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Towards 7 MPa Pressure Standards with $1{\cdot}10^{-6}$ Uncertainty

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Abstract

To determine the Boltzmann constant by means of the dielectric-constant gas thermometer technique, pressure standards measuring absolute pressures in helium in the range of 7 MPa with a relative standard uncertainty of $1{\cdot}10^{-6}$ are required. The feasibility of such a low uncertainty with pressure balances whose piston-cylinder (p-c) assemblies are characterised by dimensional measurements is considered. It is shown that the target uncertainty can be achieved when p-c assemblies with a nominal effective area of 20 cm^2 are used as primary standards characterised by the state-of-the-art dimensional techniques available at PTB, advanced theories for the effective area calculation are applied, improved cross-float techniques are employed which involve differential pressure cells (DPCs) by means of which 2 cm^2 p-c assemblies covering the 7 MPa range can be linked to the 20 cm^2 ones, and a consistency better than $1{\cdot}10^{-6}$ within a group of p-c units can be demonstrated. Some results of dimensional and cross-float measurements as well as of a DPCs investigation are reported.

1. Introduction

Recently PTB has started a project which is aimed at re-determining the Boltzmann constant (k). Within the scope of this project, the dielectric-constant gas thermometer technique (DCGT) will be applied for a simultaneous measurement of the pressure (p), the temperature (T) and the electrical capacity (c) in helium [1]. The Boltzmann constant can be determined by combining the equation of state with the Clausius-Mossotti equation to form the expression;

$$\frac{p}{T}\frac{\alpha_0}{\varepsilon_0 k} = \chi\left[1 + \frac{B}{N_A\alpha_0/\varepsilon_0}\chi + \frac{C}{\left(N_A\alpha_0/\varepsilon_0\right)^2}\chi^2 + \ldots\right] \quad (1)$$

with N_A and ε_0 being the Avogadro and the electric constants, B and C the second and third density virial coefficients, α_0 the static electric dipole polarisability of the gas atom and c the gas dielectric susceptibility.

For helium, B, C and α_0 can be calculated theoretically, whereas χ is determined by capacitance (c) measurements at vacuum and pressure conditions, $\chi = c(p)/c(0)-1$. In the first phase of the project, it is intended to demonstrate that the new k value is consistent with the known value determined by the acoustic gas thermometry method [2] and recommended by CODATA for international use [3]. Afterwards, with the new Boltzmann constant being "frozen", DCGT could be used to link the temperature unit to the pressure and electric units and to replace thus the definition of the kelvin which is currently based on the temperature of the triple point of water. The realisation of the new temperature scale is attractive because the kelvin will no longer be linked to a material (water) property and will be consistent with the definition of the thermal energy kT which appears in fundamental laws of physics. In order to achieve the relative standard uncertainty of k of about $2{\cdot}10^{-6}$, as planned for the first phase of the project, absolute pressure measurements should be performed with a relative standard uncertainty of about $1 \cdot 10^{-6}$ in the range from 2 MPa to 7 MPa, which is effective for the DCGT measurements. Pressure balances seem to be the only instruments with which the required accuracy can be achieved.

2. Feasibility Analysis

Measuring pressures between 2 MPa and 7 MPa with a relative standard uncertainty of $1 \cdot 10^{-6}$ is a challenging target whose feasibility should be carefully analysed. In the last CCM comparison in the 7 MPa range of gauge pressure, CCM.P-K1.c [4], carried out between 1998 and 1999 with the participation of five national metrology institutes (NMIs), NIST (USA), PTB (Germany), LNE (France), INRIM (Italy) and NMIJ (Japan), maximum relative differences between the results of the participants of $25 \cdot 10^{-6}$ and their standard uncertainties of $(8\text{-}17) \cdot 10^{-6}$ were observed. This comparison makes clear that it is not possible to measure pressures up to 7 MPa with an uncertainty of 1·10-6 when applying the commercial pressure balances and rather conservative uncertainty evaluation models that are normally used in routine calibrations and were also applied by the participants in that comparison. At PTB, a pressure balance model PG7601, equipped with a 0.5 cm^2, 7 MPa piston-cylinder (p-c) assembly 0302 produced by DH Instruments was used as a laboratory standard. The uncertainty budget for pressure measured using this pressure standard is presented in Table 1 in the column entitled "Actual".

The first seven uncertainty sources were insignificant in the uncertainty budget, but they will have to be considered when a final uncertainty of $1 \cdot 10^{-6}$ is envisaged.

The gravity acceleration (g) has been determined in the pressure laboratory with a relative standard uncertainty of $1 \cdot 10^{-7}$. However, the ebbs and flows as well as the changing position of the sun and the moon relative to the earth produce temporal relative changes in g of up to $5 \cdot 10^{-7}$. In addition, it was assumed that the height position of the weights loading the piston can vary within 0.5 m. This, due to a vertical gradient of g, causes an uncertainty of $1.6 \cdot 10^{-7}$. Taking into account the temporal function of g and the actual height position of each weight it is possible to determine g within $1 \cdot 10^{-7}$.

The air density was calculated from the air temperature (t_{amb}), the pressure (p_{amb}) and the relative humidity (RH) by means of a well-known BIPM formula which is given in [5], and by simply taking RH = (60±40)%. Measuring the ambient conditions with $u(t_{amb})$ = 0.1 °C, u (p_{amb}) = 10 Pa, u (RH) = 1%, and taking into account the relative accuracy of the formula within $1.3 \cdot 10^{-4}$ as well as the effect of the CO_2 concentration within $1.6 \cdot 10^{-4}$, the air density can be determined with an uncertainty of $4.7 \cdot 10^{-4}$ and will contribute only $7 \cdot 10^{-8}$ to the relative pressure uncertainty.

Table 1
Uncertainty budget for a pressure of 7 MPa, uncertainty sources and their uncertainties *($u(x_i)$)*, and relative contributions to the pressure uncertainty *($u_i(p)/p$)*, actually measured values and target uncertainties (sources with contributions $u_i(p)/p < 10^{-7}$ are not listed)

Quantity	Actual		Target	
	$u(x_i)$	$u_i(p)/p \times 10^6$	$u(x_i)$	$u_i(p)/p \times 10^6$
Gravity acceleration	$5.4 \cdot 10^{-7}\ x_i$	0.54	$1.0 \cdot 10^{-7}\ x_i$	0.10
Air density	$3.6 \cdot 10^{-3}\ x_i$	0.54	$4.7 \cdot 10^{-4}\ x_i$	0.07
Temperature measurement	20 mK	0.18	5 mK	0.05
Temperature inhomogeneity	71 mK	0.64	10 mK	0.09
Verticality	1.0 mm / m	0.50	0.1 mm / m	0.01
Ring weights on piston	20 mg	0.58	$1.0 \cdot 10^{-7}\ x_i$	0.10
Pressure distortion coefficient	$2.4 \cdot 10^{-7}$ MPa^{-1}	1.70	$5.0 \cdot 10^{-8}$ MPa^{-1}	0.35
Zero-pressure effective area	$7.6 \cdot 10^{-6}\ x_i$	7.60	$8.6 \cdot 10^{-7}\ x_i$	0.86
Type B uncertainty		7.90		0.95
Type A uncertainty		2.50		0.20
Combined uncertainty		8.30		0.97

A calibration of the platinum resistance thermometer (PRT), measuring the temperature of the p-c assembly (t) with u (t) = 5 mK instead of 20 mK, will reduce the contribution to the pressure uncertainty to $5{\cdot}10^{-8}$.

A more serious problem is the temperature inhomogeneity in the pressure balance, which means that the temperature measured by the PRT is not equal to the temperature of the p-c assembly. The temperature gradients occur mainly due to electronics, sensors and the electromotor placed in the basement of modern pressure balances, as well as to energy dissipation in the p-c gap and an instability of the ambient temperature. The value of 71 mK was estimated from the experimental temperature change rates and from the construction of the mounting post of the pressure balance. To reduce this uncertainty, a pressure balance of special design is required with all potential heat sources being removed from the basement and a PRT being kept in a good thermal contact with the cylinder. The pressure balance should be operated in the piston's free rotation regime. A high ambient temperature stability should be provided. And, finally, the temperature distribution in the mounting post and in the cylinder should be controlled by several thermocouples. With these arrangements, a thermal homogeneity within 10 mK appears to be a realistic target which will contribute $9{\cdot}10^{-8}$ to the pressure uncertainty.

The uncertainty of circular bubble spirit levels used in commercial pressure balances to control the verticality of the piston is usually about 1 mm / m. With accurate linear spirit levels as attached to the PTB primary pressure balances, the horizontality is controlled within 0.04 mm / m. The laboratory floor was shown to be stable within 0.1 mm / m. All this shows that it is possible to control the piston verticality within 0.1 mm / m with a contribution of $1{\cdot}10^{-8}$.

The smallest relative standard uncertainty of the ring weights used with pressure balances is at PTB normally limited by $5{\cdot}10^{-7}$. This is due to a non-optimal shape and surface quality as well as to long recalibration periods (5 years). The mass measurement itself is possible with a relative uncertainty below $1{\cdot}10^{-7}$. With an optimised shape of the weights (no sharp edges and corners), a better surface quality and frequent recalibrations, an uncertainty of $1{\cdot}10^{-7}$ could be expected.

The relatively large uncertainty of the pressure distortion coefficient (λ) of the commercial 7 MPa p-c assembly, as given in Table 1, can be attributed to the simplified method of its determination (Lamé equations), unknown p-c gap dimensions, uncertain elastic constants of the p-c materials and the experimental nonlinearity of the effective area (A_p) with pressure, which is evidently caused by the operation of this p-c assembly in the re-entrant mode. As the results of the recently finished project EUROMET 463 show [6], λ can be determined with an uncertainty of u (λ) = $5{\cdot}10^{-8}$ MPa^{-1}, which is equivalent to $3.5{\cdot}10^{-7}$ for u (p) / p at 7 MPa if the elastic constants and the p-c gap dimensions are accurately measured and the finite element analysis (FEA) is applied for the elastic distortion calculation. To achieve a good linearity of $A_p(p)$, the p-c assembly should be operated in the free-deformation mode and the cylinder wall thickness should be increased compared to the commercial version in order to reduce elastic distortions.

Thus, the combined contribution of the first seven uncertainty sources in Table 1 to the pressure uncertainty can be reduced to $4{\cdot}10^{-7}$. The zero-pressure effective area of the p-c assembly (A_0) and the experimental standard deviation, type A uncertainty, are two properties crucial for the target uncertainty.

3. Zero-Pressure Effective Area

At PTB, two 10 cm^2 p-c assemblies Nos. 288 and 290, operated in the range (0.06 to 1) MPa, and a 5 cm^2 p-c assembly No. 6222, operated in the range (0.08 to 2) MPa, are applied as primary gas pressure standards in combination with a primary mercury manometer (HgM) [7]. Their A_0 is found from dimensional measurements performed on the cylinder bore and piston and has also been determined from measurements with the HgM as a reference. Fig. 1 shows the results for A_0, obtained by means of the two methods for units 288 and 6222, starting with the first characterisation in 1989. For both units, the results agree within their standard uncertainties. For assembly 288, all A_0 lie within $\pm 4.5{\cdot}10^{-6}$, the scatter is mainly produced by the measurements with the HgM and evidently reflects its stability. The dimensional values determined in 1989 [8] and 2004 differ relatively by only $2{\cdot}10^{-6}$. For unit 6222, all three independent A_0 values determined in 1995 agree within $\pm 5{\cdot}10^{-7}$. A new dimensional characterisation of this assembly has been performed recently whose evaluation is in progress.

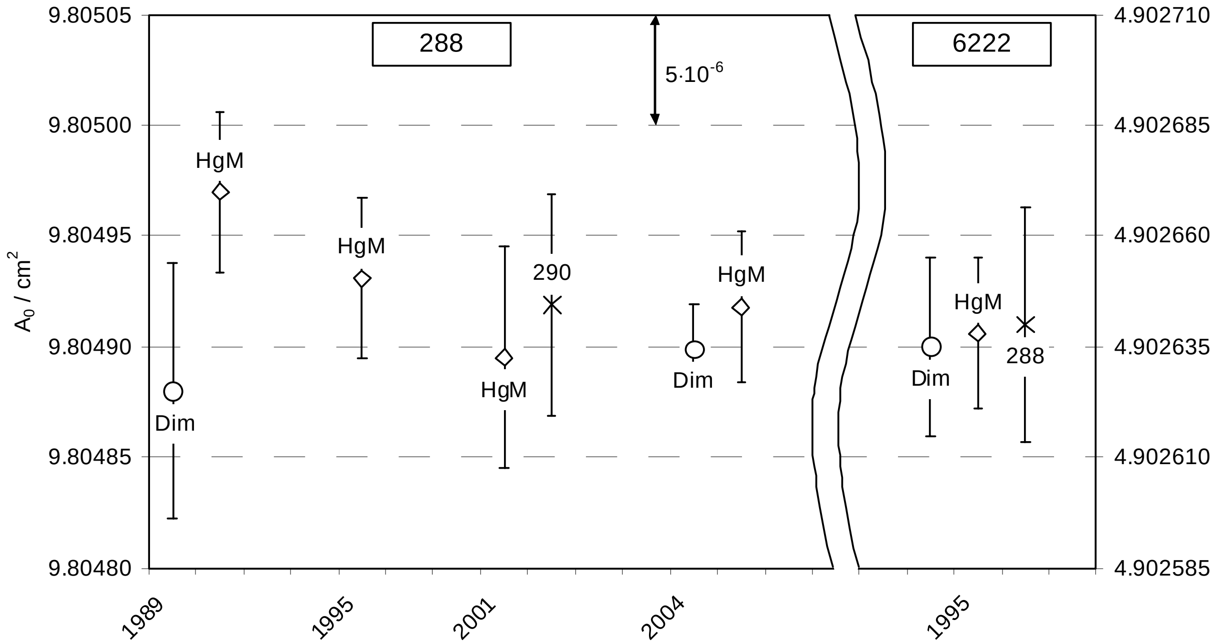

Fig. 1. Effective areas of the piston-cylinder assemblies 288 and 6222, determined in the period from 1989 to 2004 by dimensional measurements (Dim), comparison with the primary mercury manometer (HgM) and with another primary piston-cylinder assembly (290)

Consistency of the zero-pressure effective areas, determined at different pressures by means of different methods and standards, is demonstrated for assembly 6222 in Fig. 2. Here, two additional reference standards, 278 and 279, are oil-operated dimensionally characterised 5 cm^2 assemblies which are used as primary hydraulic pressure standards in the range up to 10 MPa [9]. The dimensional effective areas shown by two lines were calculated separately for dimensional data taken along the p-c generatrix and circle traces, assuming the piston and cylinder to be absolutely stiff. Experimental $A_0(p)$ were calculated from $A_p(p)$, using equation $A_0(p) = A_p(p) / (1+\lambda_p)$ and the λ value obtained by the Lamé equations. The results below 0.2 MPa have a higher scatter because the operation of unit 6222 below 0.2 MPa is not optimal. At higher pressures, consistency within $\pm 2{\cdot}10^{-6}$ is observed, except for just a few stronger deviating points which reflect the experimental difficulty of cross-floating oil- and gas-operated pressure balances with an oil-gas interface between them. The agreement of the results at pressures above 0.8 MPa is remarkable, as they were obtained with pressure balances of completely different design, different pressure-transmitting media and the assemblies' effective areas based on different theories of the flow (gas and liquid) in the p-c gap.

As follows from the results presented in Figs. 1 and 2, the target uncertainty $u(p)/p = 1{\cdot}10^{-6}$ is much closer than one might think in view of the results of CCM.P-K1.c. The same conclusion can be drawn from the results recently published in [10], in which the effective areas of two NIST primary p-c units, based on dimensional measurements performed at PTB, are compared with those determined from pressure measurements against the NIST primary ultrasonic interferometer manometer, demonstrating an agreement within $\pm 1.5{\cdot}10^{-6}$.

The effective areas of the PTB p-c assemblies were determined from p-c dimensional properties, using the theory by Dadson et al. [11] in which all forces produced by the pressure-transmitting medium on the piston are taken into account and in which its flow in the p-c gap is assumed to be viscous and laminar. This consideration leads to equation

$$A_0 = \pi r(0) R(0) + \frac{\pi r(0)}{p - p_{amb}} \int_0^l (p_z - p_{amb}) \frac{d(r+R)}{dz} dz \quad (2)$$

in which r(z) and R(z) are piston and cylinder bore radii along their engagement path of length l, p_{amb} is the ambient pressure and p_z is the pressure distribution in the p-c gap. If the pressure-transmitting

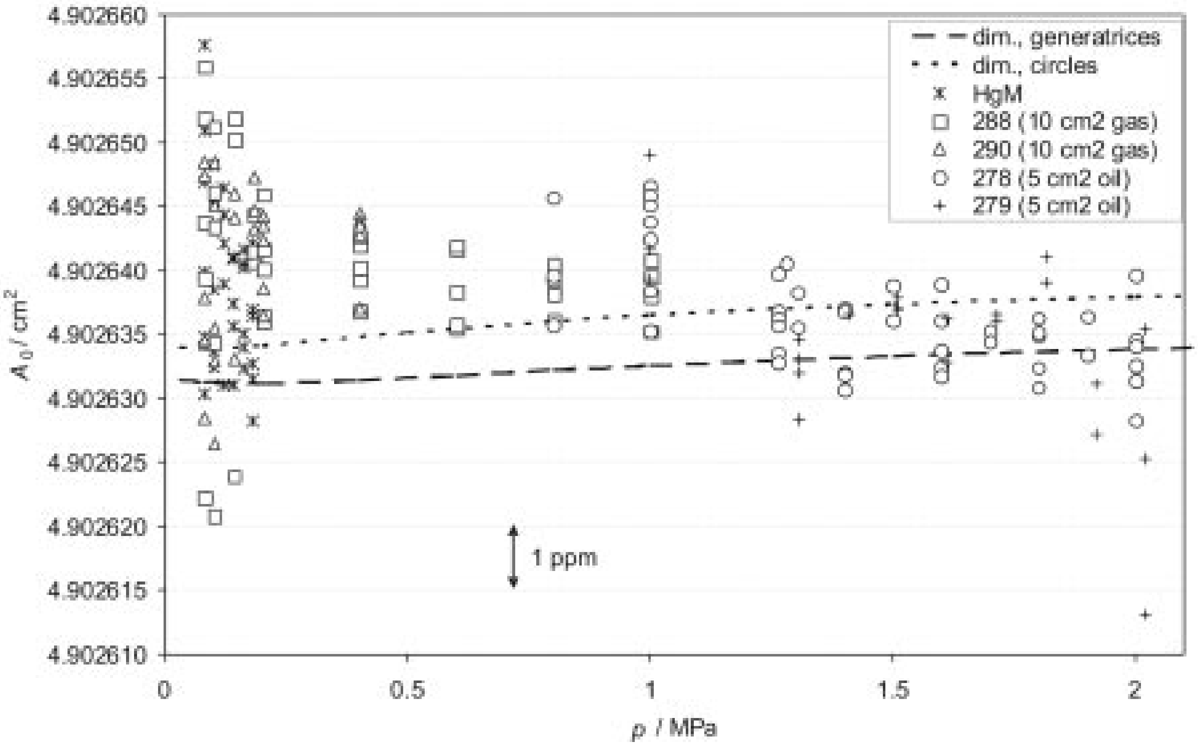

Fig. 2. Zero-pressure effective area of piston-cylinder assembly 6222, determined from dimensional data along generatrices and circles and from pressure measurements using the mercury manometer (HgM), two primary gas-operated 10 cm^2 units (288 and 290) and two primary oil-operated 5 cm^2 units (278 and 279) as a reference

medium is a gas which flows viscously and obeys the ideal gas law, the pressure distribution is defined by

$$p_z = \left\{ p^2 - (p^2 - p_{amb}^2) \int_0^z \frac{dz}{(R-r)^3} \Big/ \int_0^l \frac{dz}{(R-r)^3} \right\}^{1/2} \qquad (3)$$

The uncertainty of the effective area determined by equations (2) and (3) depends primarily on the uncertainty of r and R. The next important uncertainty contribution is usually the deviation of the real p-c geometry from the axial symmetry implied in Eqs.(2) and (3).

4. Dimensional Measurement Techniques and Data Evaluation

Dimensional measurements at PTB include the separate determination of diameters and shape deviations along selected generatrix and circle traces. The shape deviations are linked to the diameters to produce the radii required in Eqs. (2) and (3). Since the first dimensional characterisation of p-c assemblies in 1989 [8], different devices for measurements and different approaches for data evaluation have been used.

In 1989, a one-dimensional Abbe-comparator, equipped with a laser interferometer, was used for diameter (D) measurements and a universal measurement machine Moore No. 3 for roundness (R) and straightness (S) measurements. By means of those techniques, standard uncertainties u (D) =u (R) = u (S) = 50 nm were estimated.

In 1995, the diameter, roundness and straightness measurements were performed with the Abbe-comparator, an RTH Talyrond 73 device and the Moore No. 3, respectively, and the standard uncertainties u (D) = 50 nm, u (R) = 20 nm and u (S) = 30 nm were reported. A new approach to link the diameter and the shape measurement results in the way described in [12] was applied by which 3d-data having a standard uncertainty of radial values of 25 nm were generated.

Since 1997, a state-of-the-art comparator for

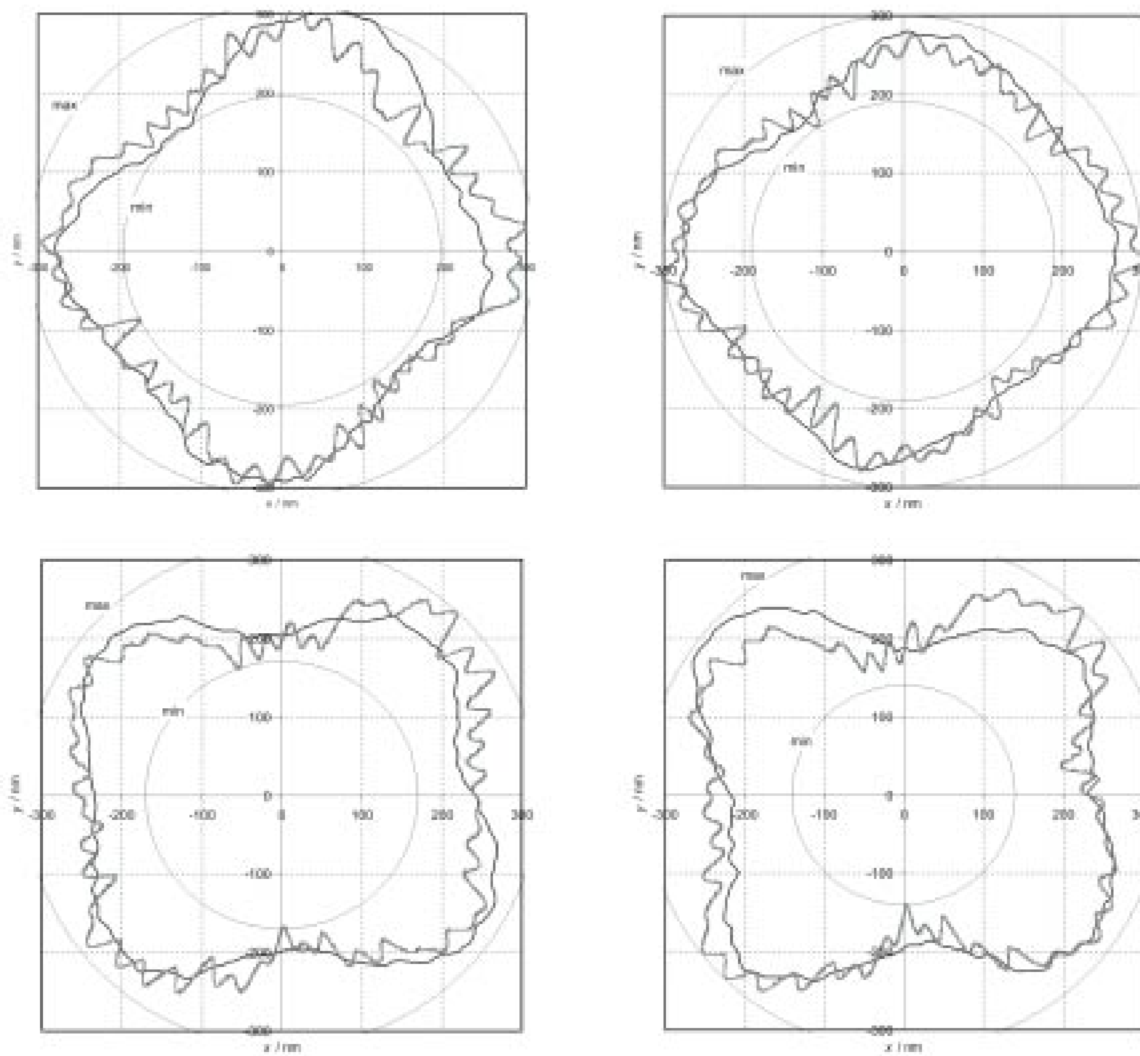

Fig. 3. Roundness deviations measured in 1989 and 2004 on the cylinder of unit 288 in the levels a) -15 mm (1989) and -14 mm (2004), b) -10 mm, c) +10 mm, d) 15 mm (1989) and 14 mm (2004), referring to the middle of the p-c engagement path

diametric measurements (KOMF) has been in service [13-14] which allows the diameters of the piston and of the cylinder bore to be measured with standard uncertainties of 5 nm and 10 nm, respectively.

Since 2000, an MFU-8 instrument has been applied which allows D, R and S to be determined with the uncertainties u (D) = 20 nm, u (R) = 30 nm and u (S) = 40 nm. Using this instrument has the advantage that all measurements are carried out in the same device, which ensures a better coordination of the three kinds of measurements.

The results of roundness measurements, performed in four levels on the cylinder of unit 288 in 1989 using the Moore No. 3 and in 2004 with the MFU-8, are shown in Fig. 3. Some differences in the shapes of the traces were due the different sizes of the contacting spheres used in the measurements as a probe (3.2 mm in 1989 and 8 mm in 2004), different low-pass filters for recorded signals (a limit wave number 512 per revolution and an attenuation 75% in 1989 and a limit wave number 150 per revolution and an attenuation 50% in 2004), and slightly deviating levels in which the measurements were performed (±15 mm in 1989 and ±14 mm in 2004). Apart from these

Table 2
Uncertainty budget for A_0, based on dimensional measurements as performed on the 10 cm² unit 288 in 2004 and as expected for a 20 cm² p-c unit characterised with the best dimensional measurement capabilities

P-c assembly	10 cm² (288), actual		20 cm² (new), optimised	
	$u(x_i)$ / nm	$u_i(A_0)/A_0$ x10⁶	$u(x_i)$ / nm	$u_i(A_0)/A_0$x10⁶
Diameter	20 (MFU-8)	1.2	10 (KOMF)	0.43
Roundness, straightness	50	1.5	25	0.53
$A_0(p)$, change with pressure		0.5		0.20
$A_0(\varphi)$, axial non-symmetry		0.7		0.49
Combined uncertainty		2.1		0.86

differences, the shapes agree within a few nanometers, which proves the good geometrical stability of the cylinder over a period of 15 years, and also a high reliability of the dimensional measurements even though they have been performed with different instruments. Also, two dimensional characterisations performed at PTB on the same NIST p-c assembly, using different measuring devices in 1999 and 2003, led to the effective areas differing by only $1.1{\cdot}10^{-6}$ [10].

In Table 2, the uncertainty budget for the last dimensional calibration of the 10 cm² unit 288, performed in 2004, is presented, along with the uncertainty budget expected for the case in which the unit has a nominal effective area of 20 cm² and the best available dimensional measurement capabilities are applied. The analysis shows that even without an improvement of the available dimensional measurement instruments, $u(A_0)/A_0 < 1{\cdot}10^{-6}$ will be possible if a larger p-c assembly is used. It must be denoted that, to claim such a small uncertainty, the analysis presented in Table 2 should be extended to include further potential uncertainty sources such as i) a limited amount of information on the topography of the piston and cylinder bore surfaces, ii) the possible non-coaxial position of the piston in the cylinder bore and iii) the behaviour of the gas in the p-c gap.

The first contribution is relatively easy to estimate by using different portions of the available dimensional information or by applying different interpolating procedures to create dimensional data in the regions not covered by the measurement.

The second effect can be quantified by repeated calculations when the piston position in the cylinder bore is varied. Here, 3-dimensional models can be useful to calculate A_0 of a non-coaxial p-c unit without a significant increase in the uncertainty which will normally take place if a 2-dimensional model is applied for different azimuthal sections of an axially non-symmetrical unit.

The third contribution may present the most serious problem dealing with the fact that, under certain conditions, the gas motion in the p-c gap can change from a macroscopic viscous flow to a molecular non-viscous flow, as outlined in [15-16]. Approaches for the A_0 calculation in the case of viscous, molecular and viscous-molecular transition flows are presented and the effect of the pressure, the operation mode and the gas species on A_0 discussed in [16-18]. Some experimental results on A_0 measured in gauge and absolute mode with different gases are reported, e.g., in [17,19-20] and were obtained at PTB. In some cases, significant differences between the experimental data as well as between experiment and theory are found, which show that there is a need for further theoretical and experimental investigations. An issue common to all theories is that the effect of the flow regime, the operation mode and the gas species is decreasing with increasing diameter of the p-c assembly.

5. Experiments

The last important uncertainty contribution listed in Table 1 is the type A uncertainty. To reduce this uncertainty, the performance of pressure balances should be improved and a cross-float technique, resulting in experimental standard deviations that are substantially lower than 1.10^{-6}, be applied. This technique should allow an accurate comparison of p-c assemblies with very different effective areas, in order to have a direct link between large primary p-c assemblies characterised dimensionally and significantly smaller ones by means of which a measurement of pressures up to 7 MPa can be carried out. Finally, a comparison of pressure balances operated with different gases would be desirable. The classical cross-float technique based on observation of the pistons fall rates evidently does not meet these requirements. Another technique, utilising differential pressure cells (DPCs) placed between two pressure balances and directly measuring the pressure difference (dp), appears to be applicable [21].

Three DPCs, LPX 900 Series with dp = ± 50 Pa by GE Sensing - Druck, Deltabar S PMD75 Series with dp = ± 1 kPa by Endress + Hauser and 3051S1CD Series with dp = ± 6.2 kPa by Rosemount, were investigated as possible candidates. Among others things, the investigation had to show whether the absolute pressure measurement in the 7 MPa range required for the Boltzmann constant experiments can be realised as a gauge pressure measurement with a simultaneous measurement of the ambient pressure. Although the dp ranges of the DPCs differed considerably, the results reported below were quite similar for all three DPCs.

The short-time zero-point stability was found to be 20 mPa, and the calibration curve obtained in the range dp = ± 50 Pa with an FRS-4 pressure balance by Furness Controls as a reference showed a reproducibility within 80 mPa.

With the DPCs installed between the two 10 cm^2 piston gauges 288 and 290 operated in gauge mode, the relative standard deviation of the effective areas measured in the range (0.1 to 1) MPa was equal to $7.6 \cdot 10^{-7}$. A similar performance could be observed even when p-c assemblies with extremely different A_0, e.g. 10 cm^2 against 8.4 mm^2, were compared. At individual pressures, the standard deviations of dp recorded by the DPCs varied typically between (140 and 620) mPa.

The integration time of the DPCs was chosen to be 1 s and allowed dp to be recorded between the cross-floated pressure balances. With the piston gauges 288 and 290, dp was found to scatter within ±600 mPa, which agrees with the standard deviation of the effective area determined from the experiment. Such a high pressure instability compared with the performance of the DPCs (80 mPa), clearly shows that this instability is produced by the pressure balances. A possible reason for this can be disturbances produced by changes in the ambient pressure, local air flows and counteraction of the rotating weights with the surrounding air when the pressure balances are operated in the gauge mode. In fact, as has recently been shown by measurements performed at the NMIJ [22] and demonstrated by a recent bilateral comparison between NMIJ and MSL (New Zealand), APMP.M.P-K5 [23], DPCs can be calibrated with much lower standard deviations of about (8 to 14) mPa at dp = 1 kPa and a line pressure of 100 kPa when the pressure balances are operated in absolute mode. This experience confirms that cross-float measurements with absolute pressure balances can be performed with a relative standard deviation smaller than $2 \cdot 10^{-7}$.

6. Conclusions

The relative standard uncertainty of the absolute pressure 7 MPa, measured by means of a pressure balance, can, as a matter of principle, be as low as $1 \cdot 10^{-6}$. A_0 and λ of the piston-cylinder assembly, as well as the performance of the pressure balance are the main uncertainty sources. The use of large p-c assemblies, an improvement of the dimensional measurement techniques and the application of advanced theories for the A_0 calculation should allow its determination with $u(A_0)/A_0 < 1 \cdot 10^{-6}$. The contribution of λ can become sufficiently small when accurately measuring elastic constants of p-c materials, taking into account dimensional properties of p-c gap and applying FEA for the calculation of p-c elastic distortions. A reduction of the experimental standard deviation can be achieved by carrying out measurements in absolute mode and applying DPCs. Two absolute pressure balances of special design, each one equipped with a mass set of 150 kg, an automatic loading system, three p-c assemblies of 20 cm^2 and three p-c units of 2 cm^2 will be built. Extensive experiments will be carried out in absolute and in gauge mode and with different gases, in order to demonstrate consistency within the group of the six p-c units.

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