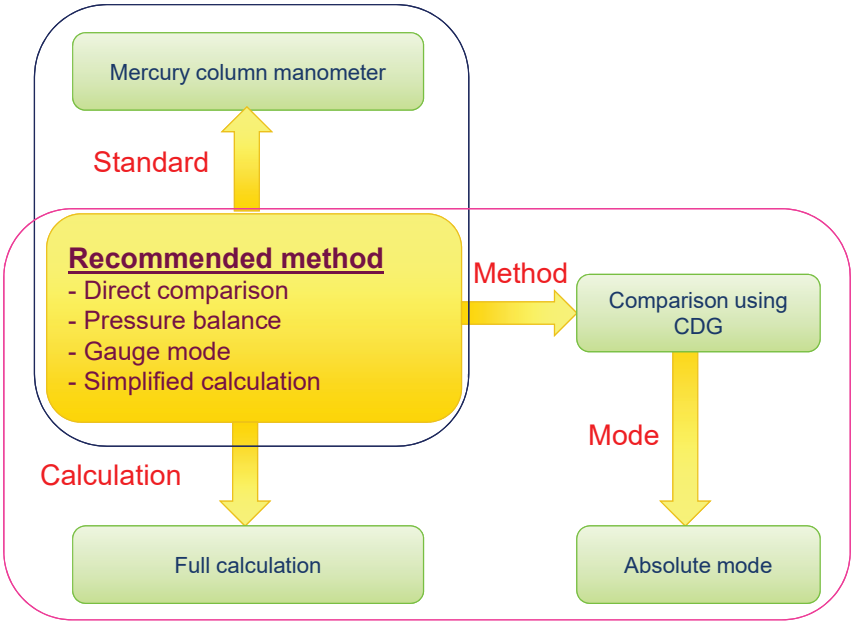
$$A_0 = A_{FPG}\cdot\left(1-\frac{\Delta p}{p_{ref}}\right)$$

K_{cal}: Load cell calibration coefficient (N.Pa^{-1})
N_m : Indication of the load cell of the FPG
δN : Corrections due to initial condition changes
p_{ref}: Reference pressure
α_p and α_c: Linear thermal expansion coefficient of the piston and of the cylinder in K^{-1}
t: Temperature of the piston cylinder assembly in degrees Celsius
A_{FPG} : Previous value of effective area
Δp: Difference between reference pressure and FPG reading.

15

Validation of traceable methods

To validate the different calibration methods, comparisons were performed by changing one parameter at a time.

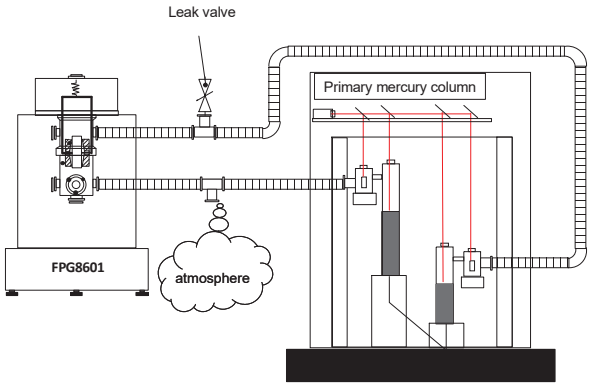


16

Comparison using a mercury column manometer

Calibrations were performed using the CEM primary mercury column manometer. Results were compared to those obtained using a Ruska balance.

Range : 1 to 15 kPa



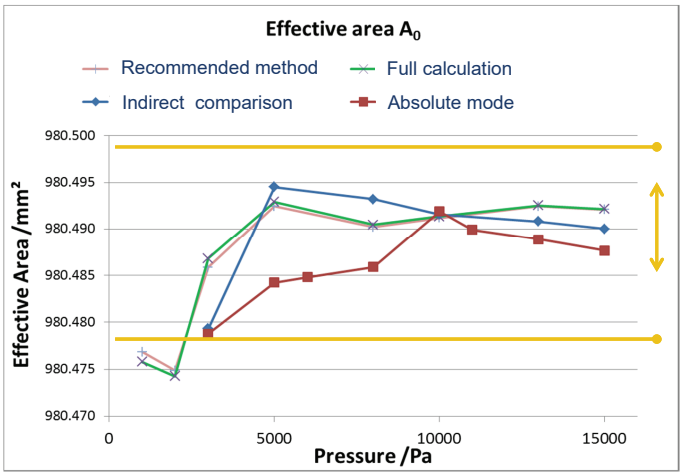
Standard	$A_{0(20\,°C)}$ (m²)	U($A_{0(20\,°C)}$) (k=2) (m²)	W ($A_{0(20\,°C)}$) (k=2)
Ruska balance	$980.453 \cdot 10^{-6}$	$0.034 \cdot 10^{-6}$	$35 \cdot 10^{-6}$
Mercury column	$980.458 \cdot 10^{-6}$	$0.040 \cdot 10^{-6}$	$41 \cdot 10^{-6}$

17

Results of the comparisons

For each configuration, the effective area is calculated for all measurement points.

The points at 1 and 2 kPa were obtained using the pressure balance without its mass bell and are excluded from the calculation of the effective area.



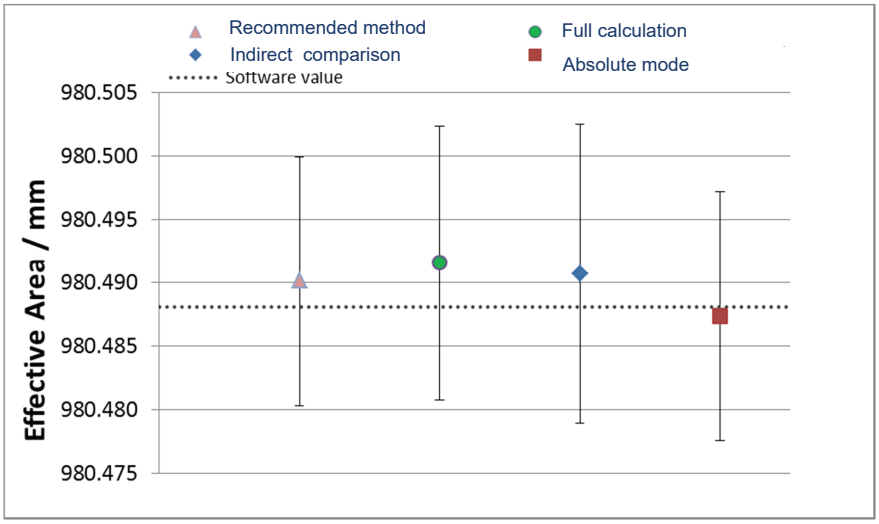
For each measurement point, the differences in the determined effective area never exceed 11 ppm regardless of the configuration.

18

Results of the comparisons

Effective areas calculated from weighted mean of the values obtained between 5 kPa and 15 kPa.

Expanded uncertainties range from 10 to 12 ppm.



Very good agreement between the methods

19

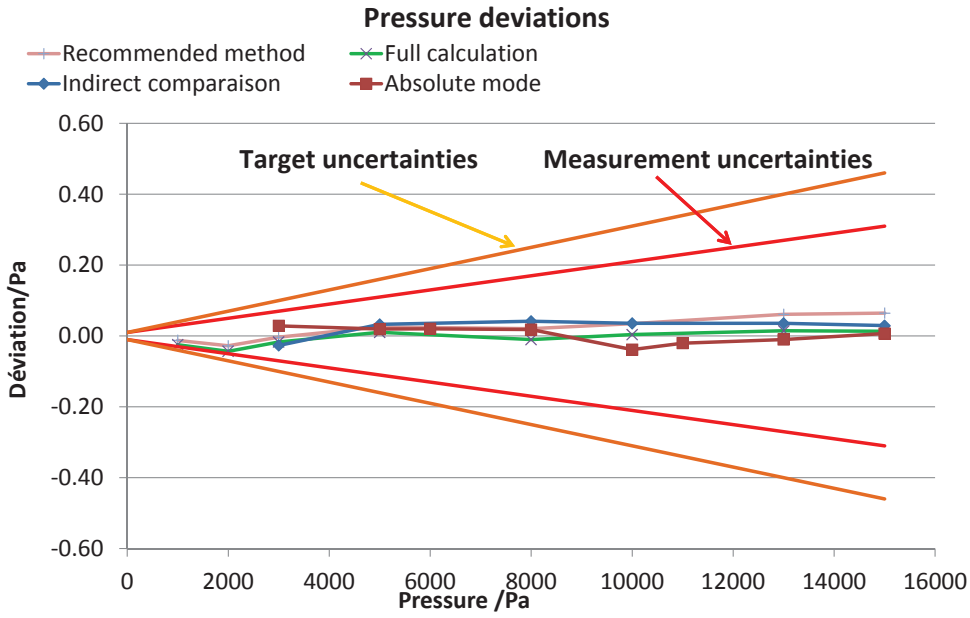
Uncertainties

The calibration of FPGs consists in the determination of its PCA effective area.
The same uncertainty components are included in all methods and measurement modes.
The expanded uncertainty is 11 ppm.
The main contributions come from the fitting and the calibration standard.

Component	Standard uncertainty 10^{-6} x A0
Standard	5.0
Fitting	1.9
Temperature	1.0
Verticality of the piston	0.70
Head correction	0.11
Expanded uncertainty (k=2)	**11**

20

Results for pressure



Results for different methods and configurations agree.

21

Results for pressure

Component	Standard uncertainty	
	mPa	10^{-6} x p
Repeatability	5.0	4.4
Resolution	0.29	
Effective area		5.5
Calibration Mass		2.6
Temperature		1.0
Linearity of the cell		1.0
Head correction		0.11
Verticality of the piston		0.70
Expanded uncertainty (k=2)	**10**	**15**

22

Conclusion

- We have investigated traceable methods for the calibration of FPGs:
  - Dead weight balance or mercury manometer
  - Direct or indirect comparison with the standard
  - Gauge or absolute mode
  - Complete or simplified calculation of area
- Very good mutual agreement between methods
- Traceability of the methods is verified
- Uncertainty for pressure is 10 mPa + 15 ppm (absolute + gauge)

  Target uncertainties were 10 mPa + 30 ppm (gauge)
  15 mPa + 30 ppm (absolute)

Thanks for your attention

Measurement of positive and negative gauge pressures using an absolute pressure balance

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[2] Laboratoire national de métrologie et d'essais (LNE), France

WP3: Task 3.2
Measurement of positive and negative gauge pressures using an absolute pressure balance.

Mark Plimmer

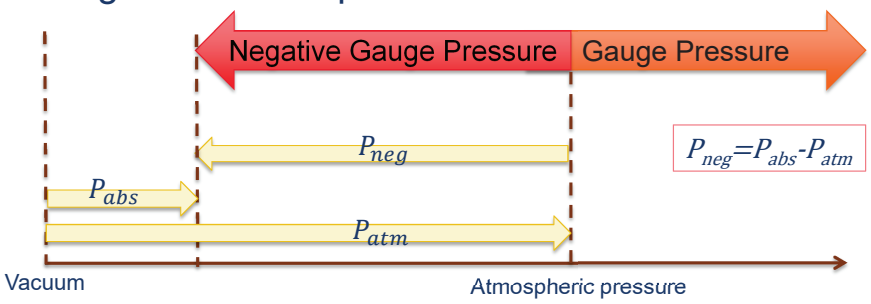
Djilali Bentouati

Pres2Vac - Final Workshop 16th – 17th May 2018, RISE – Borås, Sweden

Task 3.2: Development of methods for negative gauge pressure calibration.

- Very few primary standards had been developed for the range -100 kPa to 10 kPa.
- Needed to extend methods initially developed for positive gauge pressure to negative gauge pressure.
- Target accuracies in industry have increased in recent years: primary laboratories have had to improve their measurement capabilities by using pressure balances.

3 How to measure negative gauge pressure using an absolute pressure balance



Two possible ways to perform a calibration in negative pressure range:

- By comparison with a gauge pressure standard
  - ➢ Working conditions are different than for normal use:

  ⟹ Method had to be modified.

- By comparison with two absolute pressure standards:
  - ➢ Use of a second standard adds an uncertainty component

  ⟹ This contribution can be eliminated by respecting a particular procedure.

Ambient conditions (reference pressure) have to be controlled to reduce instability and its contribution to the uncertainty budget.

4 Control of ambient conditions

Method to achieve constant reference ”atmospheric” pressure

Constant pressure reservoir
Standard and device under test (DUT) are connected to the volume.
Thermal insulation can be added.



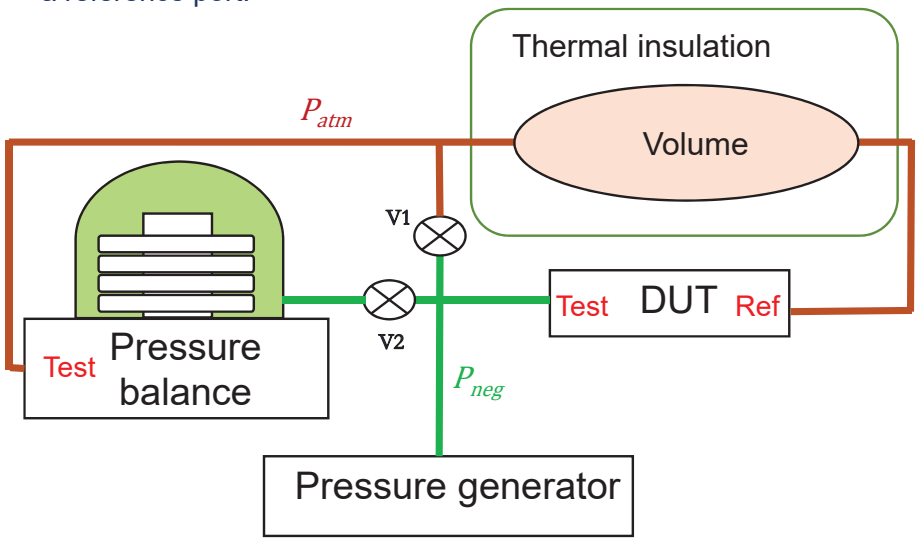
Pressure stable within ±1 mbar over 12 hours.

If the instrument is equipped with a reference “port”, the use of such a volume is recommended.

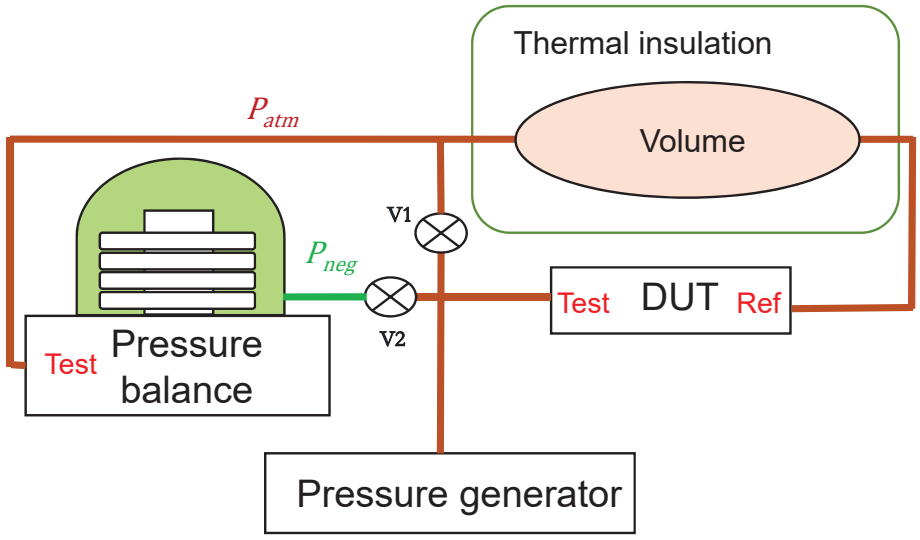
Calibration is thus performed independently of ambient conditions.

5 Method by applying negative pressure in bell jar

- Pressure balance used has to be designed to measure absolute pressure.
- Near-constant atmospheric pressure can be obtained using a volume.
- Negative pressure is applied to the bell jar port while the test port is used as a reference port.

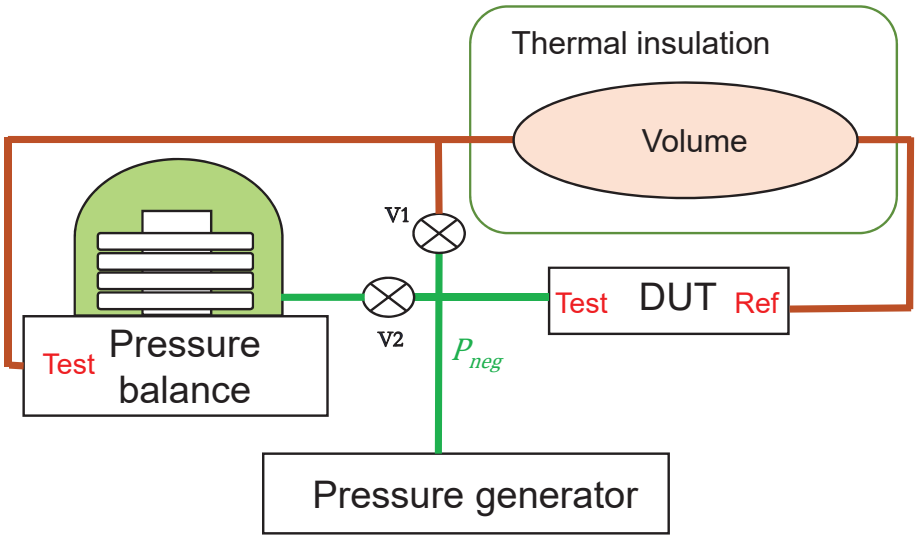


6 Step 1: Zeroing the manometer under test



- Close V2.
- Open V1 and adjust the reference atmospheric pressure.
- After 2 min of stabilization, zero the DUT.
- Repeat this step before each measurement cycle.

7 Step 2: Apply the negative gauge pressure



- Close V1.
- Open V2 and adjust the pressure of the balance to achieve equilibrium.
- After 2 min of stabilization, note the value of the DUT and balance parameters.
- Perform measurement cycles.

8 Step 3: Calculation of standard pressure

- Calculation of reference pressure is different than for normal use :
- Apparent masses are calculated using density of fluid.

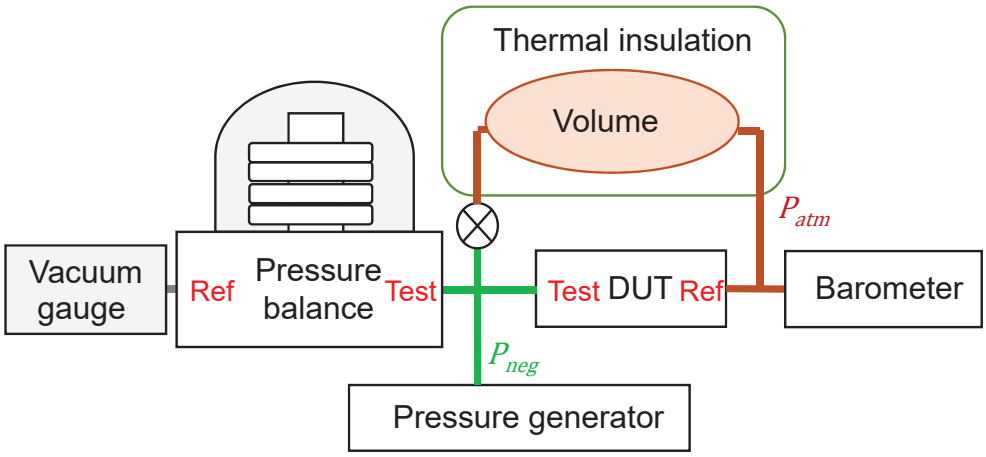
$$P_{neg} = -\frac{\sum_i \left[m_i \left(1 - \frac{\rho_f}{\rho_{m_i}} \right) \cdot g \right]}{A_0 \left[1 + \left(\alpha_p + \alpha_c \right) \cdot \left(t - t_{ref} \right) \right] \left[1 + \lambda \cdot \left| P_{neg} \right| \right]} + \left(\rho_f - \rho_a \right) \cdot g \cdot \Delta h \ .$$

- Large volume to be pumped makes it to hard to achieve pressure < -500 kPa.

➔ A second pump equipped with a valve can help ease pressure generator.

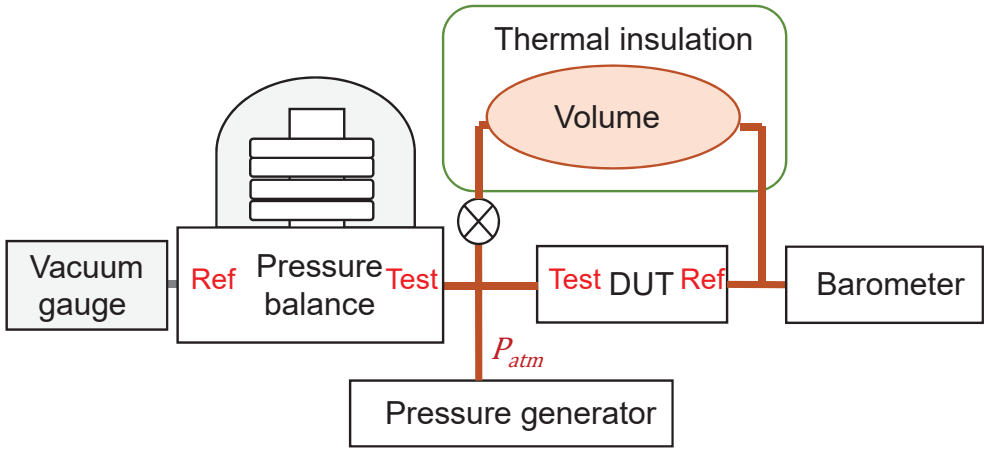
9 Method using a pressure balance and a barometer

- In this configuration, the pressure balance is used in absolute mode.



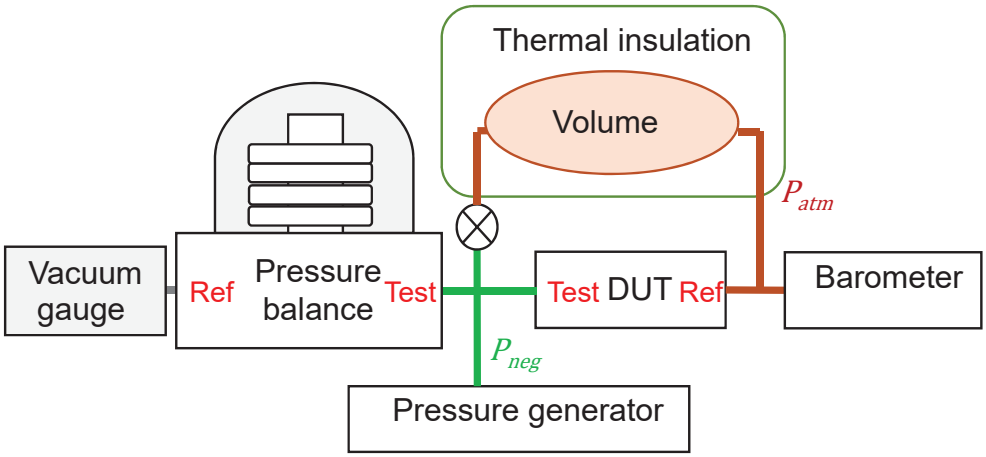
- Barometer and DUT must be placed at the same level to limit head correction.
- A particular procedure must be respected to cancel uncertainties due to barometer and vacuum gauge.

10 Step 1: Determine accuracy error of the barometer



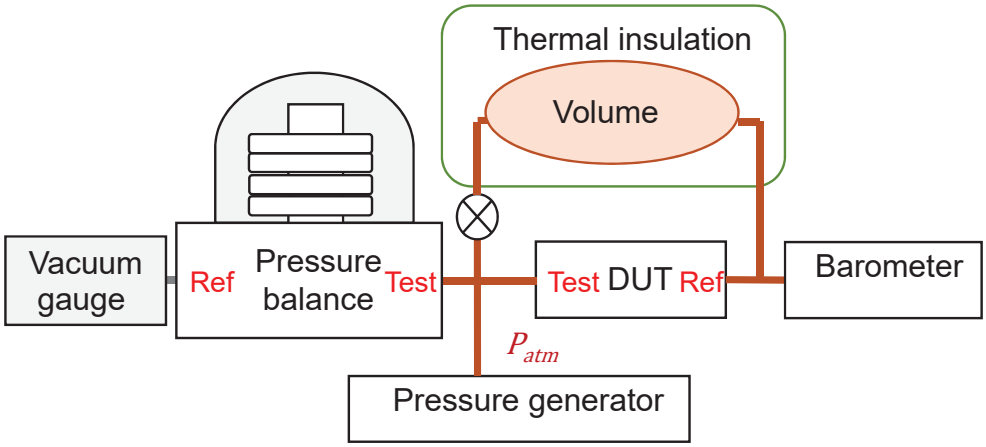
- Open valve and apply atmospheric pressure P_{abs0} .
- After 2 min stabilization, perform the zero of the instrument.
- Note the indication of the barometer $P_{m0.}$
- Calculate the accuracy error of the barometer using : $E_0 = P_{abs0} - P_{m0.}$
- Close the valve.
- From this time on, the valve must be remain closed until the end of calibration.

11 Step 2: Negative pressure measurement



- Apply the corresponding pressure P_{abs} using the relation
  $$P_{abs} = P_{neg} - P_{atm}$$
- Note that P_{atm} is the pressure in the external volume and **not** the laboratory.
- Allow 2 min stabilization
- Note the indication of the barometer P_m and the balance parameters.

12 Step 3: Recheck the accuracy error



- Apply the atmospheric pressure P_{abs0}
- After 2 min stabilization, open the valve.
- Note the barometer reading P_{m0}
- Recalculate the accuracy error of the barometer using : $E_0 = P_{abs0} - P_{m0}$
- The difference between the 2 determinations (≤ 0.1 Pa for the best instruments) increases the uncertainty budget.

13 Calculation of the negative reference pressure

- From the definition of the negative pressure :

$$P_{neg} = P_{abs} - P_{atm}$$

where $P_{atm} = P_m + E_0$ and $E_0 = P_{abs0} - P_{m0}$

Thus

$$P_{neg} = \overbrace{\frac{\sum_i [m_i \cdot g]}{A_0[1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})][1 + \lambda \cdot P_{abs}]}}^{P_{abs}} - \overbrace{\frac{\sum_i [m_{i0} \cdot g]}{A_0[1 + (\alpha_p + \alpha_c) \cdot (t_0 - t_{ref})][1 + \lambda \cdot P_{abs0}]}}^{P_{abs0}}$$

$$+(\mu - \mu_0) - (P_m - P_{m0}) + (\rho_f - \rho_{f0}) \cdot g \cdot \Delta h.$$

< 0.2 Pa $\quad$ < 1mbar

$u_{\Delta\mu} = 0.005$ Pa $\quad$ $u_{\Delta Pm} = 0.05$ Pa

14 Uncertainties

For the same balance, the expanded uncertainties are :

- **0.20 Pa + $6.6 \cdot 10^{-6} \cdot |P_{neg}|$ with barometer.**
- **0.10 Pa + $7.0 \cdot 10^{-6} \cdot |P_{neg}|$ when pressure is applied in the bell jar.**

Main contributions:

- effective area $2.8 \cdot 10^{-6} \cdot |P_{neg}|$
- its instability $3.0 \cdot 10^{-6} \cdot |P_{neg}|$

Using the aforementioned procedure, the contribution of the barometer is reduced to 0.05 Pa.

If this barometer is replaced by a second balance, the corresponding contribution can be eliminated.

15 Comparison of the methods

- Methods were implemented in the same laboratory in investigate their advantages and drawbacks.

	Bell jar method	Barometer method
Calibration time (1 cycle of 12 points)	60 min	40 min
Ease of implementation	Hard to stabilize pressure	A specific protocol must be respected
Cost	Expensive Absolute pressure balance Extra roughing pump	Very expensive Absolute pressure balance High accuracy barometer
Control of ambient conditions	Volume	Volume
Automation	*Customized* software	*Commercial* software
Level of uncertainty	National metrology institute	National metrology institute

16 Conclusion

- Methods to control ambient conditions allow one to perform calibrations in measurement mode independently of instruments involved and atmospheric pressure value and its fluctuations.

- Using them National metrology institutes are able to provide uncertainties for negative gauge pressure measurements low enough to allow industry to have a level of uncertainty of $1.10^{-4} \times |P_{neg}|$.

- With such methods, calibrations performed using commercial instruments can now be automated.

- Factors such as types of instruments to be calibrated, calibration ranges and laboratory altitude should be taken into account when one decides on which methods to implement.

- A Euramet Calibration Guide is in preparation for later this year.

Thanks for your attention

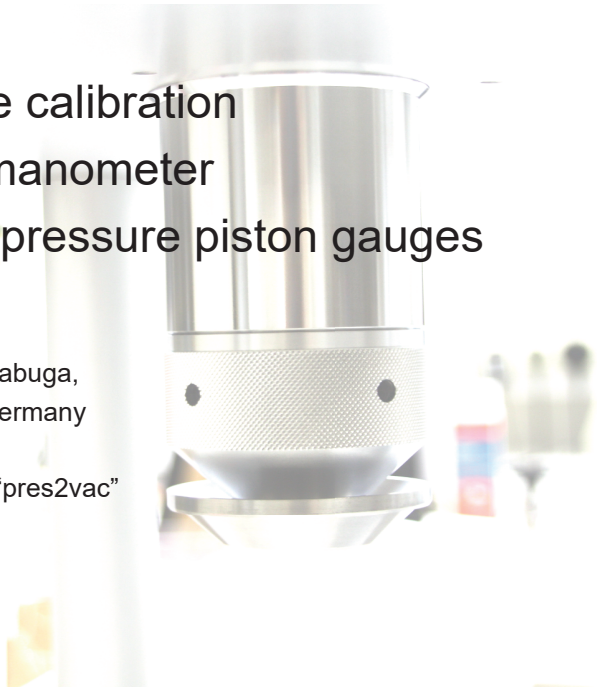
Negative gauge pressure calibration by means of a mercury manometer and conventional gauge pressure piston gauges

Sven Ehlers, Thomas Konczak, Wladimir Sabuga

Physikalisch-Technische Bundesanstalt (PTB), Germany

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Negative gauge pressure calibration by means of a mercury manometer and conventional gauge pressure piston gauges



Sven Ehlers, Thomas Konczak, Wladimir Sabuga,
PTB Braunschweig, Pressure laboratory, Germany

Final Workshop, EMPIR Research Project “pres2vac”

Borås, Sweden
May 16th-17th, 2018

Neg. Gauge?

Devices Under Calibration

PTB Standards

Differences

Summery

Neg. Gauge?

PTB

Negative gauge pressure - Facts

Definition (DIN 1314):

$$p_e = p_{abs} - p_{amb}$$

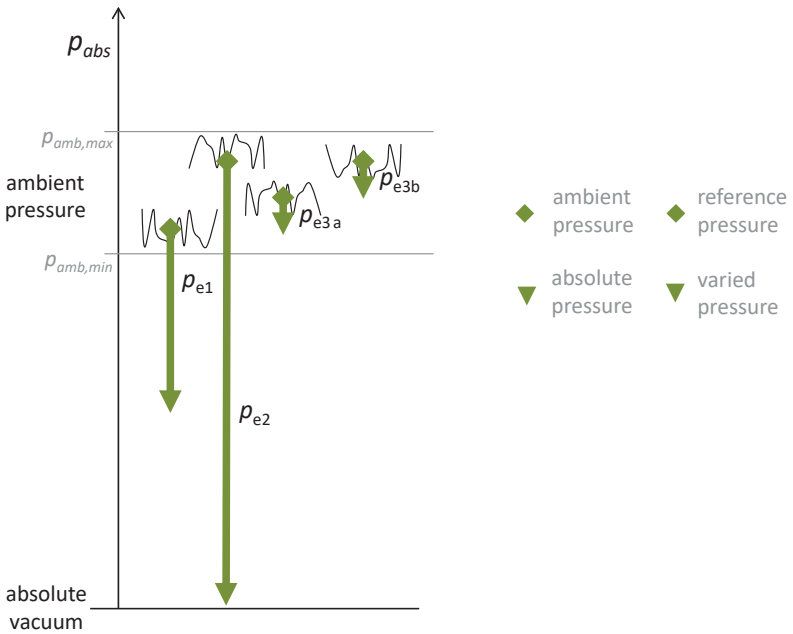
Its value is negative, when $p_{abs} < p_{amb}$.

Negative gauge pressure - Facts

Ambient pressure p_{amb} is:

- **reference** pressure
- geographical **height depending**
- **changing**
  within 100 hPa over days
  (at PTB, between 950 hPa and 1050 hPa)
- **fluctuating**
  closing doors, wind gusts: up to ±2 Pa

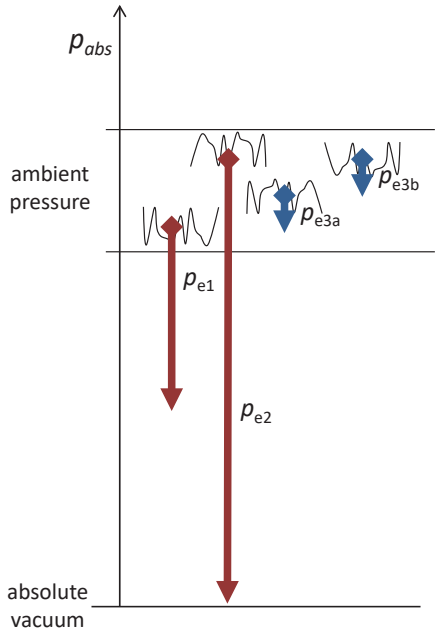
Negative gauge pressure - Facts



Devices Under Calibration (DUCs)

PTB

DUC measurement ranges



p_{e1}, p_{e2} Negative gauge pressure

p_{e3a}, p_{e3b} Small negative gauge pressure

Small when:
Maximum absolute pressure value is less than window of local ambient pressure changes.

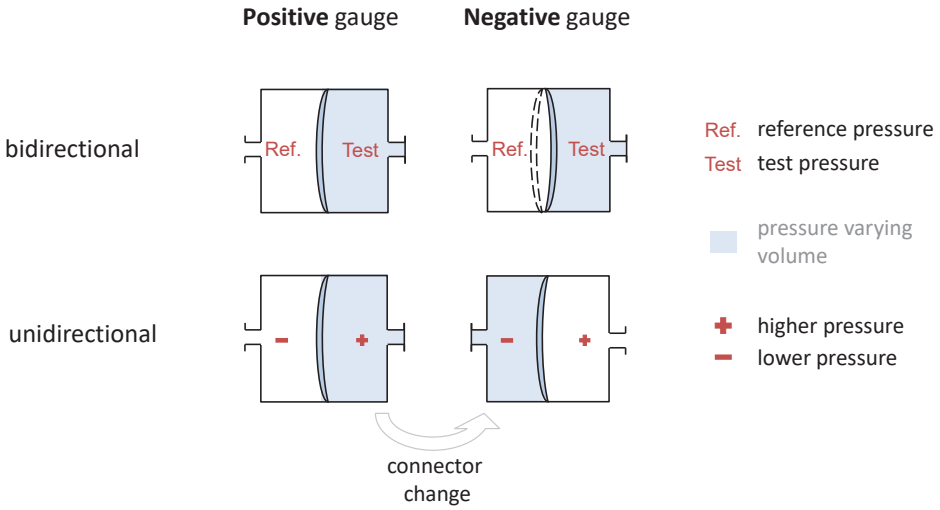
$|p_{DUC}|_{max} \leq (p_{amb,max} - p_{amb,min})$

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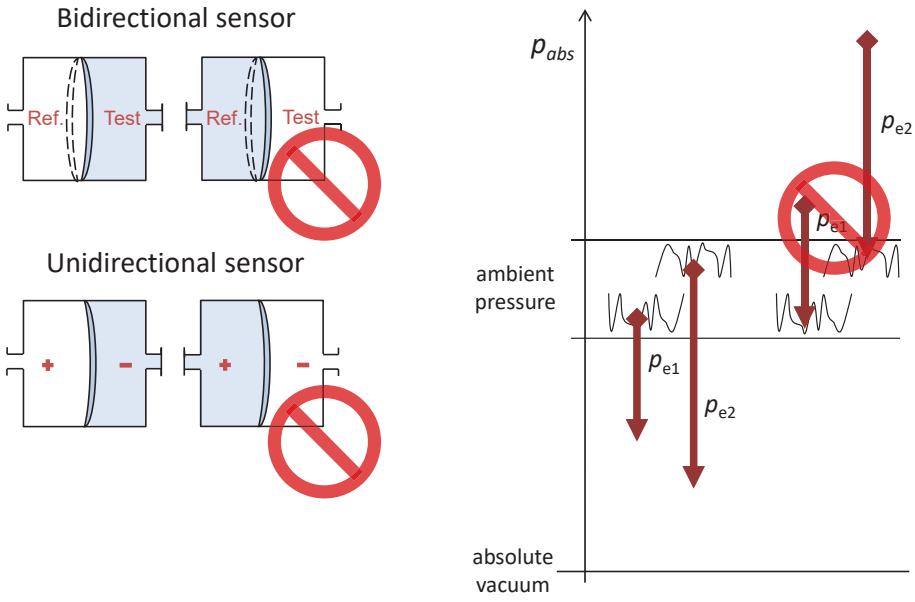
Bidirectional or unidirectional DUCs

Same basic principle for every type of DUC.
i. g.: membrane type gauges



Calibration without vacuum pump?

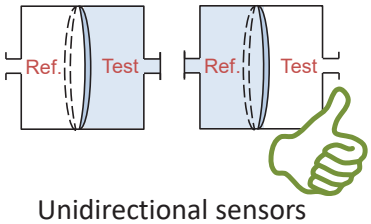
Negative gauge pressure sensors



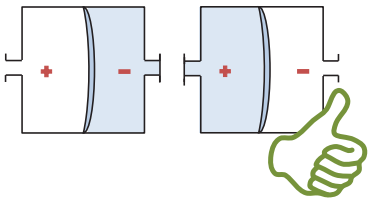
Calibration without vacuum pump?

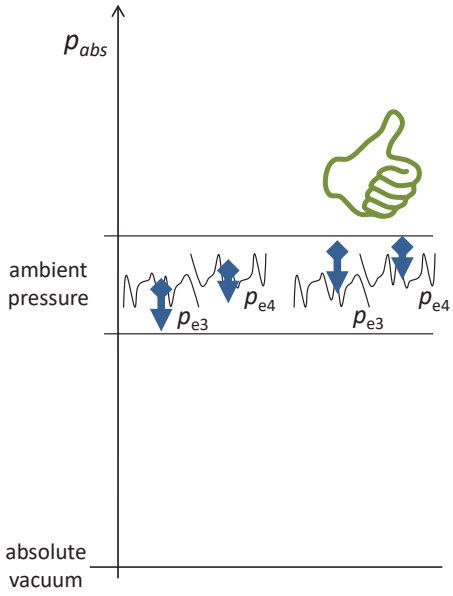
Small negative gauge pressure sensors

Bidirectional sensors



Unidirectional sensors





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PTB Standards

Negative gauge pressure calibrations at PTB

Customers:

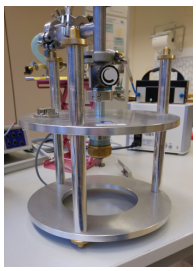
- Accreditated pressure laboratories (>80 in Germany)
- Other NMIs
- PTB internal

Negative gauge pressure calibrations at PTB

Devices under calibration (DUCs):

- Gauge pressure balances (with hanging piston extension)
- Absolute pressure balances (with bell jar)
- Bidirectional or negative pressure gauges





Negative gauge pressure calibrations at PTB

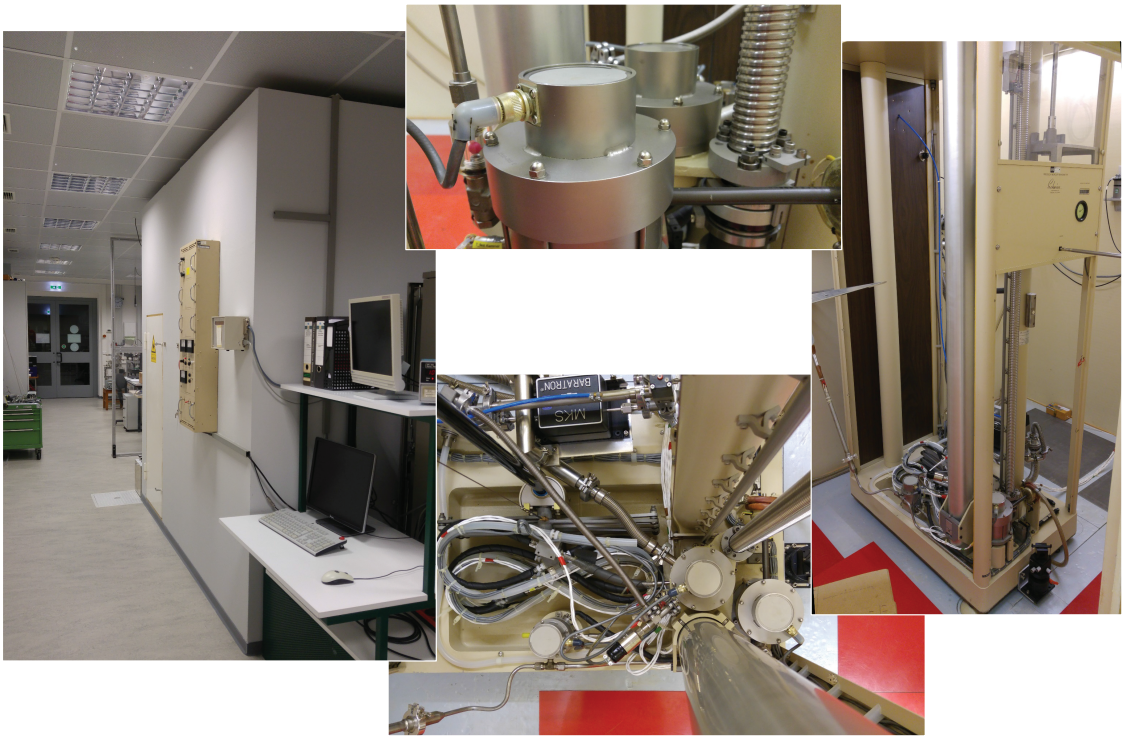
Standards:

- Mercury manometer
- Maihak pressure balance (5 cm², hanging piston)
- Ruska pressure balance (3.3 cm², bell jar pressure method)
- Two UDKMs

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PTB Mercury manometer (Schwien)

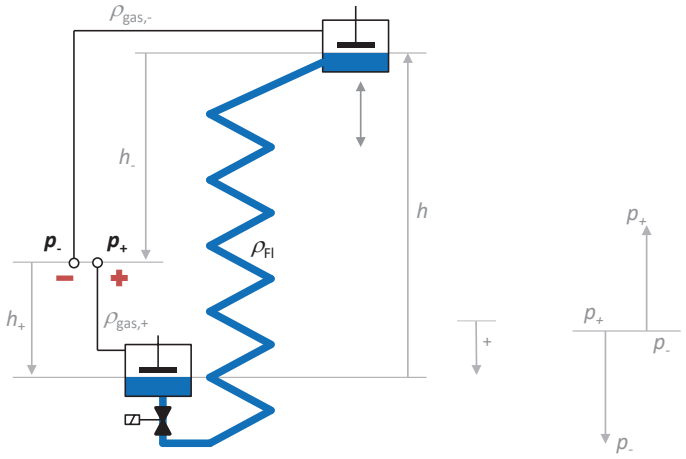


Liquid column manometer

Positive gauge pressure: $p_e = p_+ - p_- = \rho_{fl}\, g\, h - \rho_{gas,+}\, g\, h_+ - \rho_{gas,-}\, g\, h_-$

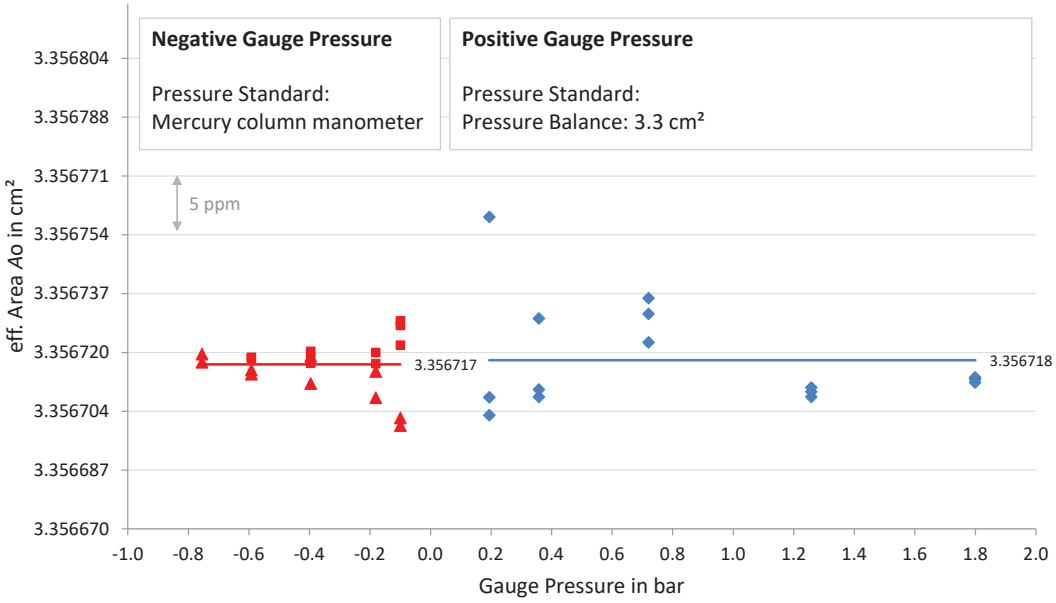
Negative gauge pressure: $p_e = p_- - p_+ = -\rho_{fl}\, g\, h + \rho_{gas,+}\, g\, h_+ + \rho_{gas,-}\, g\, h_-$

Differences: Faktor (-1) and fluid density levels

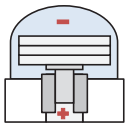


Liquid column manometer results

Calibration of a Ruska pressure balance (3.3 cm²)

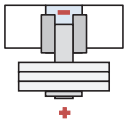


Pressure balances in negative gauge mode



„Pressure under the bell jar“ method:

- In range of low negative gauge pressure, weak sealing abilities of the bell jar can occur.
- For high negative gauge pressure, a high piston fall rate can limit the calibration range.

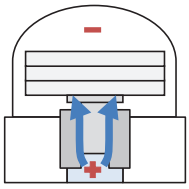


„Hanging piston“ method:

- For high negative gauge pressure, a high piston fall rate can limit the calibration range.
- In some cases, need for an additional mass adapter and mass set.

Why is the piston sinking?

Positive gauge pressure



pressure varying volume
higher pressure
lower pressure

Some gas is leaving the pressure varying volume through the PCU gap, Following the negative pressure gradient.

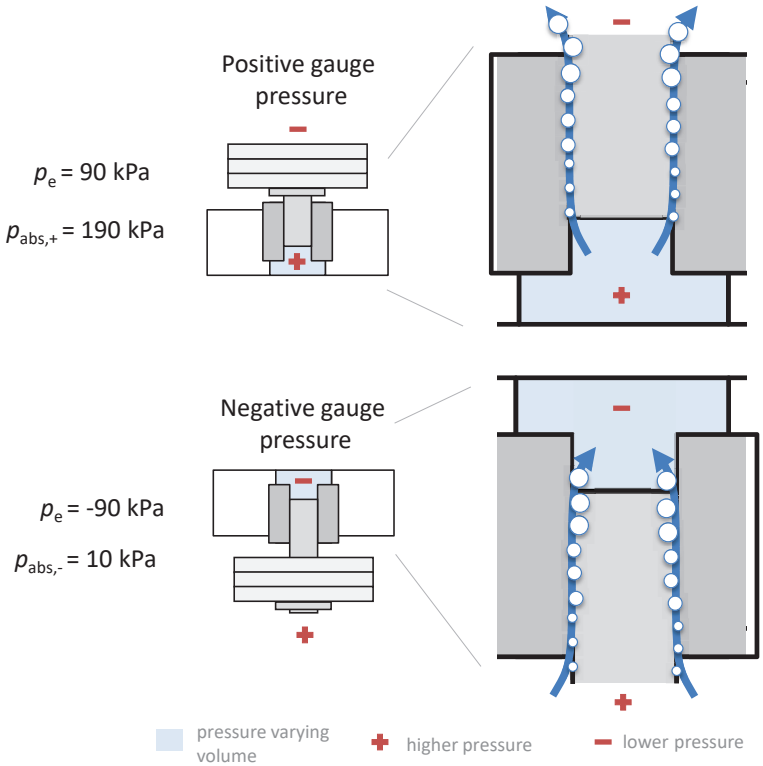
A loss in volume inside the pressure varying volume below the piston occurs.

The piston moves down.

The gasflow is driven by:
- PCU geometry (gap width)
- the pressure gradient (p_+ - p_-)

The fallrate depends on:
- the absolute pressure level inside the pressure varying volume

Why is the piston sinking faster?



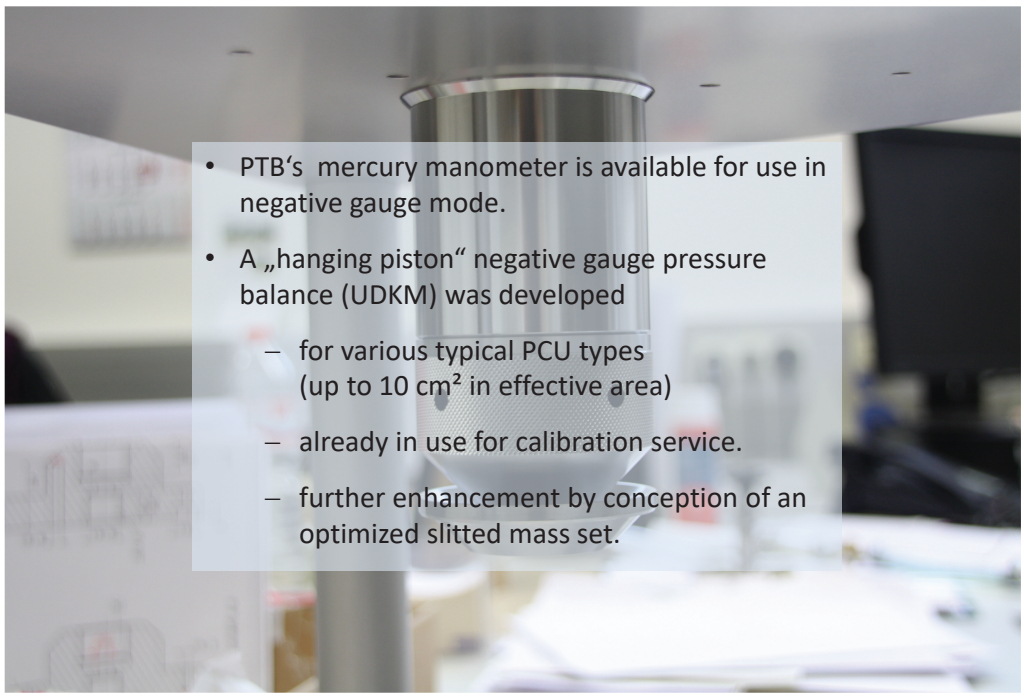
PTB UDKM (Negative Gauge Pressure Balance)

PCU mounting posts for
- DHI 10 cm²
- Ruska 3.3 cm²
- Maihak 5 cm²
- Budenberg 5 cm²

Summary

PTB

Summary



- PTB's mercury manometer is available for use in negative gauge mode.
- A „hanging piston" negative gauge pressure balance (UDKM) was developed
  - for various typical PCU types (up to 10 cm^2 in effective area)
  - already in use for calibration service.
  - further enhancement by conception of an optimized slitted mass set.

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Sven Ehlers, PTB Pressure Lab

27







Christoph Walther
Marc Wetzel

Thank you!

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Calibration of positive and negative gauge pressure – Two examples

Djilali Bentouati

Laboratoire national de métrologie et d'essais (LNE), France

Workpackage 3: Calibration of positive and negative gauge pressure

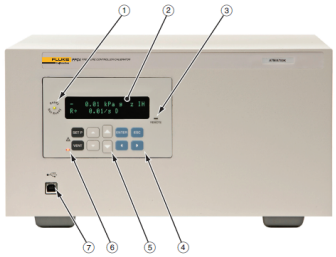
Two EXAMPLES

Djilali Bentouati

Pres2Vac - Final Workshop 16th – 17th May 2018, RISE – Borås, Sweden

Example n° 1

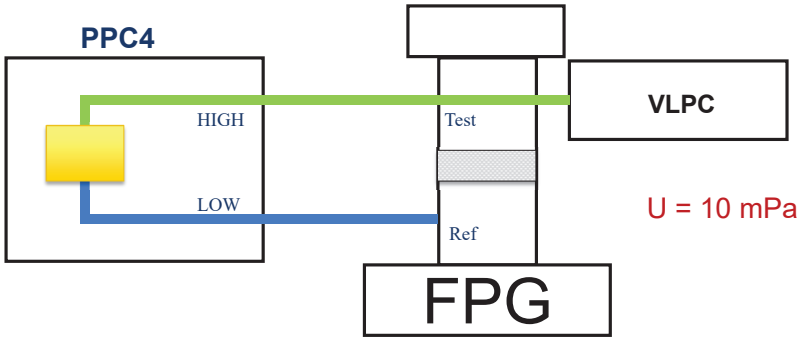
– **Digital manometer Fluke PPC4 BG15K**



- Range -15 to +15 kPa
- High accuracy device
- Internal gauges: differential transducer + barometer
- Reference pressure: 95 kPa to 110 kPa

- 3 methods of calibration have been studied:
  - FPG
  - 2 absolute pressure balances
  - Absolute balance +barometer

3 Calibration using an FPG



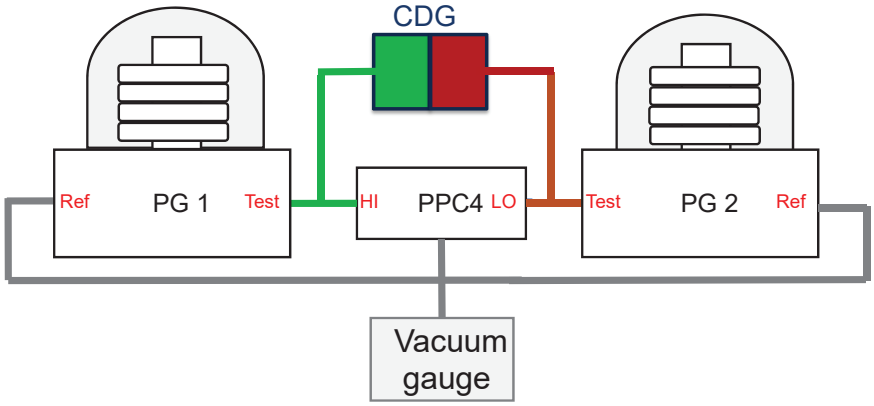
$U = 10\ mPa + 2.0 \cdot 10^{-5} \times |p|$

- FPG is the most accurate standard in this range.
- Calibration can be automated either for positive or for negative gauge pressure but not continiously from negative to positive.
- FPG is not built to work in negative pressure.
- Calibration in negative pressure is performed by swapping HIGH and LOW ports.

  Positive pressure is applied at LOW port.

  Calibration of positive and negative gauge pressures are performed separately.

4 Calibration using two absolute pressure balances



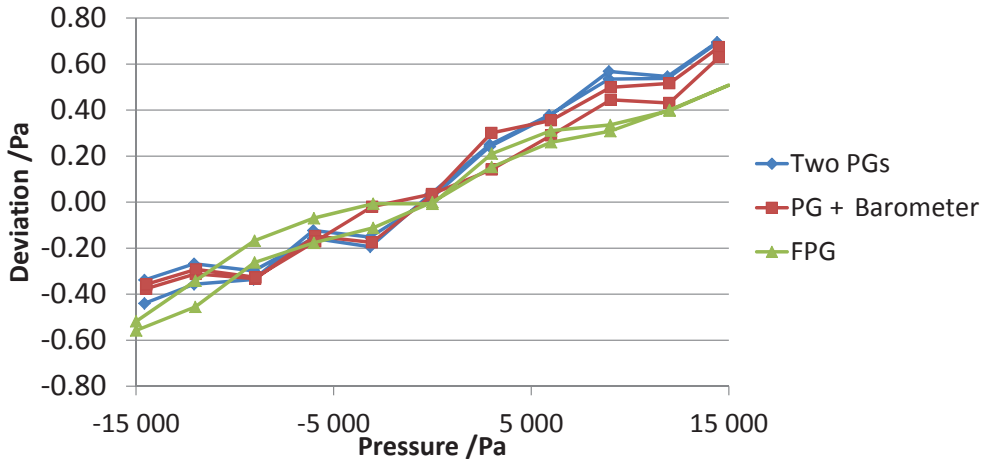
- Method based on the same principle as balance + barometer.
  - PG 2 compared to PG 1 using the CDG before and after calibration.
  - Instruments placed at the same level to eliminate correction due to head level.
  - Residual vacuum ports connected together to eliminate their contributions.
  - At each measurement point, value of PG 2 adjusted to equal that of PG 1.
  - Calibration can be automated over the whole range from negative to positive gauge pressures.

$U = 0.1\ Pa + 1 \cdot 10^{-5} \times |p|$ (0.2 Pa when a barometer is used = 3rd method)

5 Results

These three calibrations were carried out successively with a 60 s averaging time.

Using the FPG, positive and negative gauge pressures were calibrated separately.

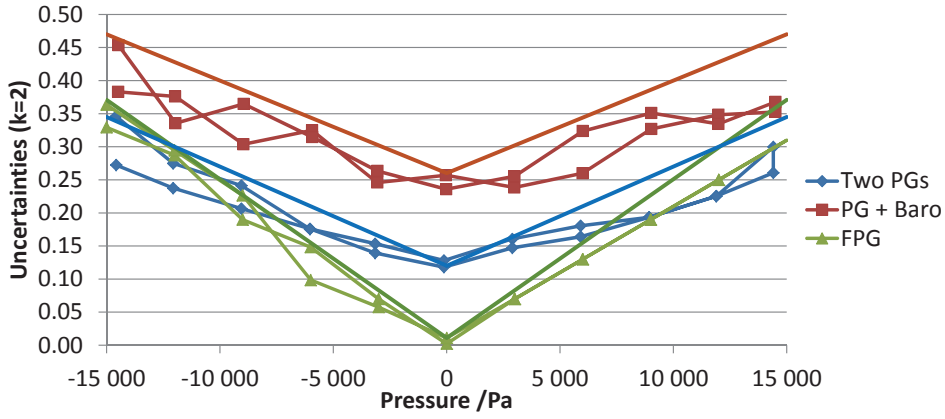


Differences between the three methods are lower than 0.2 Pa.

Deviations must be compared with their uncertainties.

6 Uncertainties

Main contributions on uncertainties budget come from the standard and repeatability.
Other contributions are negligible.

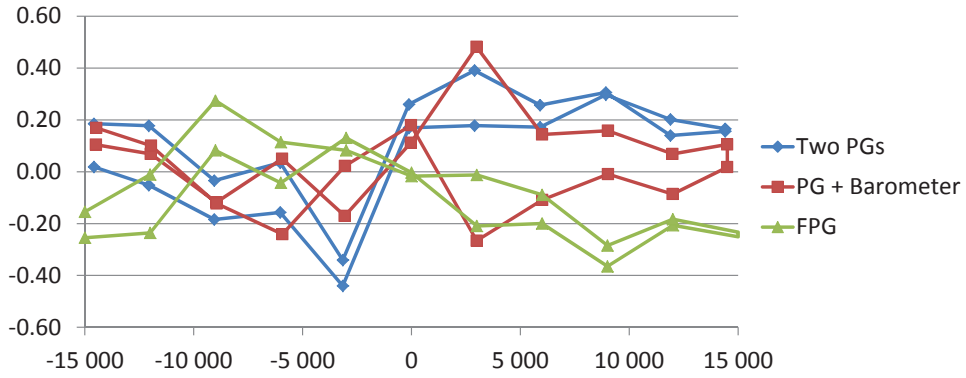


Modelled uncertainties

U = 0.26 Pa + 1.4E-05 × Ipl	PG + Barometer
U = 0.12 Pa + 1.5E-05 × Ipl	2 PGs
U = 0.011 Pa + 2.4E-05 × Ipl	FPG

7 Calibration of the PACE 1000

Reference values are calculated from the weighted mean.
Normalized deviations from this mean are calculated for each method.



Normalized deviations < 1 ====>>> **Results are consistent**

8 Example n° 2

– **Digital manometer GE measurement Pace 1000**



- Range -150 to +250 kPa
- Medium accuracy device
- 2 absolute pressure transducers
- Reference pressure: 0 to 1000 kPa
- Only a single calibration method was studied:

  – Absolute pressure balance + digital manometer RPM4 + Pressure controller
  – Calibration at different reference pressures

9 | Calibration of the PACE 1000

Instrument currently used by secondary laboratories and industry as a differential pressure standard.

Calibration with uncertainties < 100 ppm requires differential pressure balance or divider.

These standards are designed for high pressure measurements (>10 MPa)

Their uncertainties are not optimized for low line pressures <1 MPa.

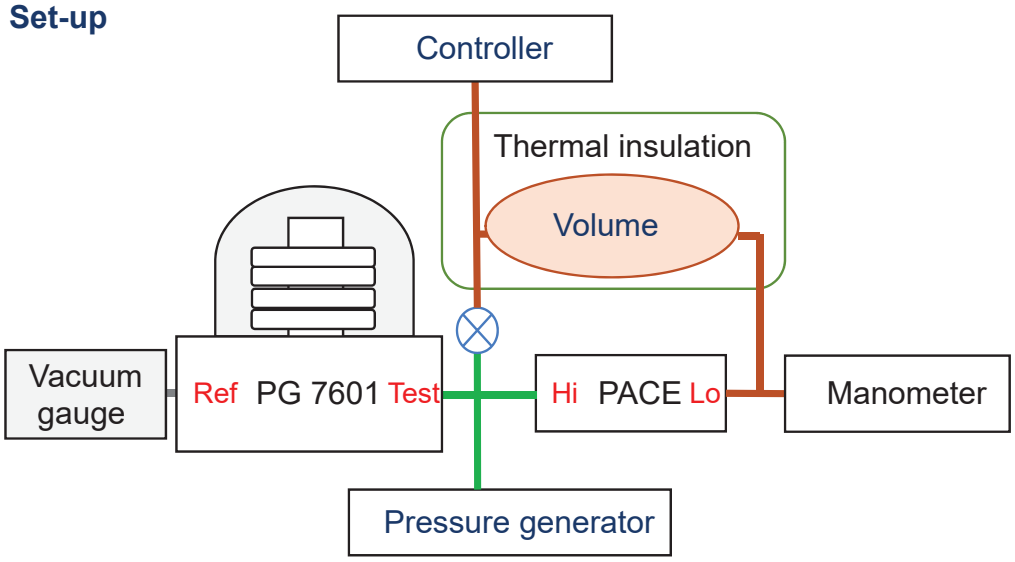
Alternative methods allow one to reduce measurement uncertainties at this low pressure using:

- Two pressure balances (recommended only for high accuracy device).
- Pressure balance and a manometer (this work).

Calibration conditions

- Measured pressure : **Differential**
- Measuring range : **from -110 to +400 kPa**
- Line pressure : **from 2 to 1150 kPa**
- Resolution : **0.0001 kPa**

10 | **Set-up**



- To compensate leaks in the volume, its pressure is regulated using a controller PPC4.
- The manometer and Lo port of PACE 1000 are placed on the opposite side of the volume to reduce the impact of pressure control.
- The protocol is the same as when barometer is used.
- Controller must be in measurement mode when accuracy error is determined,

11 Uncertainty

This method allows one to reduce several uncertainty components.

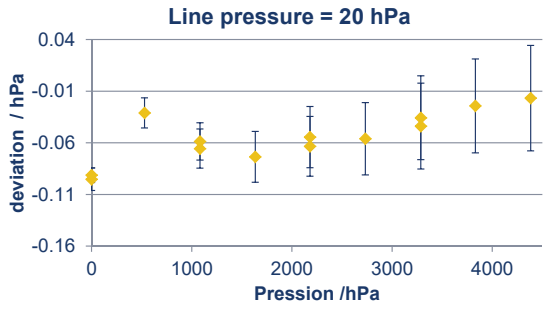
Using it, the uncertainties of the measured pressures are:

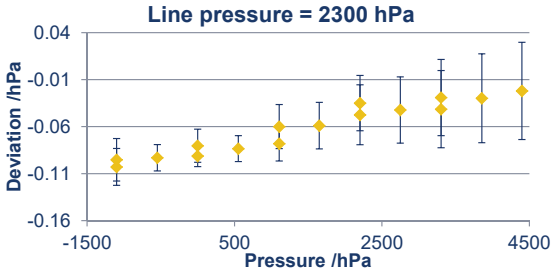
Pressure	Without zero correction	With zero correction
Low port	5.0 Pa + 2.8×10^{-5} × p_{line}	1.0 Pa + 1.0×10^{-5} × p_{line}
High port	0.5 Pa + 1.0×10^{-5} × (p_{line}+ Δp)	0.5 Pa + 1.0×10^{-5} × (p_{line}+ Δp)
Differential pressure	**5.7 Pa + 3.0×10^{-5} × |Δp| + 1.0×10^{-5} × p_{line}**	**0.7 Pa + 1.0 × 10^{-5} × |Δp|**

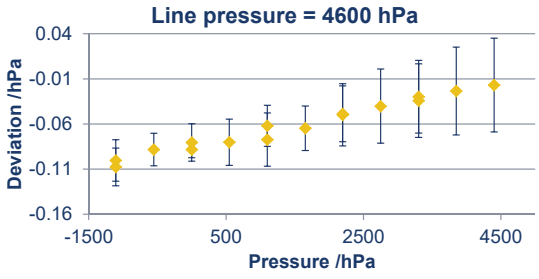
In this way, the uncertainty in **|Δp|** is independent of line pressure.

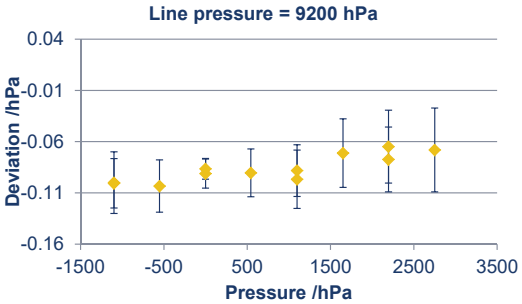
12 Results

Measurements were performed at several line pressures:
20, 2300, 4600, 9200 hPa









13 **Conclusion**

- **Low positive and negative gauge pressure measuring instruments could be calibrated by several methods.**
- **Primary and secondary laboratories could perform calibrations at low pressure using pressure balances.**
- **Independently of the line pressure and the range, measurements of positive and negative pressure could be carried out using gauge and absolute pressure devices.**
- **The choice of the method depends on the target uncertainties and availability of standards.**

14

Thanks for your attention

Calibration techniques for the intermediate pressure-to-vacuum range – Conclusion

Mark Plimmer

Conservatoire national des arts et metiers (CNAM), France

Workpackage 3: Calibration techniques for the intermediate pressure-to-vacuum range

CONCLUSION

Mark Plimmer

Djilali Bentouati

Pres2Vac - Final Workshop 16th – 17th May 2018, RISE – Borås, Sweden

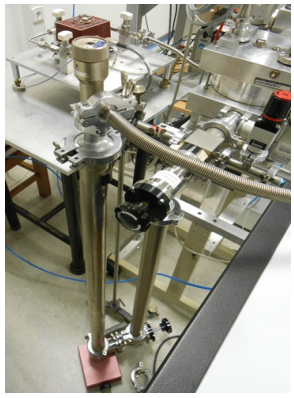
Task 3.1: Methods for accurate, **weather independent** calibration of low gauge pressure measuring instruments.

- Two ways to achieve weather independent calibration.
- Mission accomplished.



Hermetic chamber (CMI) :

- Metologically best but cumbersome.
- Expensive and long to implement.
- Best suited for FPGs.
- Really a method for primary labs.



Variable volume reservoir (PTB)

- Less expensive than hermetic chamber.
- Works below 10 kPa.



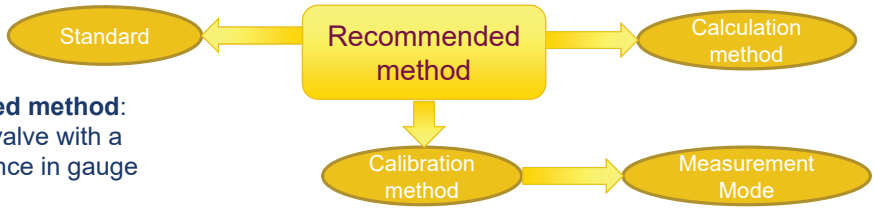
Fixed volume thermally isolated reservoir (LNE)

- Simplest, ideal for secondary labs and medium gauge pressure.
- Inadequate for low gauge pressure under 10 kPa.

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Task 3.3: Traceable methods for FPG calibration in absolute and gauge modes

- Validation of traceable methods

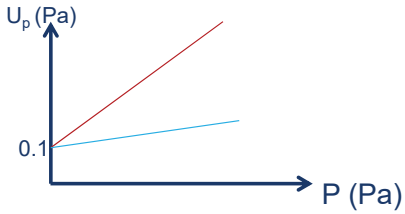


Recommended method: use of a leak valve with a pressure balance in gauge mode

- Target uncertainties
  - U_p = 10 mPa + 3×10^{-5} p (gauge)
  - U_p = 15 mPa + 3×10^{-5} p (absolute)
- Uncertainties actually achieved exceeded specifications
  - U_p = 10 mPa + 2.0×10^{-5} p (gauge)
  - U_p = 10 mPa + 2.0×10^{-5} p (absolute)

4

- Task 3.2: Methods for negative gauge pressure calibration based on absolute and gauge pressure balances, FPGs and liquid column manometers (for **primary laboratories**).
  - Target uncertainty (protocol) : U_p = 0.1 Pa + 3×10^{-5} lpl
  - Uncertainty actually achieved : U_p = 0.1 Pa + 1×10^{-5} lpl



Uncertainties for ***negative*** gauge pressure now as good as those for ***positive*** gauge pressure

→ Consequently the requirements of task 3.4 can be satisfied but only because the pressure coefficient in primary labs is **less than** 3×10^{-5} Pa^{-1}

- Task 3.4: Calibrations of positive and negative gauge pressure **devices** from -100 kPa to +10 kPa
  - Uncertainty heneceforth achievable in secondary labs : U_p < 1 Pa + 3×10^{-5} lpl

In industry U_p < 3 Pa + 2×10^{-4} lpl

→ **Goal achieved**

5

Outlook

- Where do we go from here?
- Baseline achieved for current primary standards
- Room for improvement in the dissemination to secondary labs and industry (Guides)

6

Thank you for your attention

Tack för din uppmärksamhet

Advances in pressure measurement using refractometry

Martin Zelan[1], Thomas Hausmaninger[1,2], Isak Silander[2], Ove Axner[2]

[1] RISE Research Institutes of Sweden (RISE), Sweden
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Advances In Pressure Measurement Using Refractometry

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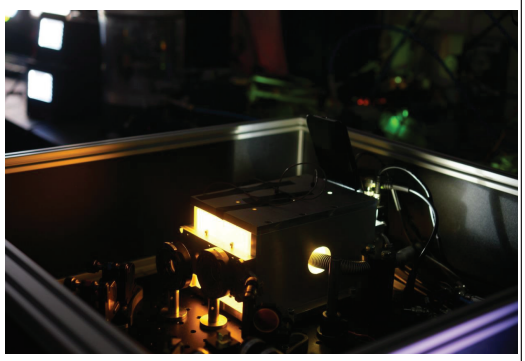
May 2018, Borås, Sweden

pres2vac
-A metrology research project within the European Union Horizon 2020 framework.

Outline of Presentation

Part I: Martin Zelan
- Basic of Refractometry
- Project Background
  - How it started and our first experiments
- EMPIR, pressure, and Dual-Cavity Refractometry

Part II: Isak Silander
- Refractometry by Gas Modulation (GAMOR)
  - Why GAMOR, and what is it?
  - The next generation
- Outlook, Challenges, and Future Plans

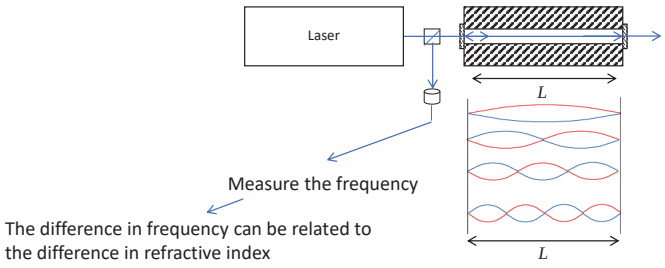


Basics of Refractometry

- Refractometry is the method of measure refractive index.

  That is; to measure how much the speed of the light and the wavelength is reduced with respect of the vacuum value. Or if you like; how much ”longer” the light experience its traveled path.

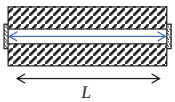
- Refractivity with a Fabry-Perot Cavity



Basics of Refractometry

- Refractometry is not at all new.
  - For example in Metrology it has been used in order to know the wavelength when measuring length.

- How to access the amount of gas or pressure with this? (Ideal gas)



$$PV = nRT \qquad \rho = \frac{n}{V} \qquad \rho = \frac{2}{3A}(n-1)$$

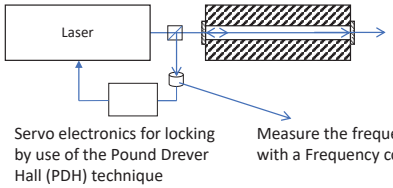
- Refractivity *(n - 1)* can be measured accurately with high precision by refractometry.

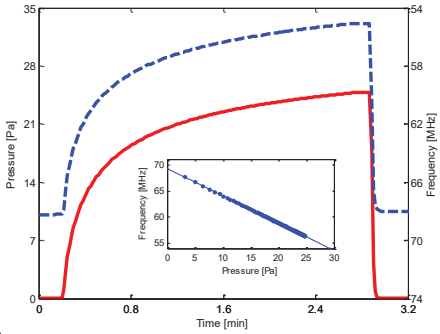
Project Background

- When I (Martin Z) started here at RISE (at that time SP). I started to look into our activities in this field (2012).
- Meet people from Umeå at a conference in Gothenburg. (Isak S and others)
- Based upon our previous joint experience, knowledge, and instrumentation, we concluded that it should be possible for us to realize a refractometry system without too much work!

Project Background: First experiment

- We decided to utilize the equipment that they had available in their lab at Umeå (Lasers, electronics, optics, Fabry-Perot cavity, frequency comb, etc.) to "quickly" setup an experiment.
- Successful experiment, but no focus on pressure

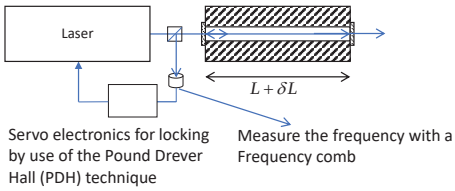




I. Silander et al., Optical measurement of the gas number density in a Fabry–Perot cavity, Meas. Sci. Technol. 24 105207 2013

Next step: EMPIR and Pressure

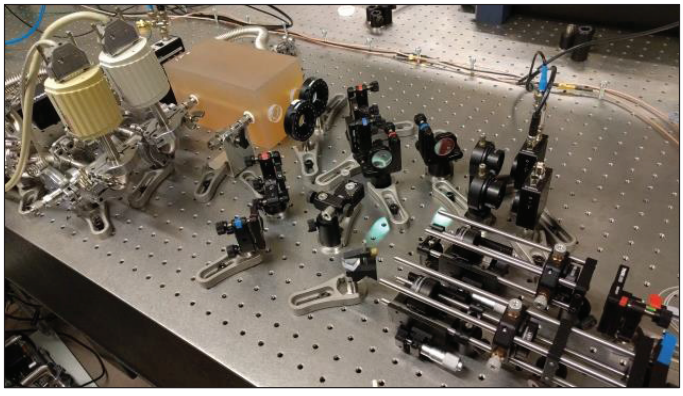
- Encouraged about the results we wanted to see how the method could be used to assess gas pressure.
  - Based on our knowledge and experience from this, we entered the EMPIR pres2vac.
    Alternative systems for pressure measurement using optical methods in the range 1 to 10^4 Pa



- What did we learn from our first experiment?
  - Frequency combs are expensive – not necessarily needed - alternative realization developed
  - Unstable cavity (drifts) is limiting factor

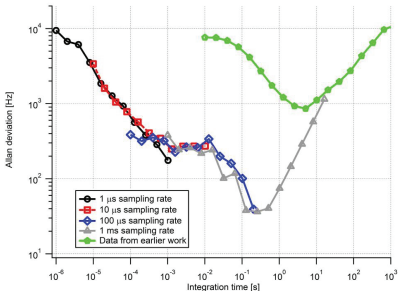
First EMPIR-system

- We built a completely new system based on two rugged, easily tunably and handable lasers and two cavities



First EMPIR-system: Results

- Our results were interesting: We manage to improve on the maximum resolution on short time scale, but drifts were still affecting us on longer times
- We summarized the results in a paper. However, it was not accepted in its present form by the journal.
  - Although we didn't agree with the referees we learned a lot from the comments and process
- This resulted in a lot of discussion and thinking, and we started to assess the true basics of the technique and review and assess all possible limitations.



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First EMPIR-system: Results

- Not only did we look into the basics of the methodology, we also tried to understand how to best utilize our systems:
  - We have a system that is limited by drifts, but seems to be able to perform great on short time scales.
- So what did we do?
  - We derived accurate expression of the relations between frequency, refractive index, density expressed in a form that is suitable when refractometry is used. arXiv:1704.01187v2
  - We started to conceptualize the best way of realizing such a system. arXiv:1704.01186v2
  - We discussed in some detail the concepts of accuracy, precision as well as the temperature dependence. arXiv:1704.01185v2
- We decided yet to improve on the system (realize a second version) with special emphasis on pressure in the 1 to 10^4 Pa range

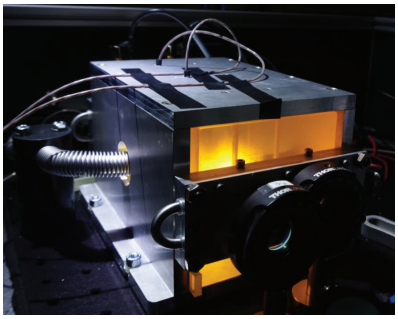
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Limitations of Fabry-Perot refractometry

For Nitrogen a change of pressure of 1 mPa corresponds to a relative change in length seen by the light of 3×10^{-12}

- For a cavity length 0.2 m this corresponds to 0.6 pm
- For Zerodur this requires a thermal stability of 30 µK

This requires carefully designed instrumentation



Limitations of Fabry-Perot refractometry

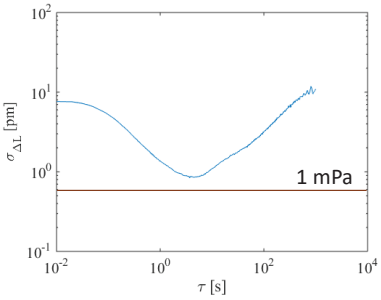
Option 1:

Build a highly stable system with extreme thermal control.

Option 2:

Measure on a time scale not affected by drifts

Implementation of gas modulation refractometry (GAMOR)



The Allan deviation of the difference between the cavity lengths in an evacuated dual Fabry–Perot cavity. (Early work 2015)

Gas Modulation Refractometry (GAMOR): The principle

- Modulate the pressure in the cavity

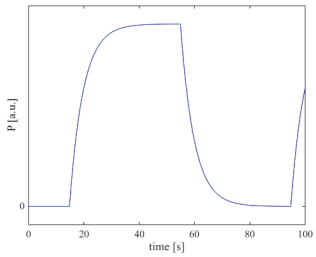


Illustration of a single measurement cycle in which the cavity, which originally is empty, first is filled with a certain pressure of gas before it is emptied again.

Gas Modulation Refractometry (GAMOR): The principle

- The measured beat frequency will be a combination of the change in density and drift in the difference in cavity length between the two cavities

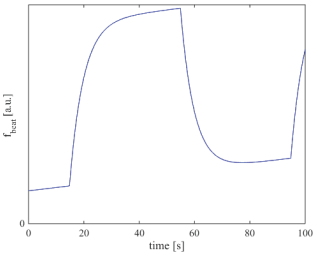


Illustration of a single measurement cycle in which the cavity, which originally is empty, first is filled with a certain pressure of gas before it is emptied again.

Gas Modulation Refractometry (GAMOR): The principle

- To differentiate the refractivity-response from the drift of the cavity lengths a linear interpolation is made between two evacuated points (before and after the filling)

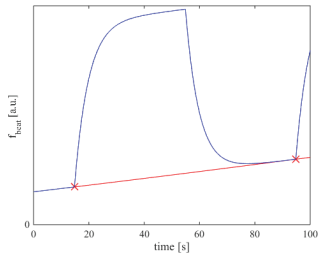


Illustration of a single measurement cycle in which the cavity, which originally is empty, first is filled with a certain pressure of gas before it is emptied again.

Gas Modulation Refractometry (GAMOR): The principle

- The linear interpolation is thereafter subtracted from the beat signal, giving rise to a “corrected” beat frequency that is proportional to the density

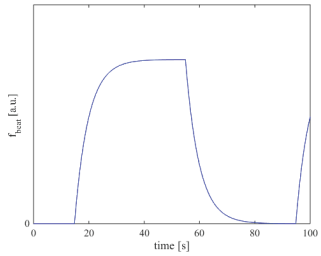
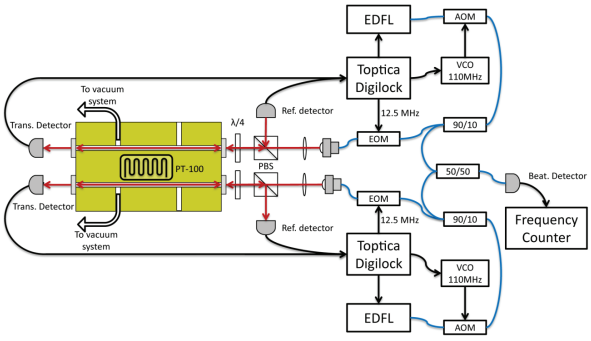


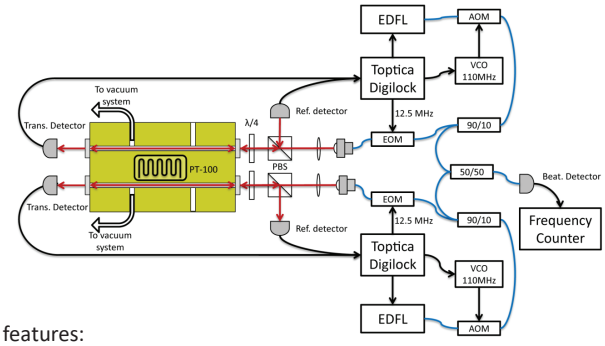
Illustration of a single measurement cycle in which the cavity, which originally is empty, first is filled with a certain pressure of gas before it is emptied again.

The Refractometer



Schematic illustration of the GAMOR system. Blue curves: optical fibers; red lines: free-space light propagation; black curves: wires; EDFL: Er-doped fiber laser; AOM: acousto-optic modulator; EOM: electro-optic modulator; PBS: polarizing beam splitter cube; λ/4: quarter-wave plate; Beat. Detector: beat note detecting detector; Ref. detector: reflected light detector; Trans. detector: transmission light detector.

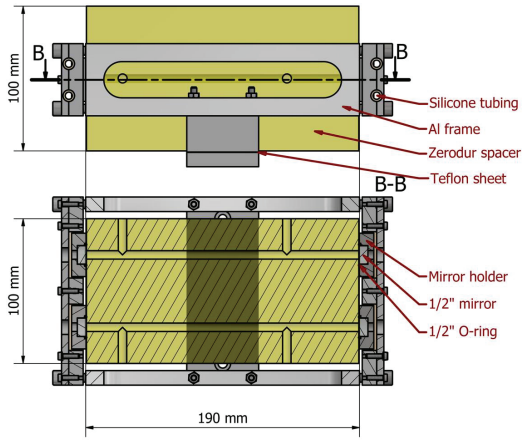
The Refractometer



Design features:

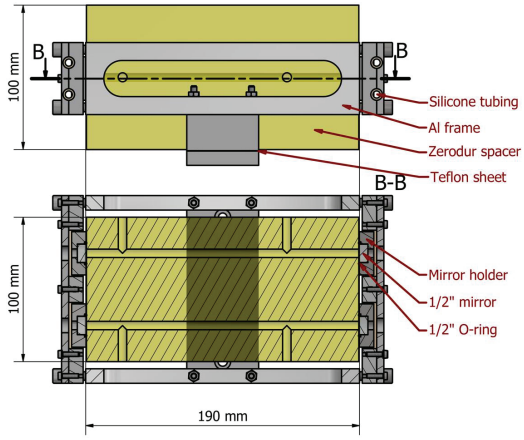
- All commercial components (at 1.5 µm)
- Automatic laser locking
- Automatic mode jumps
- Lasers locked to cavity modes with the Pound-Drever-Hall technique

The Cavity generation 1.0



CAD drawing of the DFPC assembly. Four 1/2” mirrors are mounted in mirror holders that are pressed onto the cavity spacer. The seal between the spacer and the mirror is achieved by the use of a standard 1/2” O-ring. To avoid stress passing from the frame to the cavity spacer the mirror holders are suspended on standard silicone lab tubing. The Zerodur pacer rests on a part of the aluminum frame, which is separated from the spacer by a 1 mm Teflon sheet.

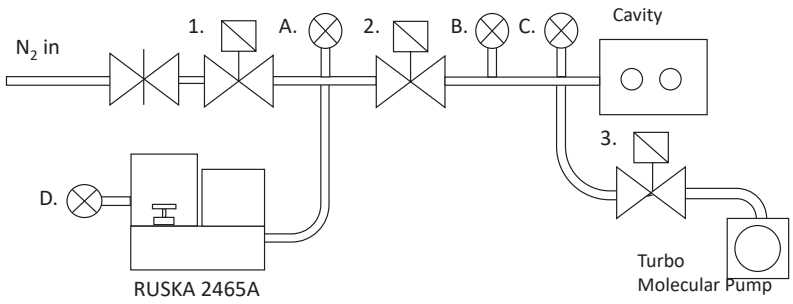
The Cavity generation 1.0



Design features:

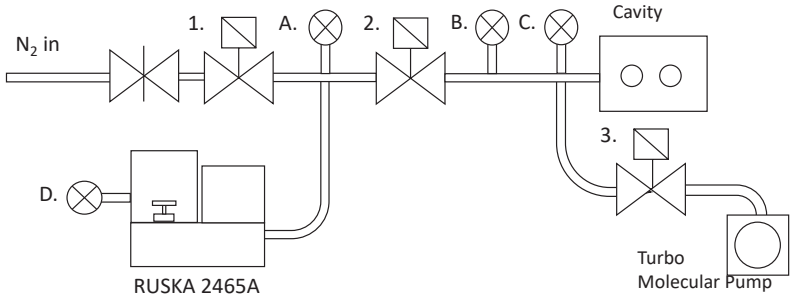
- Mirrors can be changed/cleaned within 30min
- Aluminum frame machined in-house from CAD drawings
- Zerodur spacer machined from CAD drawings by Microbas
- Standard ½” Mirrors with 0.5m Radius from Layertech
- No Clean room needed !!

Gas system



Schematic illustration of vacuum system. Solenoid valves: 1: Parker 009-0172-900; 2 and 3: Leycon 215006v01; Pressure gauges: A: Oerlikon-Leybold CTR 100 N 1000 Torr; B: Oerlikon-Leybold CTR 101 N 1000 Torr; C: Oerlikon-Leybold CTR 101N 0.1 Torr; D: Oerlikon-Leybold CTR 100 N 1 Torr. All pressure gauges are capacitive;Dead weight piston gauge (RUSKA 2465A).

Gas system

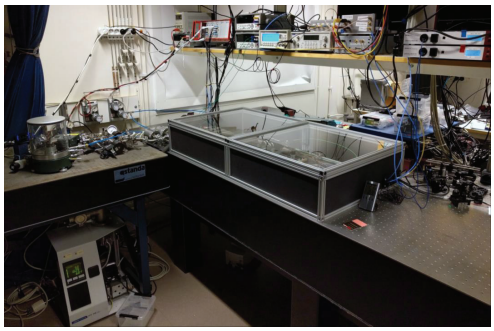


Design features:

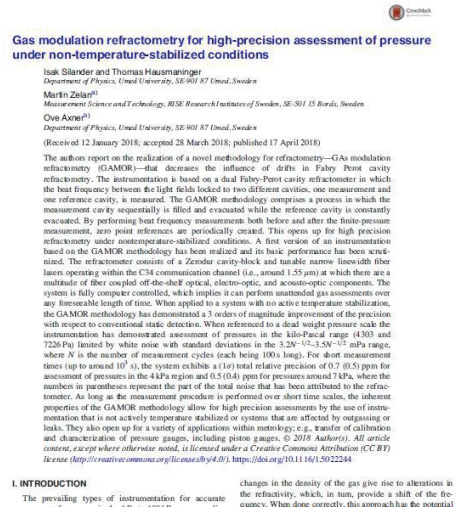
- Automatic valve control
- Automatic fill to RUSKA set pressure
- Zero point and cavity mode number measurement with gauge C and B

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https://doi.org/10.1116/1.5022244

GAMOR Realization



Picture of the GAMOR experiment in Umeå. The refractometer is placed in a box to reduce environmental influences. The dead weight piston gauge on top of the left optical table is used to generate reproducible pressures.



Gas modulation refractometry for high-precision assessment of pressure under non-temperature-stabilized conditions

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The authors report on the realization of a novel methodology for refractometry—GAs modulation refractometry (GAMOR)—that decreases the influence of drifts in Fabry Perot cavity refractometry. The instrumentation is based on a dual Fabry-Perot cavity refractometer in which the beat frequency between the light fields locked to two different cavities, one measurement and one reference cavity, is measured. The GAMOR methodology comprises a process in which the measurement cavity sequentially is filled and evacuated while the reference cavity is constantly evacuated. By performing beat frequency measurements both before and after the finite-pressure measurement, zero point references are periodically created. This opens up for high precision refractometry under nontemperature-stabilized conditions. A first version of an instrumentation based on the GAMOR methodology has been realized and its basic performance has been scrutinized. The refractometer consists of a Zerodur cavity-block and tunable narrow linewidth fiber lasers operating within the C34 communication channel (i.e., around 1.55 μm) at which there are a multitude of fiber coupled off-the-shelf optical, electro-optic, and acousto-optic components. The system is fully computer controlled, which implies it can perform unattended gas assessments over any foreseeable length of time. When applied to a system with no active temperature stabilization, the GAMOR methodology has demonstrated a 3 orders of magnitude improvement of the precision with respect to conventional static detection. When referenced to a dead weight pressure scale the instrumentation has demonstrated assessment of pressures in the kilo-Pascal 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 exhibits 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 has been attributed to the refractometer. As long as the measurement procedure is performed over short time scales, the inherent properties of the GAMOR methodology allow for high precision assessments by the use of instrumentation that is not actively temperature stabilized or systems that are affected by outgassing or leaks. They also open up for a variety of applications within metrology; e.g., transfer of calibration and characterization of pressure gauges, including piston gauges. © *2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).* https://doi.org/10.1116/1.5022244

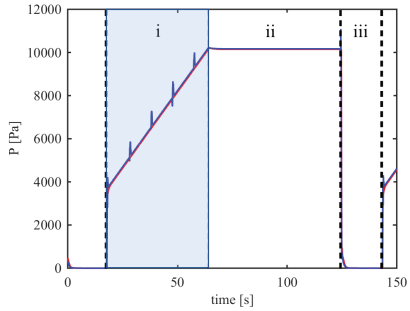
I. INTRODUCTION

The prevailing types of instrumentation for accurate

changes in the density of the gas give rise to alterations in the refractivity, which, in turn, provide a shift of the frequency. When done correctly, this approach has the potential

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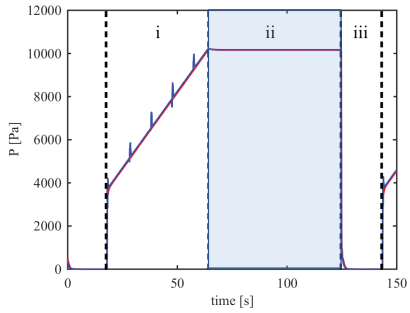
State I – filling of system (valve 1. and 2. open)



Blue curve: Pressure assessed by the refractometer from a single measurement cycle when the refractometer was connected to a piston gauge for assessments of pressure. Red curve (largely hidden) corresponds to the pressure assessed by the pressure gauge B.

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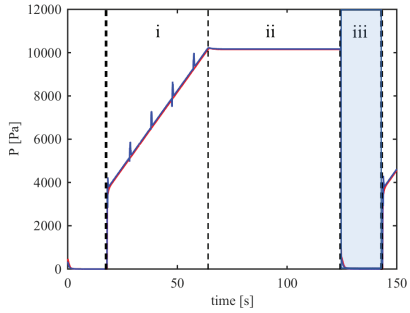
State II – equilibration (valve 2. open)



Blue curve: Pressure assessed by the refractometer from a single measurement cycle when the refractometer was connected to a piston gauge for assessments of pressure. Red curve (largely hidden) corresponds to the pressure assessed by the pressure gauge B.

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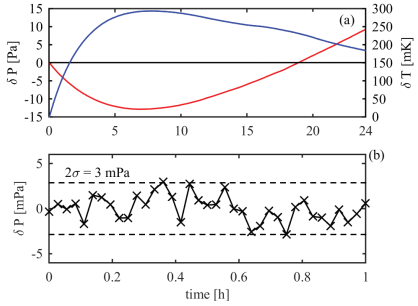
State III – pumping down (valve 3. open)



Blue curve: Pressure assessed by the refractometer from a single measurement cycle when the refractometer was connected to a piston gauge for assessments of pressure. Red curve (largely hidden) corresponds to the pressure assessed by the pressure gauge B.

J. Vac. Sci. Technol., A, **36**, 03E105 (2018).
https://doi.org/10.1116/1.5022244

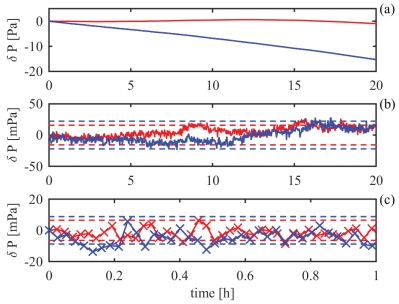
Zero pressure measurements GAMOR vs static



A 24 h long series of measurement of an empty measurement cavity evaluated by two different means: the red curve – without gas modulation, referred to as a static mode of detection, and the black curve – by use of the GAMOR methodology (both left axis). The blue curve (right axis) represents the temperature. Panel (b): a zoom in of the first hour section of the data taken with the GAMOR methodology.

J. Vac. Sci. Technol., A, **36**, 03E105 (2018).
https://doi.org/10.1116/1.5022244

Pressure Measurements GAMOR vs static



Static

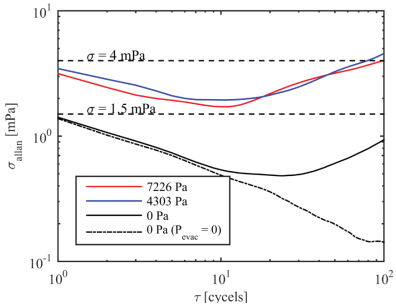
GAMOR

GAMOR

The difference in the pressure assessed by the refractometer and that provided by the piston gauge for pressures of nitrogen of 4303 and 7226 Pa (blue and red curves, respectively). Panel (a): Pressure evaluated by the static mode of detection. Panel (b): Pressure evaluated by the GAMOR methodology. Panel (c): The first hour data . The dashed lines represent the levels for ± twice the standard deviation of the data [16 and 22 mPa in panel (b), and 6.2 and 8.8 mPa in panel (c), for 4303 and 7226 Pa, respectively].

J. Vac. Sci. Technol., A, **36**, 03E105 (2018).
https://doi.org/10.1116/1.5022244

Allan Deviation- GAMOR system stability

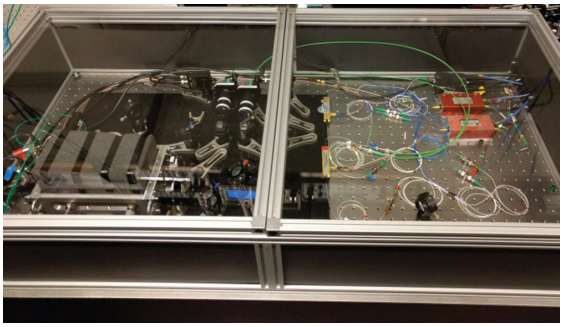


The Allan deviation of the measurements presented above. The solid blue and red curves represent the Allan deviations of the refractometer referred to the piston gauge for pressures of 4 303 and 7 226 Pa, respectively. The black curves represent the deviations of measurements of an evacuated cavity not referenced to the piston gauge. The solid and dash-dotted black curves represent data taken with and without the use of pressure gauge C, respectively.

J. Vac. Sci. Technol., A, **36**, 03E105 (2018).
https://doi.org/10.1116/1.5022244

GAMOR System - performance

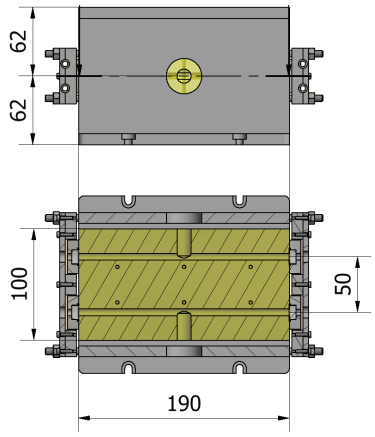
- GAMOR methodology has demonstrated **precision** in the order of 4 mPa for measurement times up to 3 h (for 4 303 and 7 226 Pa), sub-ppm.
- Pressures up to 10 kPa has been demonstrated
- The system is mainly constructed of standard of the shelf components.
- Sturdy system which can be operated by students.



“The Next Generation” Cavity Design

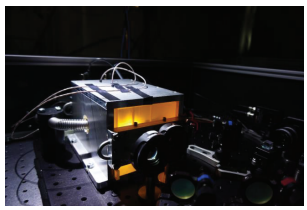
New design features:

- Holes for several Pt-100 probes drilled in the spacer
- Active temperature stabilization (Peltier elements)
- Larger vacuum connections (½”)



GAMOR “The Next Generation”

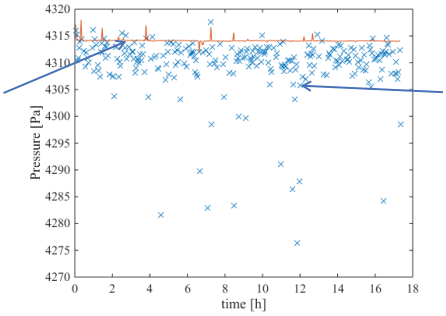
- Reduced thermal drifts and gradients
- Improved temperature sensing
- Improved gas exchange
- Upgraded data acquisition
- Two “identical” systems (Umeå and Borås)
- Transportable





Comparison of Systems – First measurements

Spacer resting on:
Teflon



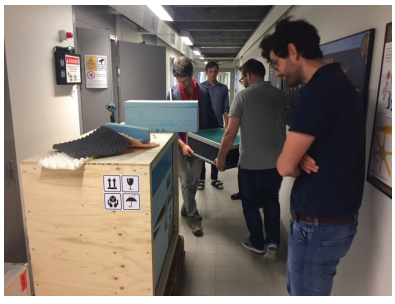
Spacer resting on:
Thermal paste

The difference in the pressure assessed by the two GAMOR systems. Both systems connected to the same piston gauge set to 4303 Pa.

Installing System at RISE

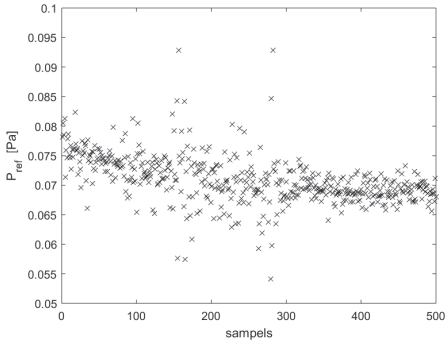
- Starting unpacking the system @10:00
- First measurements running @ 14:00





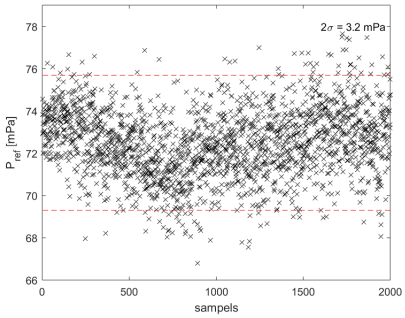
Improving the System at RISE

Thermal paste ->Teflon sheet



First test of cavity assembly with Teflon sheet at RISE. A 14 h long series of measurement of an empty measurement cavity evaluated by use of the GAMOR methodology.

Zero Pressure Measurement



A 60 h long series of measurement of an empty measurement cavity evaluated by use of the GAMOR methodology. The read dashed lines represents two standard deviations i.e. 3.2 mPa.

Challenges

- The precision of the instrument is relatively high, but limited by:
  - The cavity spacer (noise)
  - Temperature measurements (gradients)
- The accuracy needs to be improved/investigated
  - Comparison against primary standards
  - Alternatively absolute determination of all factors

Future Plans

- Increase the working range (beyond atm)
- Focus on means to improve accuracy
- Using the system to measure gas constants
- Work towards a transportable system
- Dual-system comparisons to evaluate performance

Appendix

Summary of Deliverable 6 within pres2vac

Appendix D6 Recommendation of use: Summary

- Encourage further work with current instruments with the aim to explore the technical limits of the working range. In particular, by increasing the pressure beyond the atmospheric level in order to fully investigate the practical possibilities of the optical techniques.
- In more detail investigate the potential of optical methods for transfer of calibration with the purpose to be able to improve on the accuracy of pressure assessments, in particular in the low pressure to vacuum range.
- Develop and offer a service to stakeholders that provide in-depth analysis of the dynamic response of various pressure gauges.
- Utilize the high resolution and precision of optical methods to investigate conventional techniques to provide the community with a more in-depth understanding of various techniques.
- Further investigate the potential of FP refractometer techniques for highly accurately differential pressure measurements. In particular, it would be highly interesting to benchmark the technique towards conventional state-of-the-art instruments for a variety of pressure ranges.

Appendix D6 Recommendation future work: Summary

- To improve on the accuracy of the method when used as a primary standard, improve ab-initio calculations of various relevant gas properties at different wavelengths. Specific gases of relevance are He, Ne, Ar, and N2, where wavelengths of particular interest are 532 nm, 632 nm, and 1.5 µm.
- Look into means to improve on the stability by investigating various design options and different materials in order to minimize the effect caused by the material properties.
- Work on means to minimize the influence of the temperature, take advantages of state of the art temperature assessment, and develop general methods to reduce temperature as a limiting factor.
- To improve on the accuracy when used for transfer of calibration, a methodology should be developed that allows the system to be characterized using a standard at a set of pressures.
- Actively pursue variations of the techniques and methods by investigating means to optimize the assessments and minimize the influence of the currently limiting factors, such as temperature variations/control and material limiting properties such as aging, absorption and compression. Of particular interest are methods based on modulation, which within the 14IND06 project have shown great promise.

Appendix D6 Recommendation future work: Summary cont.

- Encourage the whole scientifically community to explore several new paths and methods to allow for new measurement innovations. This is especially interesting due to the forthcoming new SI-system that will allow for a pressure definition based on the Boltzmann constant.
- Encourage NMIs and the academic community 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.
- Encourage the development of easily operated transportable instrumentation constructed around easily accessible and robust components to allow for stakeholder uptake and acceptance.
- Actively encourage the scientific community to engage in communications and discussion with stakeholders to spread the recent scientific results as well as understand the future need of the society and industry.

Evaluation of a low absolute pressure transfer standard in the range 1 Pa to 10 kPa

Frédéric Boineau

Laboratoire national de métrologie et d'essais (LNE), France

EURAMET

14IND06 pres2vac

pres2vac

pres2vac - Final Workshop

Evaluation of a low absolute pressure transfer standard in the range 1 Pa to 10 kPa

Frédéric BOINEAU

Boras, 2018 May 17th

LNE le cnam

EURAMET's Research Programmes

EURAMET

SI Unit



Environment



Energy



Fundamental



Health



Industry



- EURAMET's research programmes (EMRP and EMPIR) support the collaboration of European metrology institutes, industrial organisations and academia through Joint Research Projects (JRPs). They are structured around European Grand Challenges in such areas as Health, Energy, the Environment and also aim to progress fundamental science.
- See https://www.euramet.org/ for more details.

EMRP
European Metrology Research Programme
Programme of EURAMET

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

EMPIR EURAMET

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Frame and objective of this study

Frame: project JRP 14IND06 prestovac

14IND06 pres2vac

"Industrial standards in the intermediate pressure-to-vacuum range"

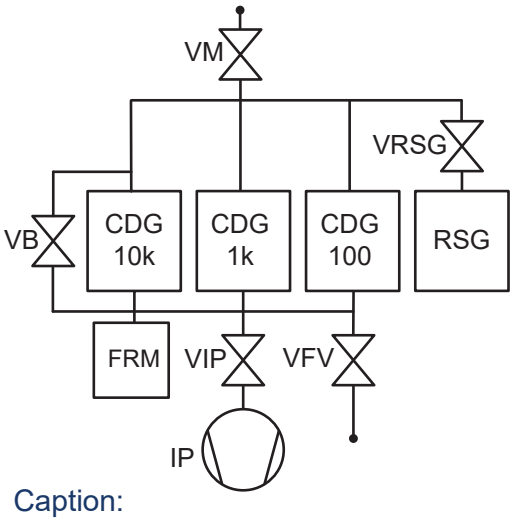
Pressure range: absolute pressure from **1 Pa to 10 kPa**

Transfer standard (TS) composed of a resonant silicon gauge (**RSG**) and 3 capacitance diaphragm gauges (**CDG**)

Objective: contribution in uncertainty lower than $1 \times 10^{-4} \times p$ ($k = 1$)

Intercomparison of calibration services or metrology institutes

Diagram of the transfer standard

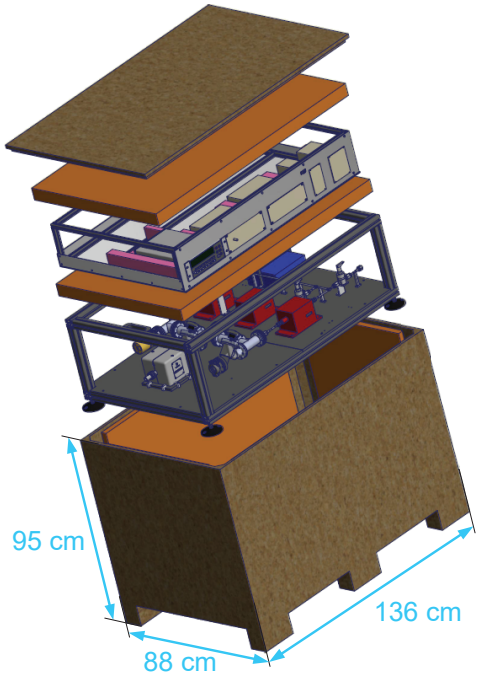


Differential CDGs were used for the needs of the study. They were also found, from our experience, to be more stable than absolute CDGs.
However, the use of absolute CDGs will greatly improve the compacity of the bench

Caption:

- CDG: capacitance diaphragm gauge (full scale 100 Pa, 1 kPa and 10 kPa)
- RSG: resonant silicon gauge (130 kPa full scale)
- IP: ion pump

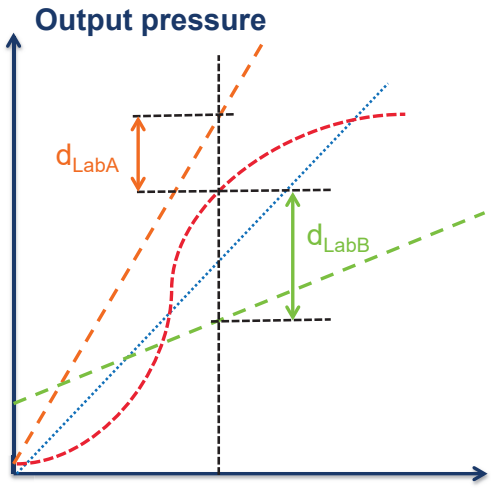
Transfer standard at a glance



Overview

- Procedure to use the transfer standard (in order to achieve the maximum benefits)
- Metrological characteristics in an actual situation (traveling)
- Budget of uncertainties
- Conclusion

General issues of a transfer standard



- Transfer standard
- Standard pressure Laboratory A
- Standard pressure Laboratory B

Provides the same output pressure over the comparison duration (does not need to be exact)

→ high resolution
→ short-term and mean-term reproducibility
→ output pressure keeps the same “shape”

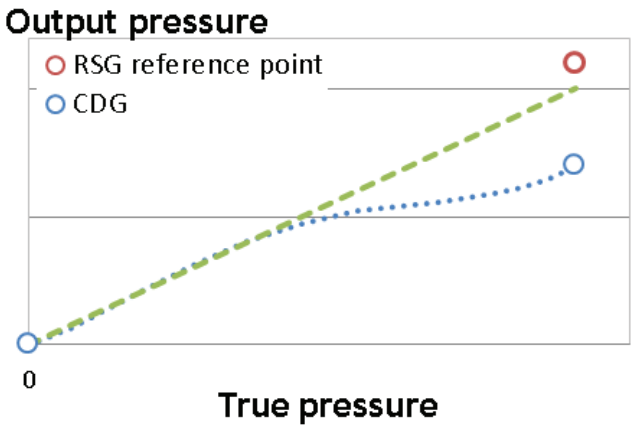
CDG features

How can CDGs be suitable as transfer standards in low absolute pressure

Resolution	$1\times10^{-5}\times p$	
Repeatability	$2\text{-}3\times10^{-5}\times p$	
Mean-term reproducibility	$5\times10^{-4}\times p$ -$1\times10^{-3}\times p$	

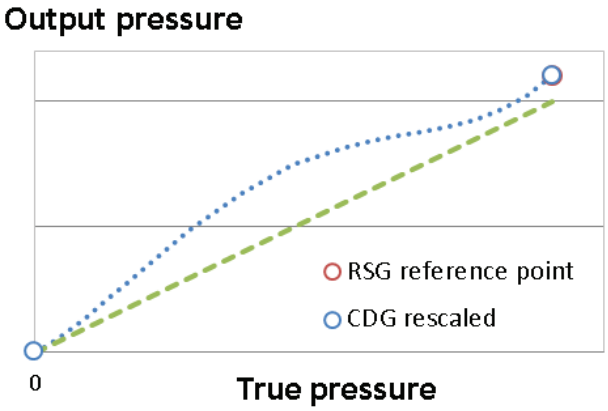
How to overcome a CDG drift ?

By rescaling it with **a RSG**



Multiply p_{CDG} by the rescaling factor $k_{CDG} = p_{RSG}/p_{CDG}$

RSG requirements



The RSG has to be stable over time

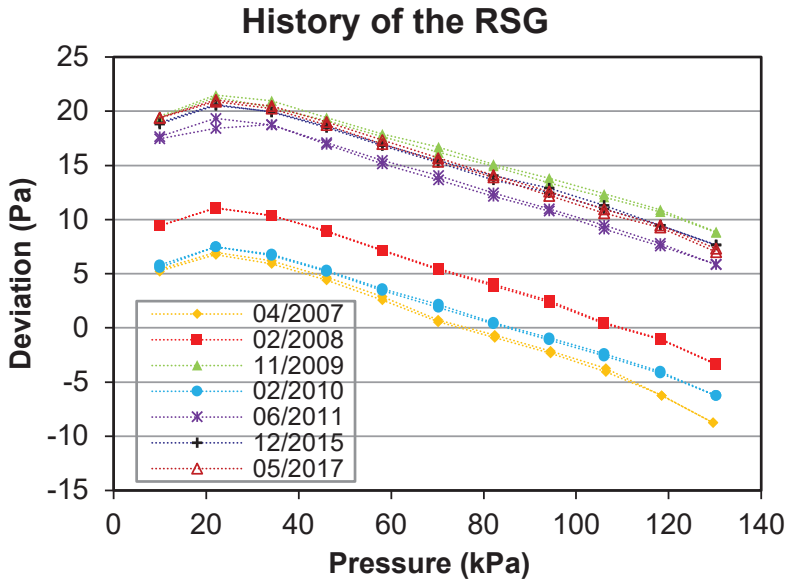
→ Key comparison CCM.P-K4 (2012) with a **RSG 10 kPa** and a **CDG 100 Pa**

Alternative solution

- Because the RSG 10 kPa of the comparison CCM.P-K4 was not available any more
- One of our RSG 130 kPa has been used
  - Not stable considering a single pressure point
  - But with a stable correction slope

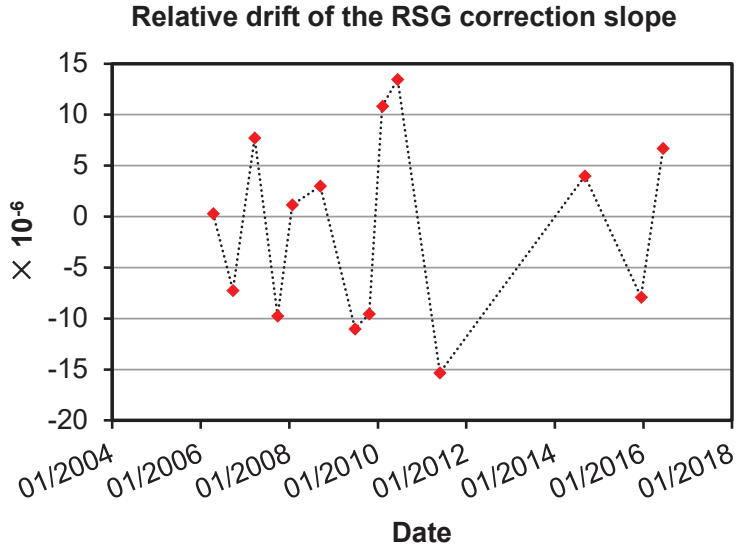
RSG 130 kPa calibrations history

The RSG seems to drift only in offset



RSG slope stability

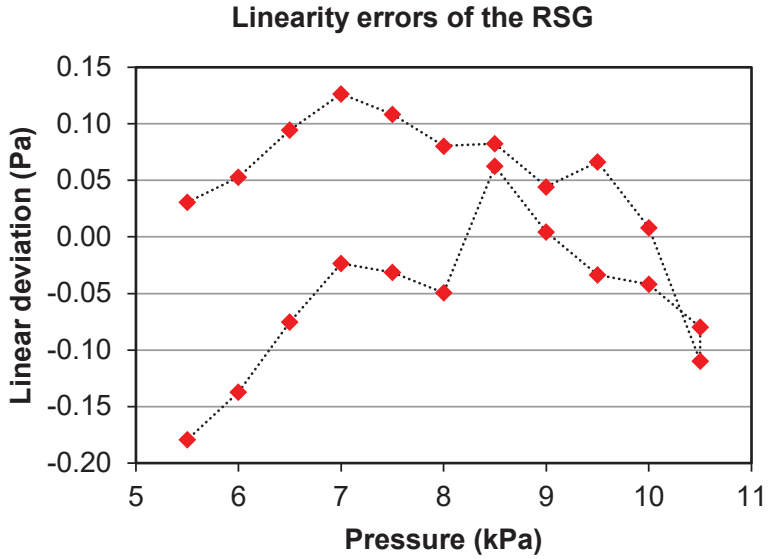
The drift in correction slope of the RSG is nicely stable



The drift is estimated to be (-1.0 ± 6.6) ppm per year

RSG linearity error

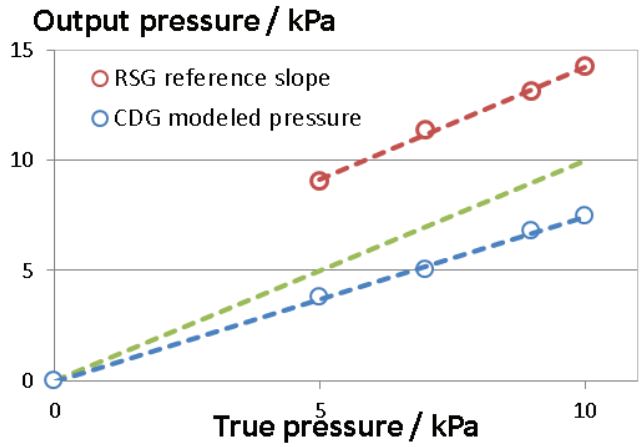
From the calibration in the range [5-10] kPa, using a Force balanced piston gauge (Fluke FPG 8601)



Rescaling method with the RSG 130 kPa

Step 1 : Comparison of instruments slopes

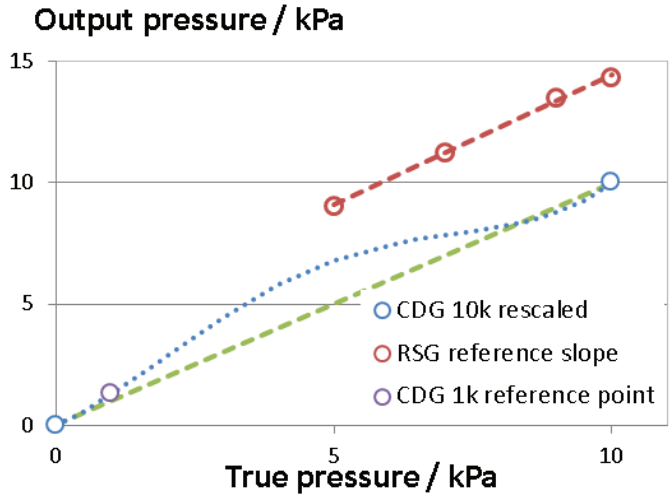
- Take RSG and CDG10k signal in 4 four points



- Multiply p_{CDG} by the rescaling factor k_{CDG} = RSG slope/CDG slope

Rescaling method with the RSG 130 kPa

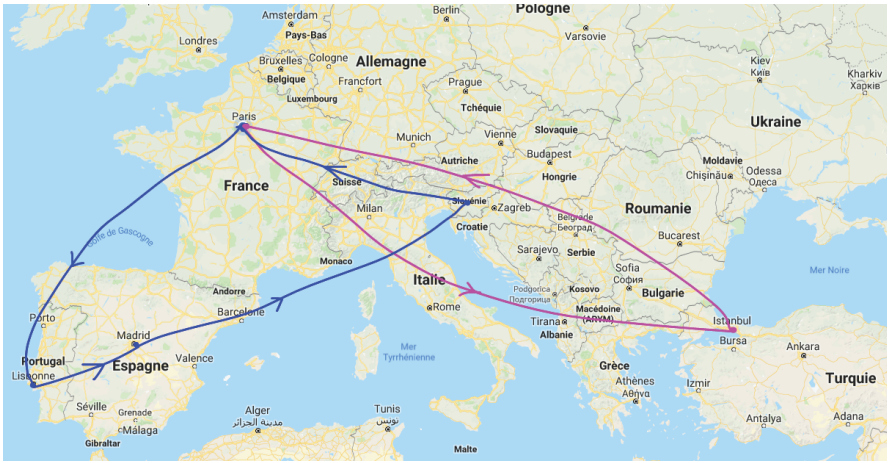
Step 2 : Slope of CDG10k is rescaled to that of the RSG



Protocol

- Calibration by increasing pressure levels
- Four cycles
- Each cycle includes rescaling points:
  - 1 common pressure point at 100 Pa for CDG 100 and CDG 1k
  - 1 common pressure point at 1kPa for CDG 1k and CDG 10k
  - 4 common pressure points for CDG 10k and RSG (5, 7, 9 and 10 kPa)

Travel of the transfer standard



First Loop:
LCM-LNE, Paris
FCT-UNL, Lisbon
CEM, Madrid
IMT, Ljubljana
LCM-LNE, Paris

Second Loop:
LCM-LNE, Paris
UME, Istanbul
LCM-LNE, Paris

Uncertainty assessment (1st loop)

RSG

Stability, temperature influence

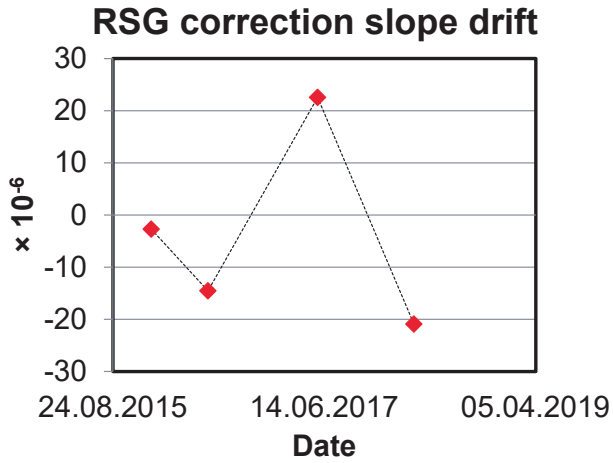
CDG

« Shape » stability, thermal transpiration

Rescaling

Scattering of rescaling coefficients during a calibration

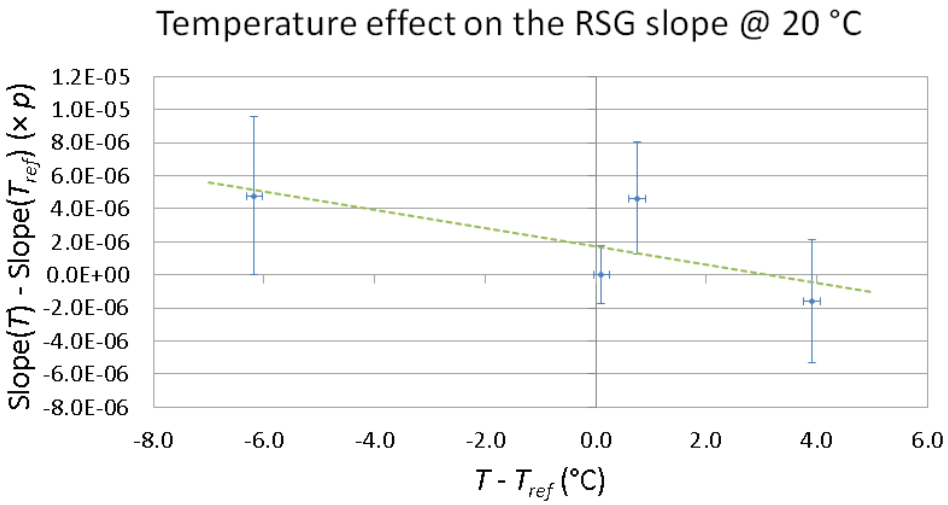
RSG Stability



Uncertainty contribution	
× *p*	Pa
1.0×10^{-5}	0

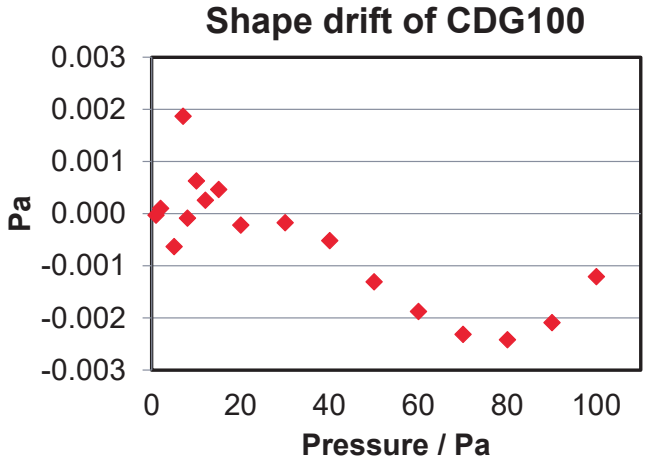
Temperature coefficient

All the participants do not operate at the same ambient temperature



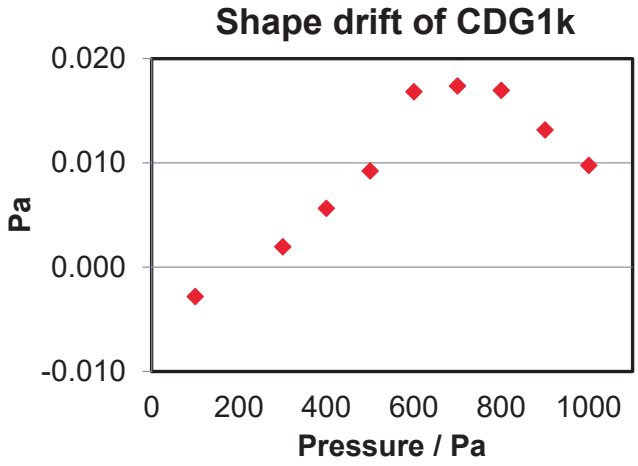
The temperature effect is less than 2×10^{-6} ($\times p$) for a 3 K variation (negligible)

Shape drift / CDG100



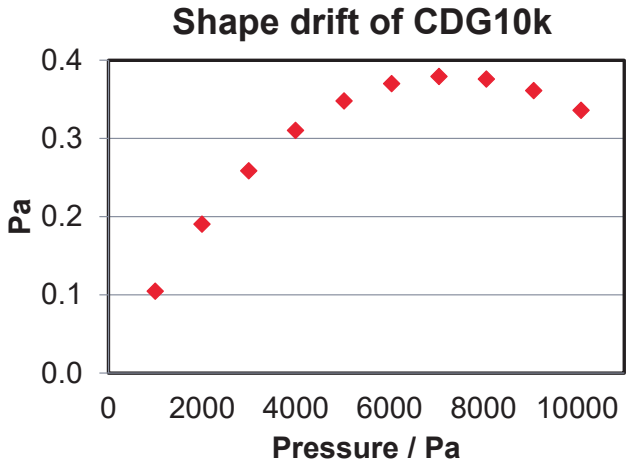
Uncertainty contribution	
× p	Pa
7.6×10^{-6}	0.0018

Shape drift / CDG1k



Uncertainty contribution	
× p	Pa
2.8×10^{-5}	0

Shape drift / CDG10k



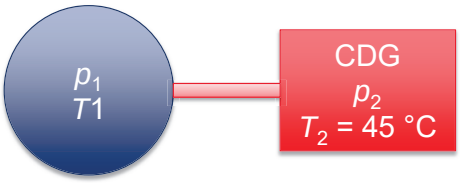
Uncertainty contribution	
× p	Pa
2.2×10^{-5}	0.24

Rescaling coefficients

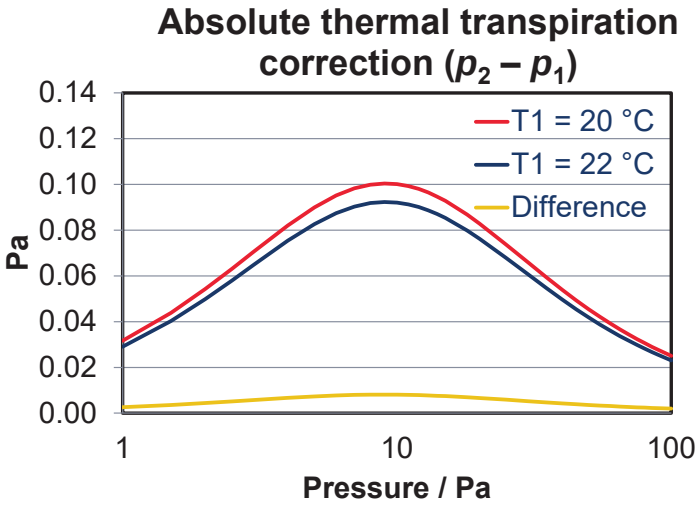
Attempt to estimate an uncertainty on the rescaling coefficients.
It can however be considered that such contribution is already included in the repeatability of the measurements

	Experimental standard deviations (ESD) of the rescaling coefficient (4 cycles)		
Laboratory	CDG 10k	CDG 1k	CDG100
A	$1.1 \times 10^{-4} \times p$	$1.1 \times 10^{-4} \times p$	$6.5 \times 10^{-4} \times p$
B	$2.6 \times 10^{-5} \times p$	$5.7 \times 10^{-5} \times p$	$1.1 \times 10^{-4} \times p$
C	$3.3 \times 10^{-5} \times p$	$8.5 \times 10^{-5} \times p$	$2.0 \times 10^{-4} \times p$

Thermal transpiration



Results are corrected for the same ambient temperature $T1$, by means of an empirical function



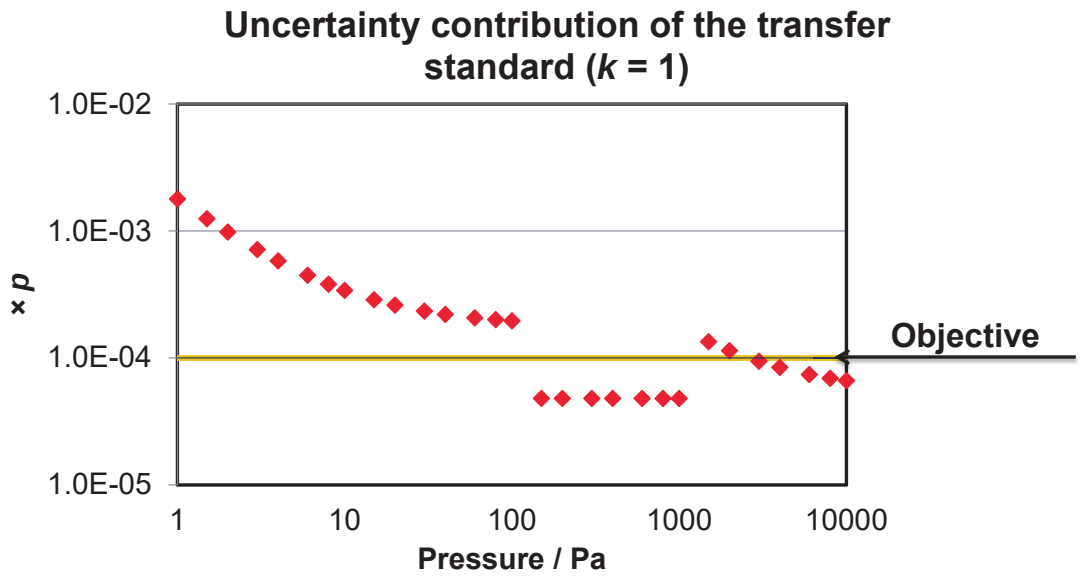
Uncertainty contribution For $u(T1)$ = 0.5 K	
× p	Pa
0	0.00068

Uncertainty budget (Lab B results)

Component	CDG 10k		CDG 1k		CDG100	
Calibration	-		3.4 × 10^{-5} × *p*		6.7 × 10^{-5} × *p*	
RSG stability	2.6 × 10^{-5} × *p*		-		-	
ESD Rescaling	3.3 × 10^{-5} × *p*		5.7 × 10^{-5} × *p*		1.1 × 10^{-4} × *p*	
Ambient temperature	-		-		-	0.00068 Pa
u(rescaling)	3.4 × 10^{-5} × *p*		6.7 × 10^{-5} × *p*		-	
Shape drift	2.2 × 10^{-5}× *p*	0.24 Pa	2.8 × 10^{-5} × *p*	0.0 Pa	7.6 × 10^{-6}× *p*	0.0018 Pa
TS Standard uncertainty	5.4 × 10^{-5}× *p*	0.12 Pa	4.8 × 10^{-5}× *p*	0.0 Pa	1.8 × 10^{-4}× *p*	0.0016 Pa

LNE le cnam

Uncertainty vs objective



LNE le cnam

Conclusion

- Contribution uncertainty of the transfer standard as low as $\mathbf{1 \times 10^{-4} \times p}$ is achieved between **100 Pa and 10 kPa**
- This contribution, however, also depends on one participant's pressure generation stability
- The principle of the transfer standard can be applied to implement a working standard with a low uncertainty in the range 1 Pa to 130 kPa:

F. Boineau, S. Huret, P. Otal, and M. Plimmer, 'A high-accuracy working standard for absolute pressure from 5 kPa to 130 kPa', *Int. J. Metrol. Qual. Eng.*, vol. 8, p. 26, 2017.

F. Boineau, S. Huret, P. Otal, and M. Plimmer, 'Development and characterisation of a low pressure transfer standard in the range 1 Pa to 10 kPa', *ACTA IMEKO*, vol. 7, no. 1, pp. 80–85, Apr. 2018.

Thanks for your attention

Investigation of the use of pressure balances as an alternative to mercury manometers

Virginia Marcos, David Herránz, Salustiano Ruiz, Nieves Medina

Centro Español de Metrología (CEM), Spain

Investigation of the use of pressure balances as an alternative to mercury manometers

14IND06pres2vac

EMPIR 2014

NMI: Centro Español de Metrología
(CEM Pressure Laboratory)

V. Marcos, D. Herranz, S. Ruiz and N. Medina

CEM CENTRO ESPAÑOL DE METROLOGÍA

Borås, Sweden 2018-05-17

Introduction:

Primary liquid column manometers, usually use mercury as a manometric liquid because of its stable density.

Mercury manometers are used by a few European NMIs and by numerous calibration, industrial and research laboratories.

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.

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Introduction:

Objective: to demonstrate that pressure balances can replace mercury manometers as standards for absolute barometric pressure with a target uncertainty of lower than $1.5 \times 10^{-5} \times |p| + 0.5$ Pa in the range from 100 Pa to 200 kPa.

- Comparison of pressure balances with mercury manometers
- Determination of the measurement capabilities of pressure balances for barometric pressure
- Analysis of CIPM and EURAMET key comparisons
- Alternative pressure standards to replace mercury manometers

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Comparison of pressure balances with mercury manometers

- Gas pressure balance
  - Range: 5 kPa – 200 kPa



- Digital piston gauge (FPG)
  - Range: 100 Pa – 15 kPa



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Comparison of pressure balances with mercury manometers

- Gas pressure balance
  - Vertical piston freely rotating within a cylinder: piston-cylinder assembly ⇒ A_{eff}
  - Pressure to be measured applied to the base of the piston, creating an upward vertical force which is equilibrated by the gravitational downward force due to loading masses submitted to the local gravity acceleration and placed on the top of the piston.
  - Pressure transmitted to the movable element by a gas.
  - Reference level of the balance: bottom of the piston when the balance is in equilibrium

Comparison of pressure balances with mercury manometers

- Gas pressure balance:

$$p = \frac{\sum_i m_i \cdot g}{A_p \cdot \left[1 + \left(\alpha_p + \alpha_c\right) \cdot \left(t - t_r\right)\right]} + \mu$$

  - Head correction:

$$p_m = p + \left(\rho_f - \rho_a\right) \cdot g \cdot \Delta h$$

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Comparison of pressure balances with mercury manometers

- FPG
  - FPG measures the pressure applied on a piston-cylinder assembly by means of the force exerted by the piston on a mass balance
  - Force applied measured by using a load cell to measure the force on the effective area of the piston
  - Cylinder has dual-cone shape and it is static (FPG8601 balance)
  - Lubrication gas: A gas is injected between the piston and the cylinder to minimize the effects due to friction

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Comparison of pressure balances with mercury manometers

- FPG

$$p = \frac{\dfrac{m_{\mathrm{cal}} \cdot \left(1 - \dfrac{\rho_{\mathrm{lub}}}{\rho_{\mathrm{m}}}\right)}{N_{cal}} \cdot (N + \delta N_1 + \delta N_2 + \delta N_3)}{A_{\mathrm{p}} \cdot \left[1 + (\alpha_{\mathrm{p}} + \alpha_{\mathrm{c}}) \cdot (t - t_{\mathrm{r}})\right]} + p_{\mathrm{ref,FPG}}$$

$$\delta N_1 = -K_{\mathrm{b}}\left(p_{\mathrm{lub}} - p_{\mathrm{lub},0}\right)$$

$$\delta N_2 = K_{\mathrm{d}}\left[\left(p_{\mathrm{lub}} - p_{\mathrm{ref}}\right) - \left(p_{\mathrm{lub},0} - p_{\mathrm{ref},0}\right)\right]$$

$$\delta N_3 = V_{\mathrm{lub}} \cdot g \left[\left(\frac{p_{\mathrm{ref}}}{T_{\mathrm{ref}}}\right) - \left(\frac{p_{\mathrm{ref},0}}{T_{\mathrm{ref},0}}\right) \cdot \frac{M}{K_{\mathrm{N}(0,\mathrm{t_r})}} \cdot Z_{\mathrm{g}(\mathrm{P_{ref,0}},\mathrm{T_{ref,0}})} \cdot R\right]$$

  - Head correction:

$$p_m = p + \left(\rho_f - \rho_a\right) \cdot g \cdot \Delta h$$

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Comparison of pressure balances with mercury manometers

- Preparation for calibration
  - Calibration room
    - Ambient temperature: within 18 ˚C and 22 ˚C, stabilised within ±1 ˚C
    - To keep a stable environment: control opening of doors and ventilation, and the movement of operator
  - Devices installation
    - Install the DUT over a stable table and as near the mercury column as possible
    - Minimise the height difference between the reference levels of the DUT and the mercury column. The height difference should be measured within ±0.5 mm
    - Minimise the tilt using the built-in spirit level or a laboratory spirit level
    - Use short, wide bore and clean pipework
    - Insure the cleanliness and the tightness of the tubing
    - Install appropriate drain to control the nature of the fluid in the tubing.
    - Attach a suitable temperature measurement system.

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Comparison of pressure balances with mercury manometers

- Preparation for calibration
  - Pressure generation
    - For gauge pressure
      - Use of nitrogen
      - Pressure input is adjusted to the range of the intercompared devices
    - For absolute pressure
      - A clean pump must be used
      - Use of a calibrated vacuum gauge to measure the residual pressure

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Comparison of pressure balances with mercury manometers

- Preparation for calibration
  - Preparation of the DUT
    - The DUT shall be placed in the laboratory at least 24 hours before the calibration, to reach thermal equilibrium
    - Clean the piston-cylinder assembly (if necessary)
    - Minimise the difference in height between the mercury column and the DUT
    - In the case of pressure balance: the loading masses should have been calibrated and the free rotation time and the descent rate of the piston must be examined.
    - In the case of FPG: the zero stability and stability in 15 kPa must be checked, and before the start of the comparison measurements, the FPG will be zeroed and then adjusted internally.

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Comparison of pressure balances with mercury manometers

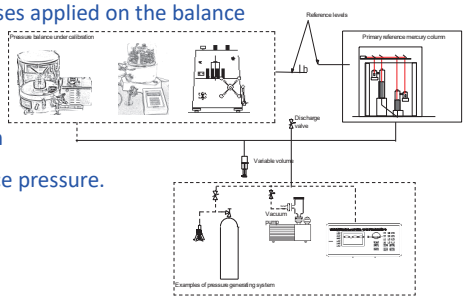
- Example of calibration procedure
  - Method
    - Comparison of the DUT and the mercury column when both are submitted to the same pressure
    - Scope: to determine the effective area referred to a reference temperature.
  - Determination of the effective area
    - Carry out 3-5 measuring series with at least six pressure points. Points should be spaced over the whole range of the DUT.
  - Calibration procedure: Gas pressure balance. Gauge pressure mode
    - Place the loading masses on the pressure balance to be calibrated
    - Adjust the pressure to equilibrate the balance under calibration.
    - Register at least the following information:
      - » Reference number or mass value of the loading masses applied on the balance.
      - » Temperature of the piston-cylinder assembly.
      - » Height difference between DUT and mercury column
      - » All the parameters required to calculate the reference pressure.

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Comparison of pressure balances with mercury manometers

- Example of calibration procedure
  - Calibration procedure: Gas pressure balance. Absolute pressure mode
    - Open the bell jar.
    - Place the loading masses on the pressure balance to be calibrated, so that their mass values correspond to the fixed nominal reference pressure.
    - Close and evacuate the bell jar at least until it reaches a pressure value below 10 Pa and at least during 5 min.
    - Adjust the pressure to equilibrate the balance under calibration.
    - Register at least the following information:
      - » Reference number or mass value of the loading masses applied on the balance
      - » Temperature of the piston-cylinder assembly
      - » Residual pressure in the bell jar
      - » Height difference between DUT and mercury column
      - » All the parameters required to calculate the reference pressure.

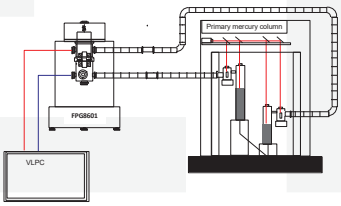


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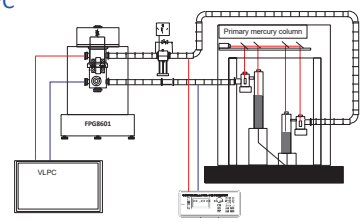
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Comparison of pressure balances with mercury manometers

- Example of calibration procedure
  - Calibration procedure: Digital piston gauge (FPG)
    - To adjust the pressure at the nominal reference value to be calibrated. In this case several methods can be used to reach a stable value:
      - Direct connexion + VLPC



      - Connexion of a differential pressure gauge in the pressure line + use of control system of mercury column and VLPC

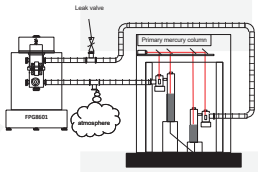


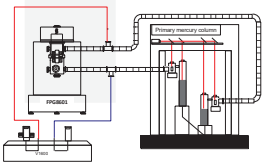
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Comparison of pressure balances with mercury manometers

- Example of calibration procedure
  - Calibration procedure: Digital piston gauge (FPG).
    - Direct connexion + low flow metering valve to compensate for the lubrication flow of the FPG





    - Direct connexion + V1600 balance (only in gauge mode)

- To register at least the following information:
  - » Counts number *N*, indicated by the FPG7601 and δN_1, δN_2 and δN_3.
  - » Temperature of the piston-cylinder assembly.
  - » Height difference between DUT and mercury column.
  - » All the parameters required to calculate the reference pressure.

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Comparison of pressure balances with mercury manometers

- Estimation of the uncertainty
  - Gas pressure balance
    - Type A uncertainty
      - Repeatability of the balance, estimated from the values of the standard deviation of the effective area
    - Type B uncertainty
      - Uncertainty of the mercury density
      - Uncertainty of air density
      - Uncertainty due to local gravity acceleration
      - Uncertainty due to height difference between the surfaces of mercury
      - Uncertainty of the residual pressure (absolute mode only)
      - Uncertainty due to the mercury temperature
      - Uncertainty due to the head correction

The combined standard uncertainty and the expanded uncertainty are calculated according to the Guide to the Expression of Uncertainty in Measurement (GUM)

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Comparison of pressure balances with mercury manometers

- Estimation of the uncertainty
  - FPG
    - Type A uncertainty
      - Repeatability of the balance, estimated from the values of the standard deviation of the effective area
    - Type B uncertainty
      - Uncertainty of the reference pressure
      - Uncertainty of the indications of the load cell;
      - Uncertainty due to the local gravity acceleration;
      - Uncertainty due to the temperature of the piston-cylinder assembly;
      - Uncertainty due to the thermal expansion coefficient of the piston-cylinder assembly;
      - Uncertainty due to the head correction;
      - Uncertainty due to tilt (negligible if it was duly avoided);
      - Uncertainty of the residual pressure (absolute mode only).

    The combined standard uncertainty and the expanded uncertainty are calculated according to the Guide to the Expression of Uncertainty in Measurement (GUM)

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Determination of the measurement capabilities of pressure balances for barometric pressure

- Uncertainties to 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

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Determination of the measurement capabilities of pressure balances for barometric pressure

- Uncertainties budget:

Uncertainty components	Uncertainty
Effective area	$u_1(p)/p \leq 6 \times 10^{-6}$
Mass	$u_2(p)/p = 2.6 \times 10^{-6}$
Temperature of the piston-cylinder assembly	$u_3(p)/p = 2.6 \times 10^{-6}$
Thermal expansion coefficient of the piston-cylinder assembly	$u_4(p)/p = 9 \times 10^{-7}$
Local gravity acceleration	$u_5(p)/p << 10^{-6}$
Residual pressure	$u_6(p) = u_\mu(p) = u(\mu) < 0.25$ Pa
Remaining effects	$u_7(p)/p = 2 \times 10^{-6}$
Expanded uncertainty ($k = 2$)	$\boldsymbol{U(p) = 1.5 \times 10^{-5} \times \lvert p \rvert + 0.5}$ **Pa**

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Analysis of CIPM and EURAMET key comparisons

Mercury manometers and pressure balances were used as reference standards.

The analysis is focused on the uncertainties achieved for the transfer standards by the different types of the reference pressure standards.

Comparison	Range	Participants	Comments
CCM.P-K2	10 kPa-120 kPa absolute	NMIA (Australia), INRIM (Italy), NPL (UK), LNE (France), NIST (USA), PTB (Germany), METAS (Switzerland), NCR (Canada).	Only the 10 kPa will be included in the report
CCM.P-K4	1 Pa - 1000 Pa absolute	CSIRO (current NMIA, Australia), IMGC (current INRIM, Italy), KRISS (Korea) NIST (USA).	There are other participants, but with static expansion systems
CCM.P-K5	1 Pa -1000 Pa differential with 100 kPa line pressure	MSL (New Zealand), CSIRO (currently NMIA, Australia), NPL (UK), NIST (USA).	
CCM.P-K6	10 kPa-120 kPa gauge	NIM (China), VSL (Netherlands), NPL (UK), NIST (USA), PTB (Germany), METAS (Switzerland), NCR (Canada).	Only the 10 kPa will be included in the report. VSL did not present results for this target pressure.
EUROMET.M.P-k1.a	1 Pa - 1000 Pa absolute	OMH (currently MKEH, Hungary)	There are other participants, but with static expansion systems
EUROMET.M.P-k4.2010	1 Pa -15 000 Pa absolute and gauge	CMI (Czech R), INRIM (Italy), LNE (France), MIKES (currently VTT, Finland), PTB (Germany)	
EUROMET.M.P-S2	30 Pa -7 000 Pa absolute	CMI (Czech R), PTB (Germany)	
EURAMET.M.P-S12	300 Pa -15 000 Pa negative gauge pressure	CMI (Czech R), INRIM (Italy).	

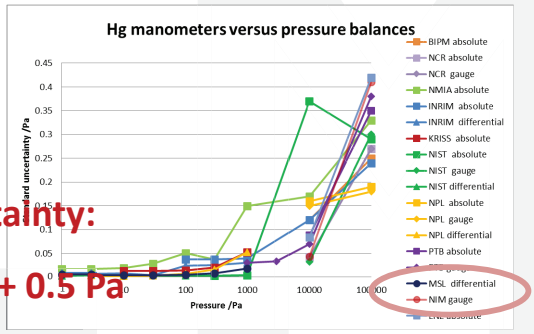
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Analysis of CIPM and EURAMET key comparisons

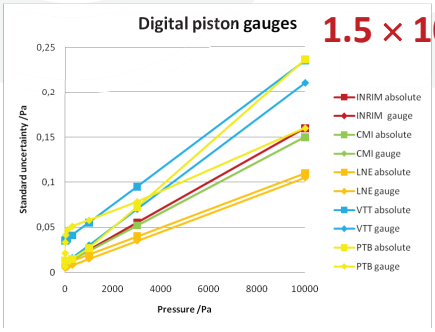
Mercury manometers and pressure balances (except digital piston gauges)

- Uncertainties of pressure balances are similar or even lower than uncertainties obtained by mercury manometers



Target uncertainty:

$1.5 \times 10^{-5} \times |p| + 0.5$ Pa



Pressure balances

Digital piston gauges

- Uncertainties are larger than in the case of using mercury manometers.

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Alternatives pressure standards to replace mercury manometers

- Gas pressure balance
- FPG
- Dual Fabry-Perot cavity refractometer:
  - Improvement: use of gas modulation refractometry (GAMOR)

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Thank you for your attention
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Stability study of the low-range CDGs

Martin Vičar[1], Janez Šetina[2]

[1] Český metrologický institute (CMI), Czech Republic
[2] Institute of Metals and Technology (IMT), Slovenia

Stability study of the low-range CDGs

Martin Vičar, Janez Šetina
CMI, IMT

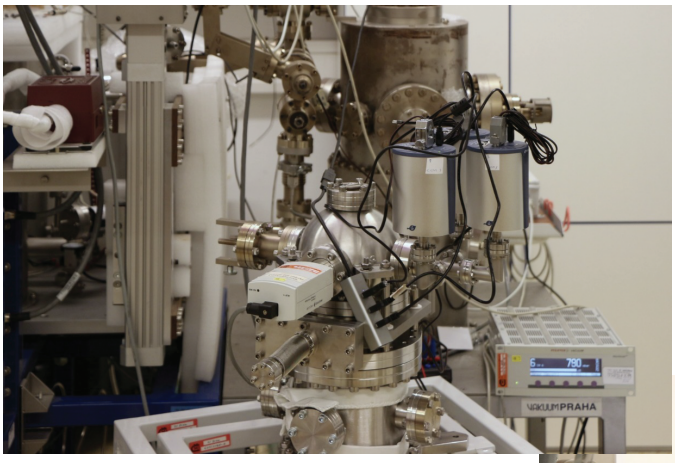
pres2vac
-A metrology research project within the European Union Horizon 2020 framework.

- The object of the study was Inficon CDG 045 Dhs (10 mTorr) of absolute pressure range (0.013 to 1.33) Pa. They are based on Al_2O_3 ceramic sensor and have accuracy 0.2% of the measured value.
- 3 CDG gauges were provided by Inficon.
- They were measured 3 times: CMI – IMT – CMI.

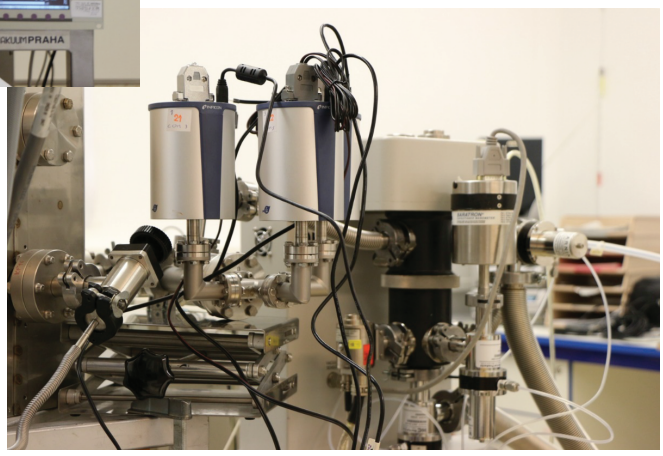
- CMI – March 2017.
- IMT – March/April 2018.
- CMI – May 2018.



The first measurements at CMI



Using the „transition range expansion“ and the FPG.



The measurements at IMT

were performed by a static expansion.

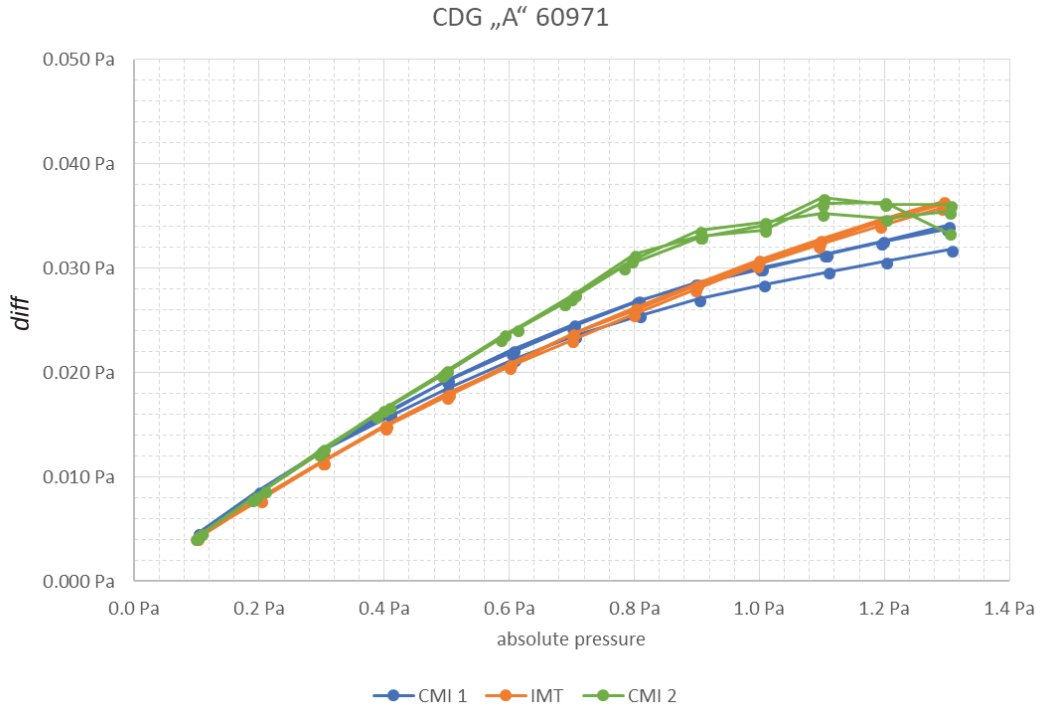
The second measurements at CMI

The turbo pump of our „transition expansion“ broke. The preliminary measurements were performed by an SRG and the FPG.

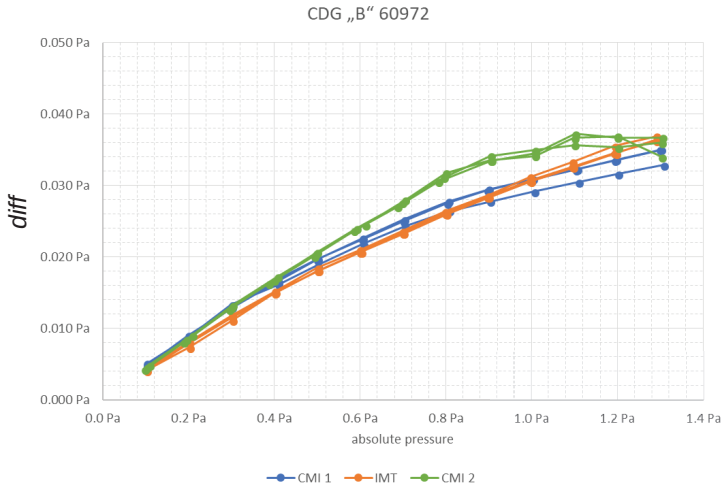
Digital outputs of CDGs were logged using TGExpress software

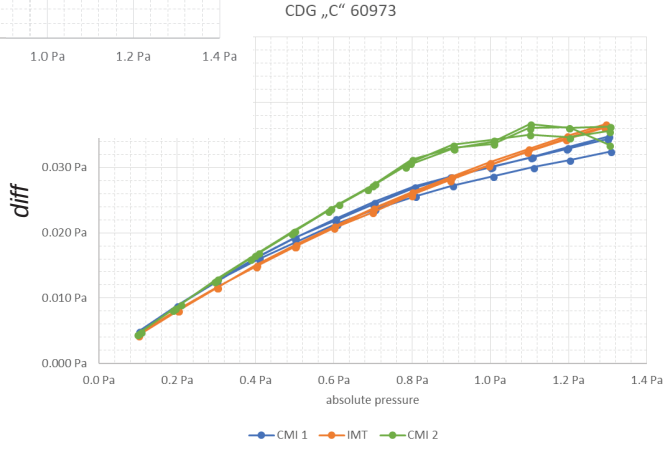


The first CDG gauge during three measurements (3 series each); *diff* = P_{ind} - P_{ref}

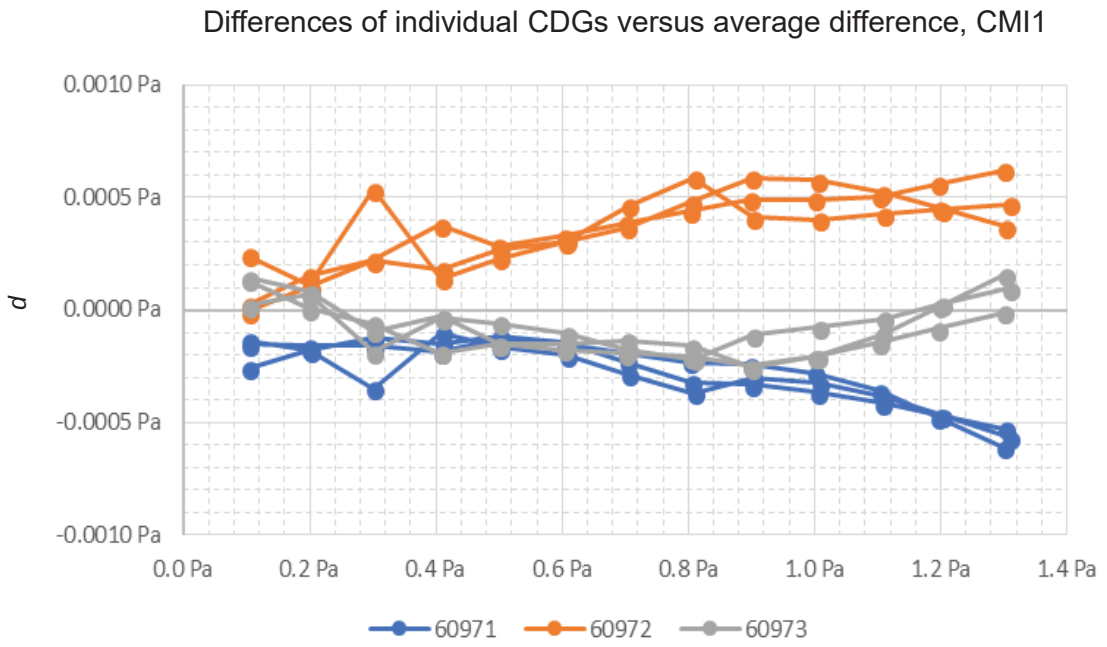


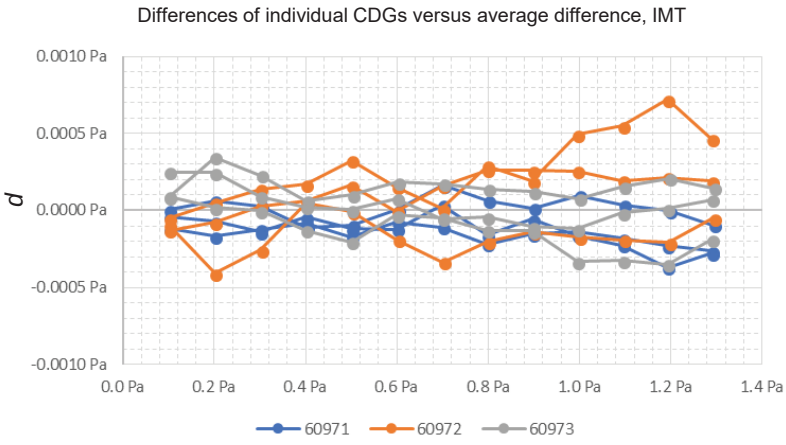
The second and the third CDG gauge

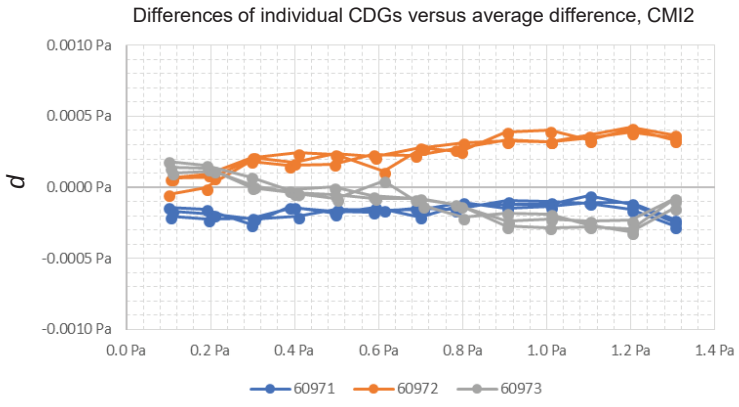


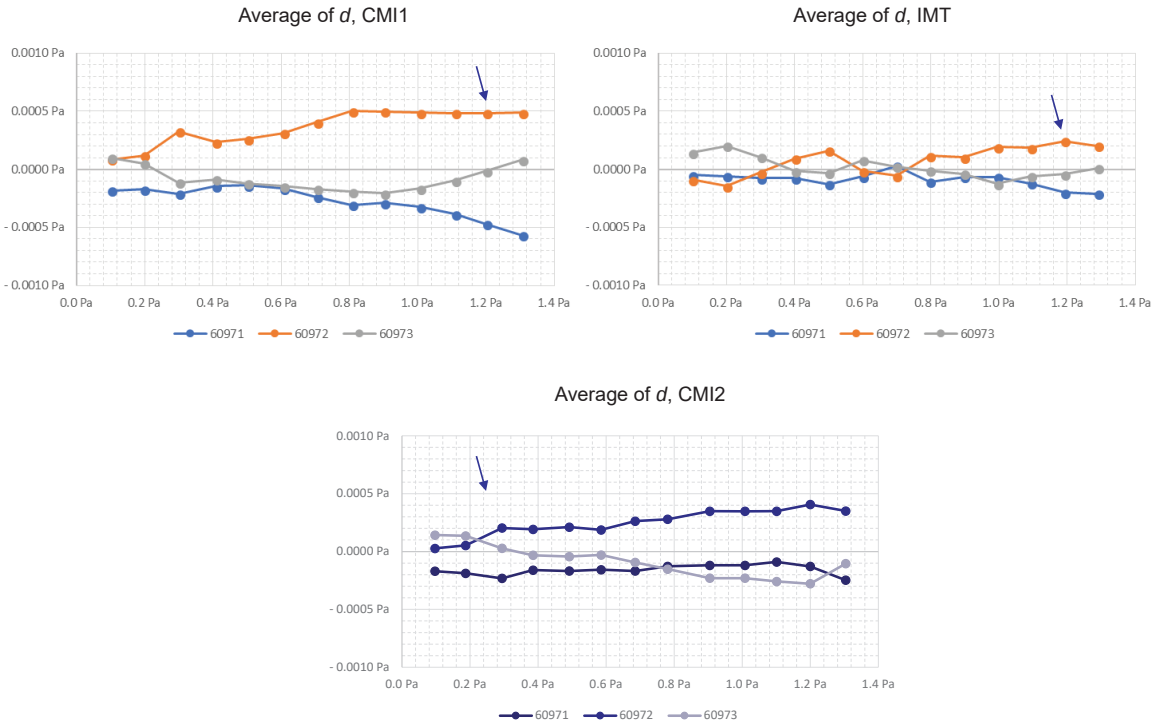


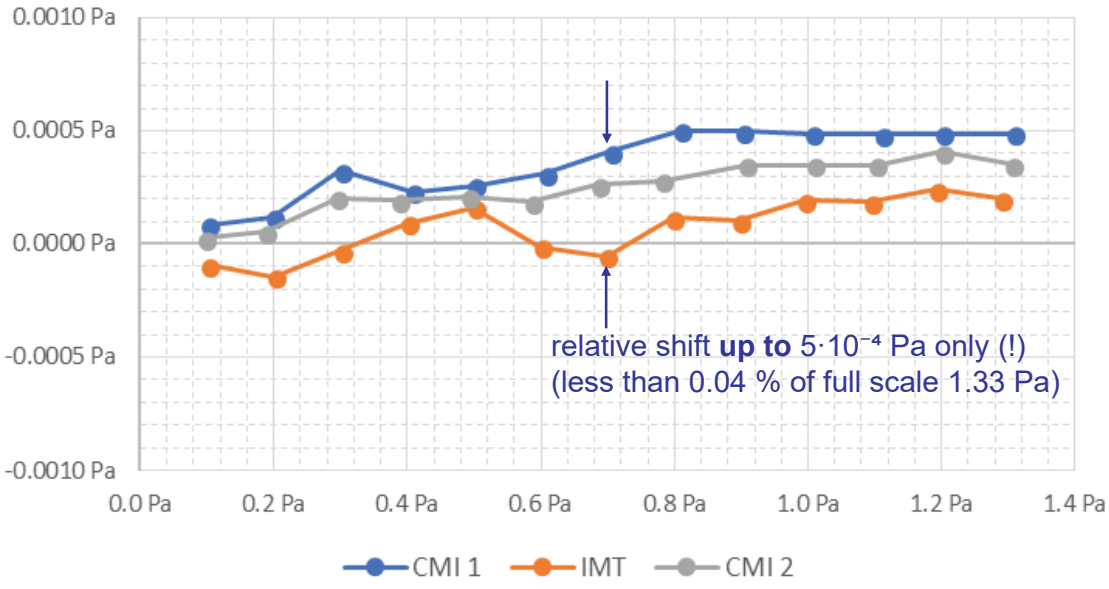
The long-term stability may be estimated from the shifts of the individual gauge deviations $d = diff - diff_{av}$



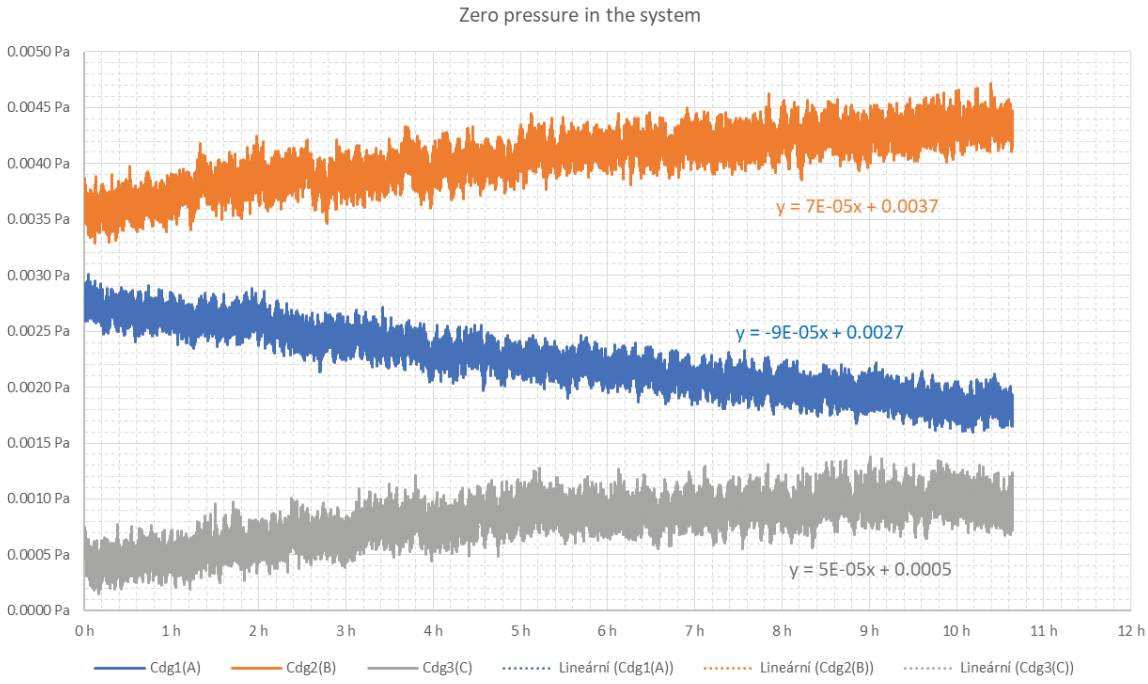




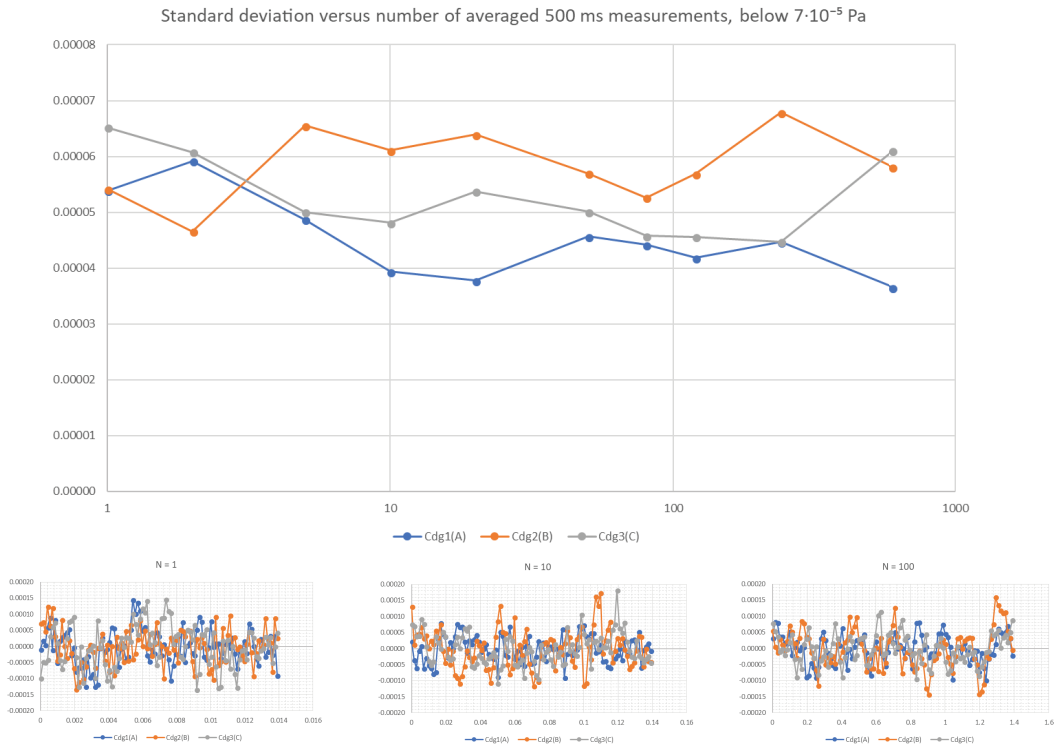

Average of d, CMI1
0.0010 Pa
0.0005 Pa
0.0000 Pa
- 0.0005 Pa
- 0.0010 Pa
0.0 Pa
0.2 Pa
0.4 Pa
0.6 Pa
0.8 Pa
1.0 Pa
1.2 Pa
1.4 Pa
60971
60972
60973
Average of d, IMT
0.0010 Pa
0.0005 Pa
0.0000 Pa
- 0.0005 Pa
- 0.0010 Pa
0.0 Pa
0.2 Pa
0.4 Pa
0.6 Pa
0.8 Pa
1.0 Pa
1.2 Pa
1.4 Pa
60971
60972
60973
Average of d, CMI2
0.0010 Pa
0.0005 Pa
0.0000 Pa
- 0.0005 Pa
- 0.0010 Pa
0.0 Pa
0.2 Pa
0.4 Pa
0.6 Pa
0.8 Pa
1.0 Pa
1.2 Pa
1.4 Pa
60971
60972
60973


Average of d, CDG 60972
0.0010 Pa
0.0005 Pa
0.0000 Pa
-0.0005 Pa
-0.0010 Pa
0.0 Pa
0.2 Pa
0.4 Pa
0.6 Pa
0.8 Pa
1.0 Pa
1.2 Pa
1.4 Pa
relative shift up to 5·10⁻⁴ Pa only (!)
(less than 0.04 % of full scale 1.33 Pa)
CMI 1
IMT
CMI 2

Short-term stability of the CDG signal at zero pressure: slope < 10^{-4} Pa / h



Short-term stability – typical σ of the output signal



Conclusions

- The second set of measurements at the CMI must be repeated after repairing our turbopump.
- However, the first results are very promissing.

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Thank you very much for your attention.

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Temperature controlled transfer standard package for high accuracy pressure transducers with improved temperature stability

Rifat Kangi

TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Turkey

pres2vac
-A metrology research project within the European Union Horizon 2020 framework.

TÜBİTAK UME

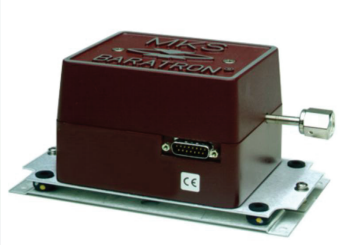
EMPIR EURAMET
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Temperature controlled transfer standard package for high accuracy pressure transducers with improved temperature stability

Rifat Kangi

RISE-Borås, Sweden
17th May, 2018

High accuracy capacitance diaphragm gauge (CDG)

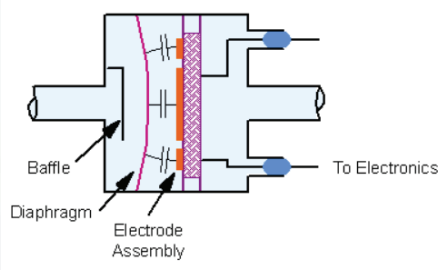


Type 690 CDG (MKS Instruments. Inc.)

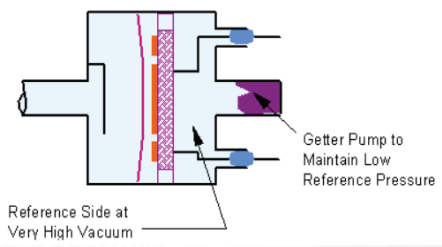
0.1 Pa – 100 kPa (optimum range)
4×10^{-3} …. 1×10^{-3} (lowest uncertainty, k=2)

Suitable as secondary and reference standard.
- High accuracy
- Highest resolution
- Good linearity and repeatability
- Fair robustness against temperature changes
- Good transport and long-term stability

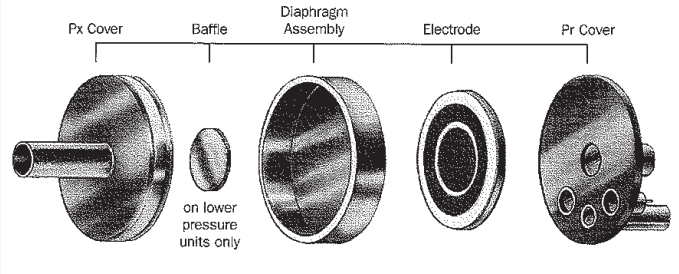
High accuracy capacitance diaphragm gauge (CDG)



Baratron® Capacitance Manometer



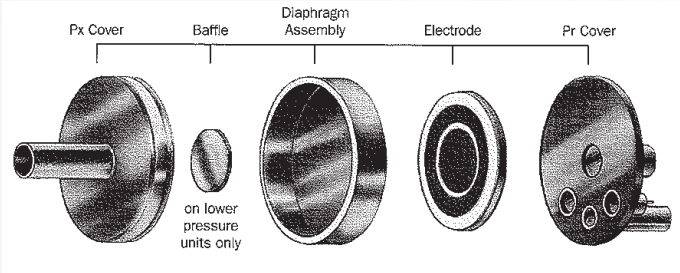
The Sensor Capsule



3

Factors limiting measurement performance

At the lowest pressure:
- Random noise
- Zero-pressure short-term instability (hours to day)
- Thermal transpiration at absolute pressures below 100 Pa (for heated gauges)



All CDGs are subject to errors due to differences in expansion coefficients of the sensor materials.
- Electrode substrate material (Ceramic forsterite)
- Sensor body and diaphragm material (Inconel)

4

Ways to minimize these effects

1. Temperature compensation:

• Improve coefficient by using a temperature-sensitive device (attached to the sensor to compensate the output signal appropriately for temperature changes (thermal lags and nonlinear temperature coefficient)

2. Enclose the sensor in an accurately temperature-regulated environment

• This minimized temperature changes. Cast aluminum housing/shell with cartridge-type heaters will achieve temperature coefficients less than 4ppm on zero and 20 ppm on span

5

Thermal transpiration effects

Thermal transpiration (non-linear gas-species-dependent response of the gauge at absolute pressures below 100 Pa.)

When the heated CDG is operated at the temperature of 45º C, the maximum measurement error for a system controlled at 20º C will is approximately 4%.

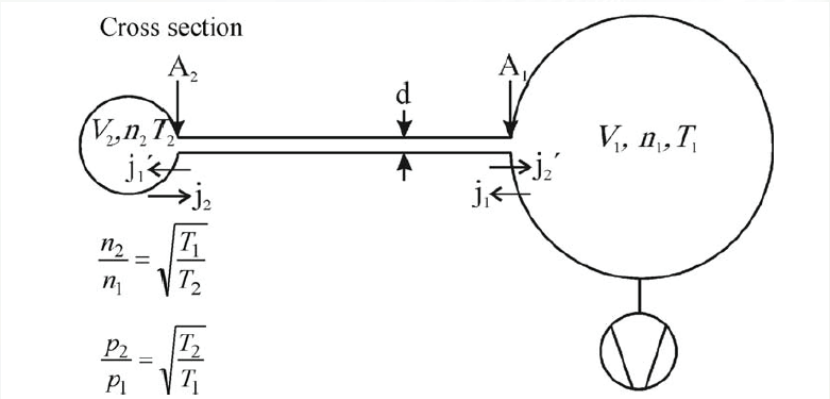


Fig. 16: Illustration of the effect of thermal transpiration. Two volumina of different temperature are connected together where the Knudsen number is > 0.5. From *Wutz Handbuch Vakuumtechnik* by K. Jousten (ed.), Vieweg Verlag.

6

Corrections to the CDG measurements

Takaishi and Sensui's equation

$$\frac{p_2}{p_1} = \frac{AX^2 + BX + C\sqrt{X} + \sqrt{\frac{T_2}{T_1}}}{AX^2 + BX + C\sqrt{X} + 1}$$

$A = A^*(T^*)^{-2}$
$B = B^*(T^*)^{-1}$
$C = C^*(T^*)^{-0.5}$
$T^* = 0.5\,(T_1+T_2)$ and $X = 0.133\,p_2\,d$.

Values of the constants A*, B* and C*			
Gas	**A***	**B***	**C***
He	1.50×10^5	1.15×10^2	19.0
N_2	1.20×10^6	1.00×10^3	14.0
Ar	1.08×10^6	8.08×10^2	15.6
SF_6	1.53×10^7	2.42×10^4	4.4

From their results Takaishi and Sensui derived the following simple empirical equations relating the constants A*, B* and C* to the molecular diameter (D) of the gas. Equations can be written:

$A^* = 1.4 \times 10^4 \exp(1.17 \times D \times 10^{10})$
$B^* = 5.6 \exp(1.40 \times D \times 10^{10})$
$C^* = (1.10 \times 10^{-8}/D) - 14$
D - molecular diameter.

Takaishi and Sensui's Correction

K F Poulter et al: Thermal transpiration correction in capacitance manometers

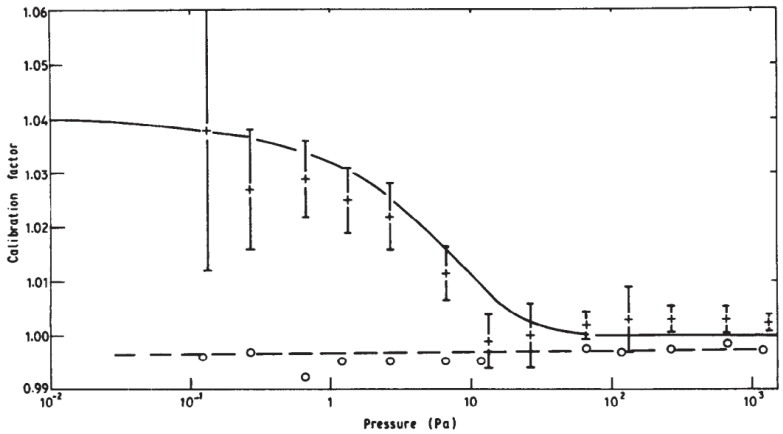
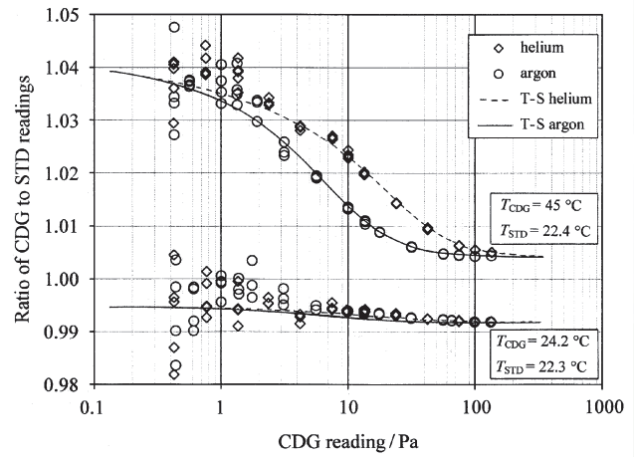


Figure 2. The nitrogen calibration for the first capacitance manometer with the heater on (+) and off (○). The error bars represent the random uncertainty in each point at a 95% confidence level. The solid curve is calculated using the Takaishi and Sensui equation.

Hybrid systems

Temperature stability ± 20 mK for room temperature changes of up to a few degrees



Absolute-mode calibration data for a 133 Pa CDG when controlled at 45 C and at 24.2 C.

Due to heating from the electronics the error induced by thermal transpiration effect is up to 0.5 %

Metrologia, 1999, **36**, 617-621 9

Newly constructed temperature controlled transfer standard package

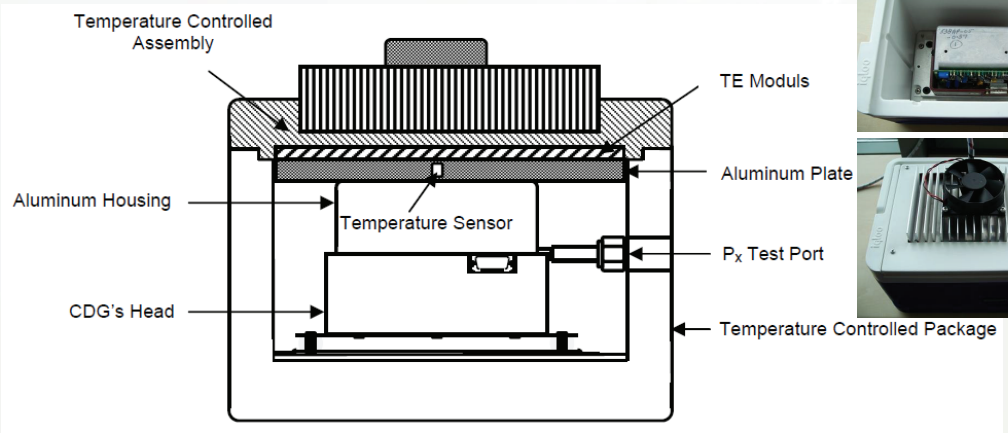
Stability is achieved by providing the accurate and precise temperature control of the CDG placed inside of miniature environmental box and kept at the fixed temperature with stability better than ± 2 mK.

Transfer standard package minimizes the ambient temperature effects upon the sensor, denies the need to apply a correction for thermal transpiration effect and thereby increases accuracy of the transducer.

10

Newly constructed temperature controlled transfer standard package

TCTS package consists of a high accuracy CDG placed inside a thermal enclosure with a temperature control assembly and external temperature controller



To reach better temperature stability of the CDG's sensor the plate surface of the temperature control assembly directly contacts the upper surface of the aluminum housing of the sensor where before that the upper part of the CDG's cover body has been removed.

11

Newly constructed temperature controlled transfer standard package

The CDG's own heater is switched off.

External temperature controller is used to control the electrical current to the (TE) modules. This arrangement enables the CDG's sensor to be controlled to better than ± 2 mK for room temperature changes of up to several degrees.

The package allows perform measurements by using CDGs at any fixed temperature in the range from 15 °C to 30 °C with stability better than ± 2 mK.

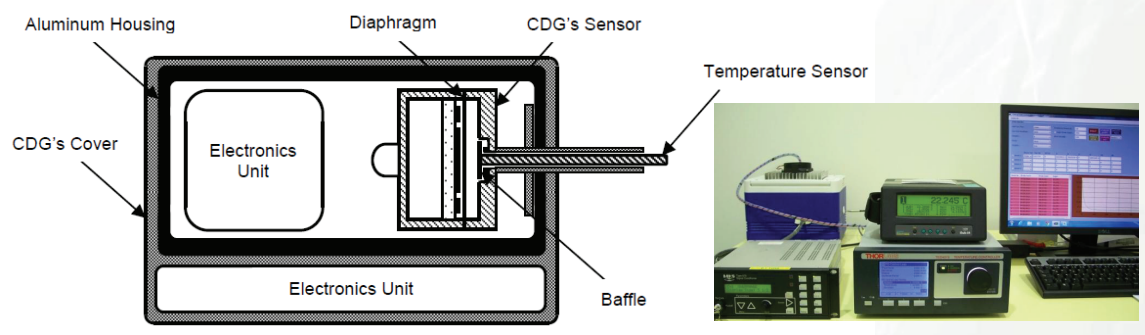
To determine the real temperatures of the CDG's sensor inside of aluminum housing in the temperature range from 15 °C to 30 °C some preliminary measurements were performed.

Initially, sensor temperature of the temperature control assembly has been checked with previously calibrated thermistor type temperature sensor at room temperature and corrected value has been set to the temperature controller.

12

Newly constructed temperature controlled transfer standard package

CDG's sensor temperatures were measured in the temperature range from 15 °C to 30 °C using same calibrated temperature sensor.



The temperature control was provided at different fixed temperatures (15 to 30 °C).

After stabilization of CDG's sensor temperature controller setting temperature and CDG's sensor real temperature data were taken.

Finally, when all the data of two temperature sensors in the range from 15 °C to 30 °C have been taken and behavior of dependence of two temperature sensors was determined, temperature controller can easily set to provide room temperature conditions.

13

Results

Two CDGs (A and B) with their own transfer standard packages were evaluated for **zero instability, zero drift** and **zero temperature coefficients**.

To determine CDG's zero instability absolute CDG A (130 Pa full scale) and CDG B (1.3 kPa full scale) were placed into the package 1 and 2 respectively.

Both CDGs were operated with their own control and measuring unit where own heaters were switched off.

The temperature controls of both CDGs were provided by their own temperature controlled transfer standard packages at room temperature 20 °C.

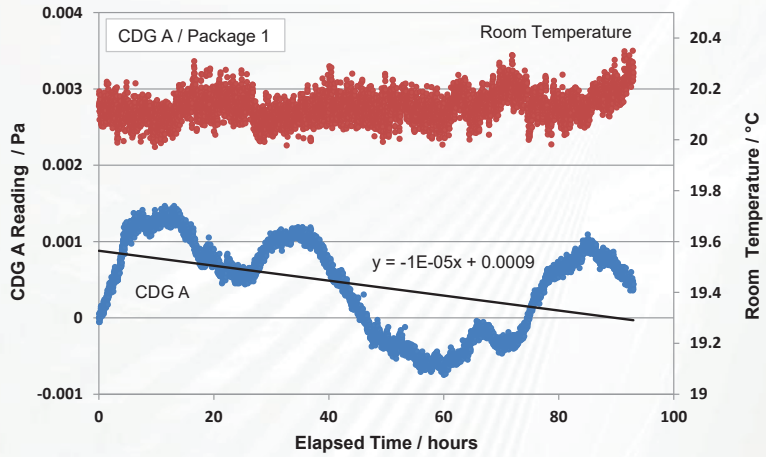
1. Noise-limited pressure resolution:
   a) CDG A = 6.6×10^{-7}
   b) CDG B = 4.3×10^{-7}

(given by twice the STD of repeated readings at a stable pressure as a function of CDG's FS range).

14

Results

2. Results for zero instabilities and room temperature changes during measurements.



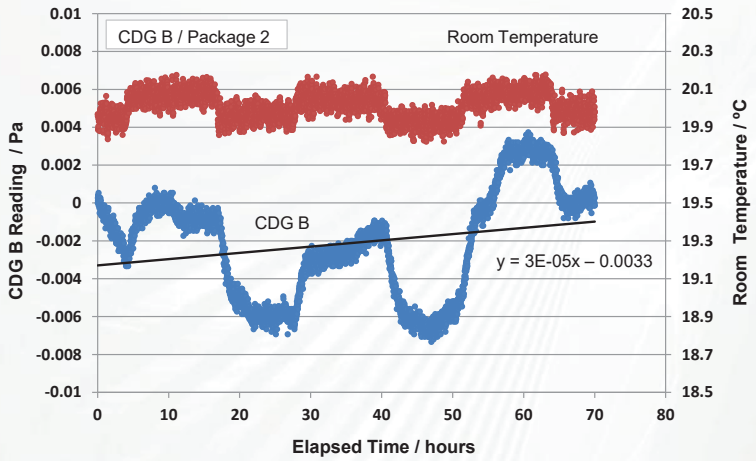
Plot of zero-pressure stability and room temperature of CDG A (130 Pa) in the Package 1 within 93 hours

Maximum drift rate of 0.01 mPa h^{-1} and ± 1.1 mPa maximum zero instability over 93 hours

15

Results

2. Results for zero instabilities and room temperature changes during measurements.



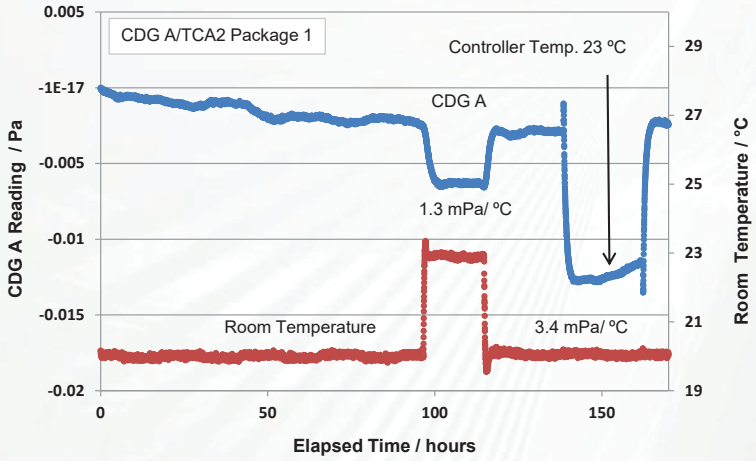
Plot of zero stability and room temperature of CDG B (1.3 kPa) in the Package 2 within 70 hours

Maximum drift rate of 0.03 mPa h^{-1} and ± 5 mPa maximum zero instability over 70 hours

16

Results

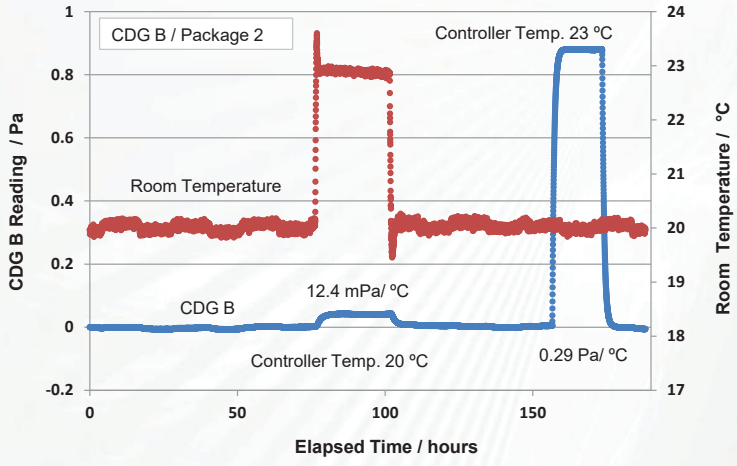
3. Influence of room temperature changes on CDG's zero-pressure



Plot of zero stability and room temperature of CDG A (130 Pa) in the Package 1

Results

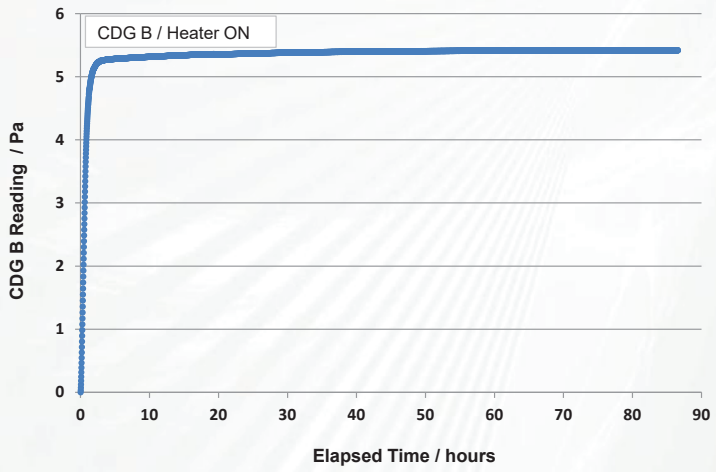
3. Influence of room temperature on CDG's zero-pressure



Plot of zero stability and room temperature of CDG B (1.3 kPa) in the Package 2

Results

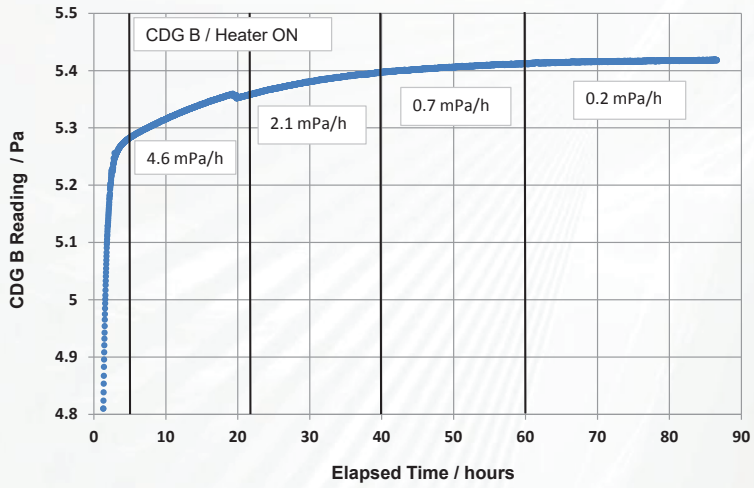
4. CDG B's zero-pressure stability result where CDG's own heater was switched on (45 °C)



19

Results

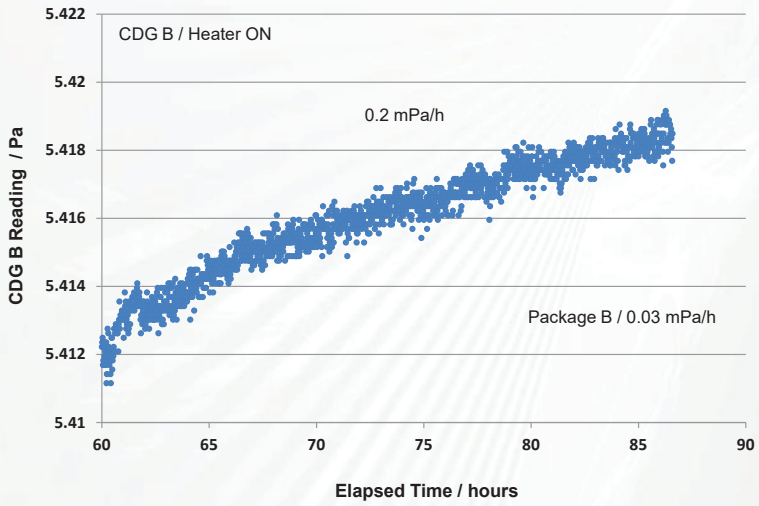
4. CDG B's zero-pressure stability result where CDG's own heater was switched on (45 °C)



20

Results

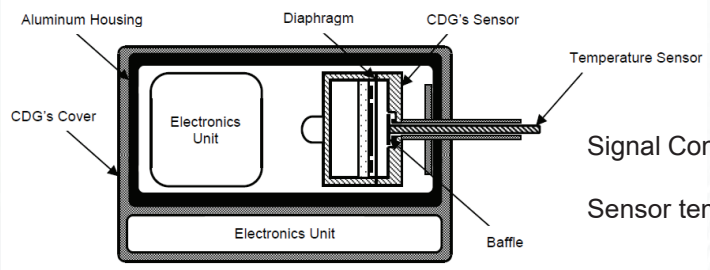
4. CDG B's zero-pressure stability result where CDG's own heater was switched on (45 °C)



Same CDG has zero-pressure stability 7 times better in transfer standard package

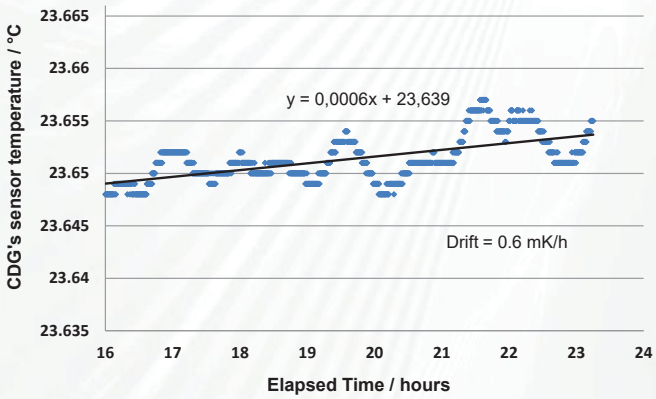
Results

6. Electronics' influence on CDG's sensor temperature



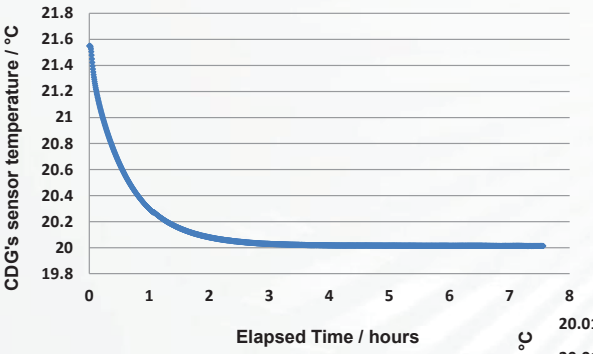
Signal Conditioner 670B's Heater OFF

Sensor temp = Room temp + 3,05 °C



Results

8. CDG's sensor temperature stability at 20 °C



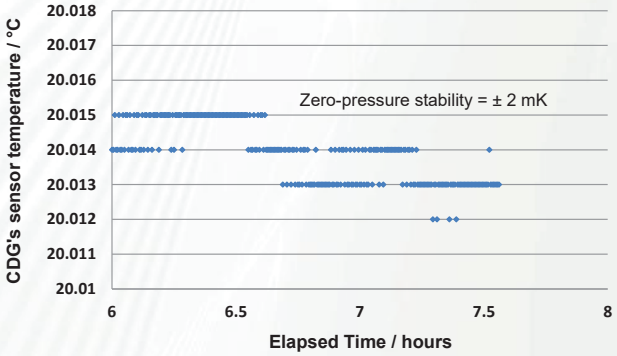
Signal Conditioner 670B's Heater is OFF

CDG is inside of Transfer Standard Package

Sensor temp = 20 °C

Temperature Stability = ± 2 mK (after 6 h)





23

Conclusions

1. Developed TCTS packages are suitable for intercomparisons of pressure standards (13 Pa – 13 kPa)
2. Principally new technique for temperature control of CDG's is proposed (which allows control CDG's sensor in the range from 15 °C to 30 °C with stability better than ± 2 mK)
3. TCTS packages have demonstrated excellent short-term zero stability ± 5 mK (max) and a maximum drift rate of 0.03 mPa h^{-1}
4. TCTS package minimizes the ambient temperature effects upon the sensor, denies the need to apply a correction for thermal transpiration effect and thereby increases accuracy of CDGs

24

Thank you !



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