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Development and characterisation of a low pressure transfer standard in the range 1 Pa to 10 kPa

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ABSTRACT
We describe a transfer standard for low absolute and gauge pressure in the range 1 Pa to 10 kPa. This transfer standard is composed of three differential capacitance diaphragm gauges (CDGs) of full-scale 130 Pa, 1.3 kPa and 13 kPa respectively and one absolute 130 kPa resonant silicon gauge (RSG). The objective for the relative uncertainty contribution (k=1) of this standard during a comparison is a few tens of ppm at 10 kPa to a few hundred ppm at 1 Pa. It relies on a good long-term stability of the calibration slope of the RSG used, between 5 kPa and 10 kPa, disseminated to CDGs in absolute mode and subsequently in gauge mode. The methods to assess such uncertainty and the preliminary characterization of the transfer standard are presented.

Section: RESEARCH PAPER

Keywords: pressure; vacuum; transfer standard; resonant silicon gauge; capacitance diaphragm gauge

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1. INTRODUCTION

We have developed a low pressure transfer standard in the pressure range from 1 Pa to 10 kPa in both absolute and gauge modes, in the frame of the EMPIR project 14IND06 “Industrial standards in the intermediate pressure-to-vacuum range”. The objective for this transfer standard is to get an uncertainty contribution (k = 1) in relative value, of the order of 1×10^{-4} so as to use this standard in comparisons between calibration services on a national level.

For pressures lower than 1 kPa, capacitance diaphragm gauges (CDGs) are usually employed as transfer instruments, but they suffer from a relative mean-term instability of a few 10^{-4} which can dramatically increase after transportation. In the upper pressure range, between 1 kPa and 10 kPa, the best quartz reference pressure transducers (Q-RPT) or resonant silicon gauges (RSG) we have used provide a stability of 0.5 Pa over their whole range, too high to meet our objective.

In the recent key comparison CCM.P-K4.2012, in the same range [1], the pilot laboratory has developed a transfer standard based upon a CDG 100 Pa and a special 10 kPa RSG. Over the course of the comparison, this latter, with a resolution of 0.01 Pa, showed a stability between a few ppm at 10 kPa to about 1×10^{-4} at 100 Pa, allowing one to rescale the CDG which could consequently provide a quite low uncertainty contribution as a transfer standard between 1 and 100 Pa.

This method is also commonly used for very low pressure in the vacuum range, where the pressure given by an ionisation gauge is normalised by a comparison measurement with an associated spinning rotor gauge [2]. In the work described in this paper the rescaling procedure is applied in a slightly different way. We have used a 130 kPa RSG which is poorly stable if we observe its calibration history for a single pressure point. However, the used instrument has a good long-term stability of the correction slope and a nice linearity between 5 and 10 kPa. By performing, in absolute mode, a slope comparison in this range with a CDG 13 kPa full-scale, it is then possible to rescale the CDG signal which in turn is used to normalise a CDG 1.3 kPa full-scale, between 500 Pa and 1kPa.

The latter finally allows one to rescale a CDG 130 Pa full-scale, between 50 Pa and 100 Pa. Rescaled CDGs can afterwards be applied in the relative mode.

The paper describes first the preliminary observations we have made about the metrological performances of our pressure standards. Thereafter, the method for rescaling the CDGs is presented and the experimental setup is detailed. From the performance of the gauges and the characterisation of the transfer standard, the uncertainty is assessed.

2. METROLOGICAL PERFORMANCES OF THE STANDARD PRESSURE GAUGES

2.1. Resonant silicon gauge 130 kPa

A resonant silicon gauge (Druck type DPI142[1]) was acquired for the daily calibrations in the pressure range 10 to 130 kPa, in the vacuum department. From the successive calibrations of the RSG with a pressure balance, this RSG was found to drift mainly in offset (Figure 1).

The Figure 2 shows the scattered drift of the correction slope (determined by means of a simple linear least squares line). The drift is estimated to be (-1.0 ± 6.6) ppm per year.

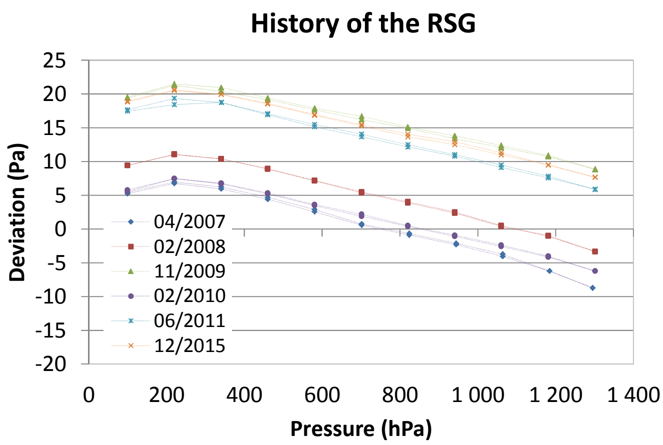

Figure 1. Several RSG calibrations by means of a pressure balance.

It highlights the stability of the correction slope over time.

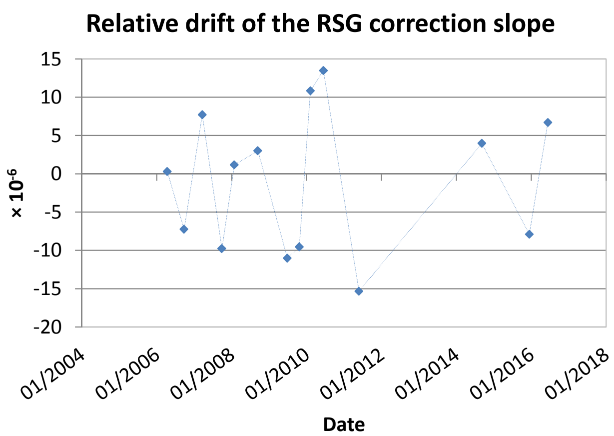

Figure 2. Relative drift in the slope correction coefficient of the RSG between the current calibration and the previous one.

As the nominal range of the sensor is 3.5 to 130 kPa, we then decided to characterise it also in the range 5 to 10 kPa. The calibration with the force-balanced piston gauge (Fluke FPG 8601) of the LNE-LCM[2] has shown a nice linearity of the RSG in this range (Figure 3). So far, only three calibrations were performed in this range. The maximum drift for the correction slope was found equal to 14.5 ppm, compatible with observations of Figure 2. The RSG can then be applied to check and possibly rescale the calibration function of our working standard CDG 10 kPa used to calibrate customer's gauges.

2.2. Capacitance diaphragm gauges

Relative and absolute capacitance diaphragm gauges from the manufacturer MKS (full scale 130 Pa, 1.3 kPa and 13 kPa) are currently used as secondary and working standards at LNE-LCM. The use of the analogue output U (0-10 V), rather than the digital one, is generally preferred to enhance the gauge resolution. Thus the calibration function is expressed with an equation of the form:

$$p = f(U - U_0), \qquad (1)$$

where U_0 is the output signal of the gauge when a zero pressure is applied (which in absolute mode corresponds to a pressure lower than one tenth of the gauge resolution) and f is a fourth degree (at the maximum) polynomial function, to which the Takaishi-Sensui thermal transpiration correction [3] is applied to calculate the reference pressure p. In absolute mode, the polynomial function and the gauge temperature are determined from a calibration by means of the FPG8601, between 1 Pa up to 13 kPa. We fit the calibration data corrected with the thermal transpiration correction in which we assign a temperature to the gauge by successive approximations. To get a better estimate of this temperature, fifteen calibration points are performed below 50 Pa, where the thermal transpiration correction is significant.

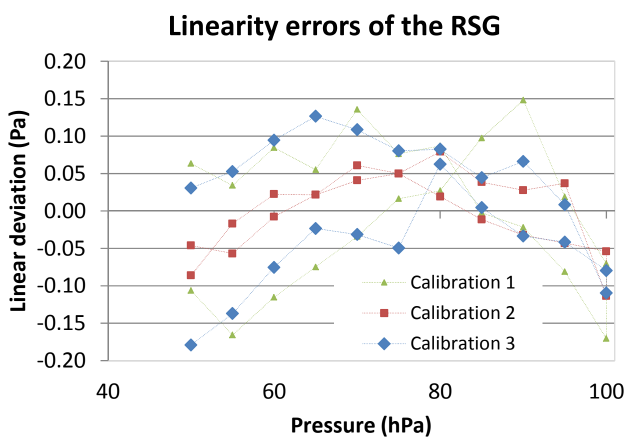

Figure 3. Linearity deviation from a regression line of the RSG, stated from three successive calibrations.

Each calibration consists of three runs performed by increasing and decreasing pressure steps: linearity deviations also include hysteresis effects.

[1] Identification of commercially available instruments in this paper does not imply recommendation or endorsement.

[2] Sub-division of LNE dealing with primary metrology in mass, pressure, temperature, radiometry and spectrophotometry, and dimensional metrology.

From the numerous performed calibrations of CDGs, it was stated that, on the mean term, the shape of a calibration curve does not change much, as one can see at a glance in Figure 4; consequently, it is possible to estimate the new calibration function f_t by a linear correction of the former one, f_{t-1}. Let us denote by f_t' the function determined by calculation. f_t and f_{t-1} are the calibration functions obtained from the CDG successive calibrations. We have:

$$f_t'(U-U_0) = k_{CDG} \times f_{t-1}(U-U_0). \quad (2)$$

The correction factor k_{CDG} is the slope coefficient of the least squares line that is estimating the reference pressure as a function of $f_{t-1}(U-U_0)$, in the range between 40 % and 80 % of the full scale of the CDG[3]. This is the method used to rescale a CDG. From the example of Figure 4, with a quite large drift of deviation of the CDG (of about 1.2×10^{-3} in relative value), the aforementioned method was applied and the difference $(f_t' - f_t)$ is plotted in Figure 5 as a function of the pressure. On this same graph, the residuals of the CDG calibration curve, *i.e.* the difference between $f_t(U-U_0)$ and p_{FPG} the reference pressure given by the standard FPG8601, are plotted. As one can see in Figure 5, the residuals and the deviation between the rescaled pressure and the modelled pressure are of the same order of magnitude and lower than 4.0×10^{-5} in relative value.

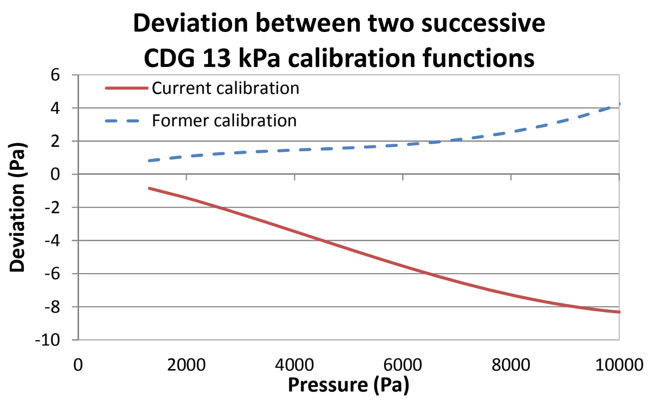

Figure 4. Plot of the deviation of two calibration functions of a CDG 13 kPa full scale, in absolute pressure mode. The deviation is the difference between the CDG calibration function and a linear function $g(U-U_0) = a \cdot (U-U_0)$, where a is an arbitrary coefficient.

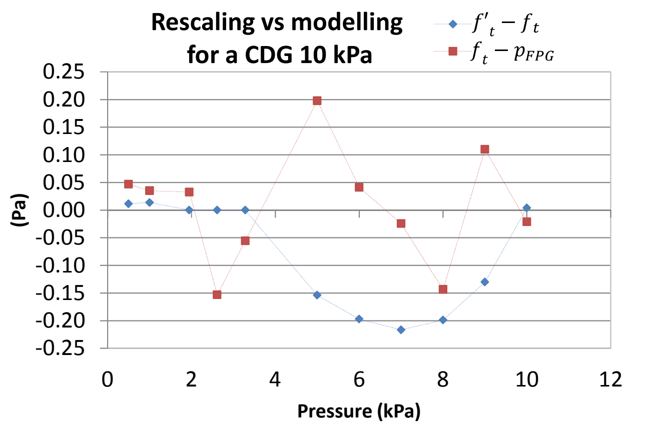

Figure 5. Difference between the calibration function of a CDG 10kPa obtained in one case by modelling the calibration data (f_t), and in the other case by applying a correction factor k on the former calibration function $(k \times f_{t-1})$. k is the slope coefficient of the least squares line that is estimating the reference pressure p_{FPG} as a function of the CDG pressure modelled with the function (f_{t-1}), in the range between 40 % and 80 % of the full scale of the CDG. This difference is plotted together with the residuals of the model: $(f_t - p_{FPG})$.

[3] A large part of the CDGs used at LNE-LCM exhibits a significant non linearity between 80% and 100% of the full scale, and is not used in this range.

3. EXPERIMENTAL SETUP

The metrological features of the instruments, described in § 2, make possible the rescaling of three CDGs of respective full scale 13 kPa, 1.3 kPa and 130 Pa, starting with a calibration of the CDG 13 kPa with the RSG between 5 kPa and 10 kPa. When rescaled, the CDG 13 kPa is used to rescale the CDG 1.3 kPa and applying the same method the CDG 130 Pa is rescaled. The experimental setup of the transfer standard is described in Figure 6.

The CDGs from the manufacturer MKS Instruments are the relative pressure transducers 698. They are used as absolute CDGs by means of an ion pump IP which maintains a stable vacuum on their reference port. Each CDG is connected to a 3-channel multiplexer 274, which allows to thermostat the transducers around 45 °C, itself connected to the electronics 670. The analog (0-10 V) pressure reading is made *via* a digital multimeter Agilent type 34401 linked to the 670. The RSG is the Druck DPI142 silicon gauge, isolated with the valve VRSG as long as the calibrated pressure is lower than 5 kPa.

Figure 7 shows a general view of the transfer standard with the plate where the transducers, vessels and ion pump, the module with displays, pump controller and the transportation box are placed.

4. PROCEDURE TO USE THE TRANSFER STANDARD

To obtain the lowest uncertainty contribution of the transfer standard, it is necessary to rescale the CDGs at each calibration cycle, in absolute pressure mode. In others words, in addition to the calibration pressure points of the comparison protocol, some common measurements have to be performed between

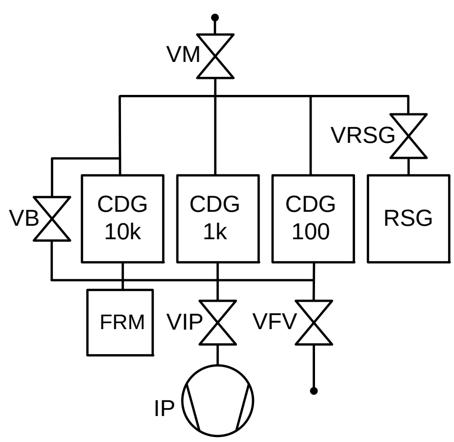

Figure 6. Setup for the transfer standard.

CDG100, CDG1k, CDG10k capacitance diaphragm gauges MKS type 698 of respective full range 130 Pa, 1.3 kPa and 13 kPa; RSG: resonant silicon gauge Druck type DPI142 (3.5-130 kPa); IP: ion pump; FRM: combined Pirani-Penning manometer; VM: isolation valve of the transfer standard; VRSG: isolation valve of the RSG VB: bypass valve; VIP: isolation valve of the ion pump; VFV: Valve used to connect a fore vacuum pump with ultimate pressure suitable for the ion pump.

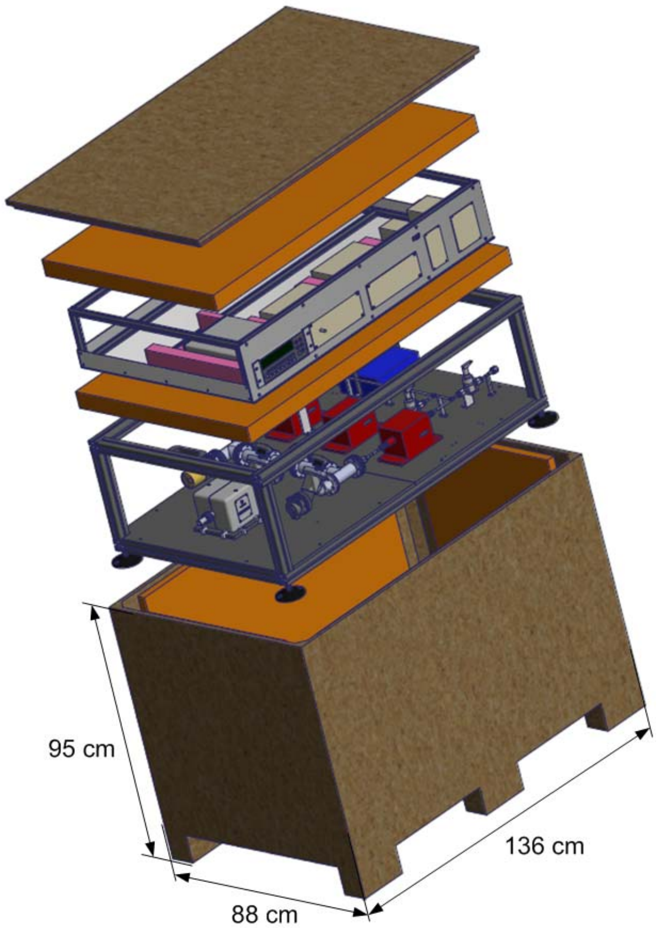

Figure 7. General view of the transfer standard with its transportation box.

each couple of gauges: RSG-CDG10k, CDG10k-CDG1k, CDG1k-CDG100, to rescale each CDG according to the method described in § 2.2. The common measurements points are defined in Table 1.

The initial calibration functions of the different manometers are f_{RSG}, f_{10k}, f_{1k} and f_{100} for RSG, CDG10k, CDG1k and CDG100 respectively. f_{RSG} applies on the reading in pressure value p_{RSG} of the RSG, as the other functions apply on the analog output U of the CDGs corrected with the corresponding zero value U_0 (see § 2.2).

When the transfer standard is used, the actual calibration functions of CDGs, f'_{10k}, f'_{1k} and f'_{100} are determined after the post-processing of the measurements data common to different gauges. $f_{10k}(U - U_0)$ is plotted as a function of $f_{RSG}(p_{RSG})$ for the four corresponding pressure levels of Table 1 and the linear rescaling coefficient for the CDG10k k_{10k} is determined by means of a least squares regression. The linear rescaling coefficient for the CDG1k, k_{1k}, is determined in a similar way by plotting $f_{1k}(U_{1k} - U_{1k;0})$ as a function of $f'_{10k}(U_{10k} - U_{10k;0})$ and finally the linear rescaling coefficient for the CDG100, k_{100}, is determined from the plotting $f_{100}(U_{100} - U_{100;0})$ as a function of $f'_{1k}(U_{1k} - U_{1k;0})$. It is implied that the thermal transpiration correction is applied to each CDG signal.

Table 1. Additional calibration points during a comparison, used to rescale the CDGs.

	CDG1k	CDG10k	RSG
CDG100	50 Pa, 70 Pa, 90 Pa, 100 Pa		
CDG1k		500 Pa, 700 Pa, 900 Pa, 1 kPa	
CDG10k			5 kPa, 7 kPa, 9 kPa, 10 kPa

As the CDGs are rescaled from the dissemination of the stable correction in slope of the RSG, this procedure allows the correction of the drift of the transfer standard for each participant in the comparison, who performs a calibration in absolute pressure between 1 Pa and 10 kPa. Once the calibration of the transfer standard in absolute mode has been performed, one can use it in gauge mode (the reference ports of CDGs are put under atmospheric pressure). It will be then assumed that the mean rescaling coefficient of each CDG determined in absolute mode is available, with a supplementary source of uncertainty to be taken into account for gauge mode (§ 6).

5. CHARACTERISATION OF THE TRANSFER STANDARD

5.1. Rescaling of CDGs in absolute mode

The transfer standard was connected to a vacuum chamber and some calibration points were performed by increasing pressure levels (including the additional points of Table 1), after the zero of each CDG has been recorded. Five calibration cycles were achieved. The data were processed in order to determine at each calibration cycle the rescaling coefficients k_{10k}, k_{1k} and k_{100} (§ 4). The corresponding experimental standard deviation of the linear regressions ESD_{10k}, ESD_{1k} and ESD_{100} are shown together with the coefficients in Table 2. Except for the first value of ESD_{1k}, most of the standard deviations lie below 5×10^{-5}.

Relative drifts in the successive coefficients (compared with the previous determined one) are plotted in Figure 8, in which the uncertainty bars denote single standard deviations. CDG1k exhibits a poorer stability in between cycles, up to 3×10^{-4}, compared with the other CDGs. This confirms the necessity to perform the additional calibration points at each cycle to keep the relative standard uncertainty below the objective of 1×10^{-4}.

5.2. Temperature coefficient of the RSG

As the overall performance of the transfer standard is based

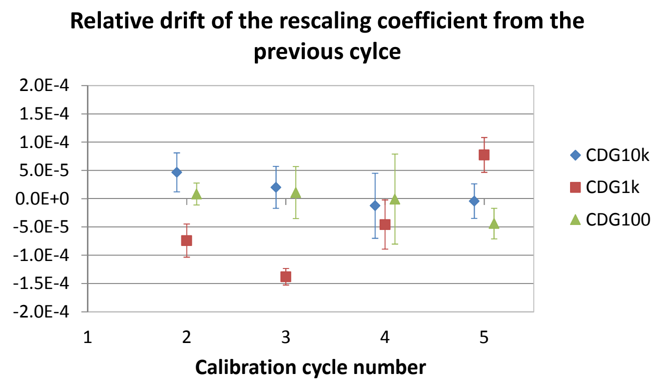

Figure 8. Relative drifts of CDGs correction coefficients determined by means of the rescaling, at each cycle. The whole calibration was performed over five days.

Uncertainty bars correspond to the standard deviation of the coefficient.

Table 2. Rescaling coefficients of the CDGs and corresponding standard deviations.

	k_{10k}	ESD_{10k}	k_{1k}	ESD_{1k}	k_{100}	ESD_{100}
Cycle 1	0.999 607	1.3×10^{-5}	0.999 901	1.5×10^{-4}	0.999 719	1.7×10^{-5}
Cycle 2	0.999 654	3.4×10^{-5}	0.999 827	2.9×10^{-5}	0.999 727	1.9×10^{-5}
Cycle 3	0.999 674	3.7×10^{-5}	0.999 689	1.5×10^{-5}	0.999 738	4.6×10^{-5}
Cycle 4	0.999 661	5.7×10^{-5}	0.999 643	4.3×10^{-5}	0.999 737	7.9×10^{-5}
Cycle 5	0.999 657	3.0×10^{-5}	0.999 720	3.1×10^{-5}	0.999 693	2.7×10^{-5}

on the correction slope of the RSG, it is important to check to what extent it is affected by temperature. Furthermore, an intercomparison is planned, in the frame of the project EMPIR 14IND06, between four institutes which have different reference temperatures for calibrations (20 °C or 23 °C).

To determine the temperature coefficient, the RSG was placed in a climatic chamber successively at 20 °C, 15 °C, 25 °C and back to 20 °C and was compared with a similar calibrated RSG which was left at the ambient temperature of 20 °C. The variation in the correction slope of the transfer standard RSG was studied as a function of temperature. The temperature coefficient was determined to be $(-5.5\times10^{-7} \pm 3.8\times10^{-7})$ K^{-1}. For a difference of only 3 K, the temperature effect never exceeds 2×10^{-6} in relative value and so can be neglected.

5.3. CDGs in gauge mode

In gauge mode, each CDG has its calibration function determined from a calibration with FPG8601. It is assumed that the rescaling coefficients established in absolute mode, also apply in gauge mode, provided the CDGs are calibrated exactly at the same time in both modes. In practise, this means CDGs have to be consequently calibrated in gauge and absolute modes within a short period of time (two weeks).

6. UNCERTAINTY BUDGET

An uncertainty budget of the contribution of the transfer standard is established from the metrological features of the RSG and CDGs (§ 2) and the characterisation of the standard (§ 5). Table 3 presents this budget for the absolute mode. The uncertainty $u_{10k}(p)$ of the CDG10k depends on the uncertainty of the correction slope of the RSG (calibrated with the FPG8601), its drift over time (Figure 2) and the linearity error (Figure 3). The uncertainty of the rescaling coefficient was estimated from the standard deviation ESD_{10k} to be roughly $5\times10^{-5}\times p$ and it is assumed that it also includes the linearity error of the RSG. According to Figure 5, rescaling errors and modelling errors of the CDG10k are of the same order of magnitude. In the uncertainty budget, only the latter are taken into account and added to the combined uncertainty ($2\times u_{10k}$). Uncertainty for CDG1k, $u_{1k}(p)$, is based on the uncertainty of $u_{10k}(p)$ (without taking into account the modelling errors of CDG10k) and the uncertainty of the rescale coefficient σ_{1k}, also estimated to be roughly $5\times10^{-5}\times p$; the modelling errors are added linearly to the calculated combined uncertainty ($2\times u_{1k}$). The uncertainty for CDG100, $u_{1k}(p)$, is based on the uncertainty of $u_{1k}(p)$ (without taking into account the modelling errors of CDG1k), the uncertainty of the rescale coefficient ESD_{100}, also estimated to be roughly $5\times10^{-5}\times p$ and the uncertainty from the ambient temperature disseminated by the thermal transpiration function (an uncertainty of 1 K associated to a rectangular distribution is considered); the modelling errors are then added linearly to the calculated combined uncertainty ($2\times u_{100}$).

The relative standard uncertainty of the transfer standard is finally plotted Figure 9. As one can see on the graph, it lies between 1×10^{-4} and 5×10^{-5} in the range from 100 Pa to 10 kPa and rises up to 3×10^{-3} when the pressure falls to 1 Pa.

For uncertainty assessment in gauge pressure, we have considered an additional contribution due to the short-term

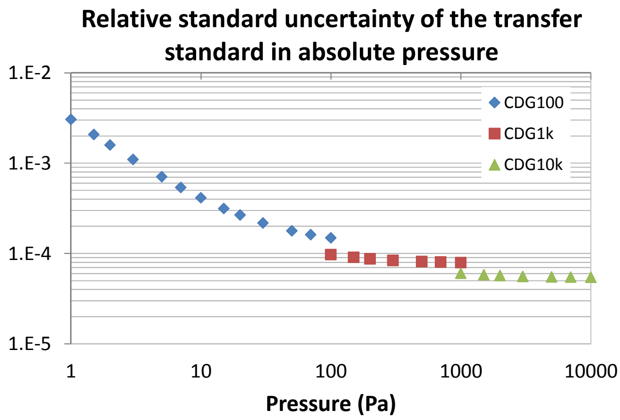

Figure 9. Relative uncertainty (k=1) of the transfer standard as a function of absolute pressure.

Table 3. Uncertainty budget of the transfer standard in absolute pressure.

Uncertainty component	CDG10k	CDG1k	CDG100
Calibration	$1.5\times10^{-5}\times p$ (FPG8601)	$5.3\times10^{-5}\times p$ (u_{10k})	$7.3\times10^{-5}\times p$ (u_{1k})
RSG slope stability	$6.6\times10^{-6}\times p$	-	-
Rescale coefficient	$5.0\times10^{-5}\times p$	$5.0\times10^{-5}\times p$	$5.0\times10^{-5}\times p$
Ambient temperature at ± 1K (thermal transpiration effect)	Negligible	Negligible	$8.1\times10^{-5}\times p$ + 0.0017 Pa
Combined uncertainty	$u_{10k} = 5.3\times10^{-5}\times p$	$u_{1k} = 7.3\times10^{-5}\times p$	$u_{1k} = 1.2\times10^{-4}\times p$ + 0.0017 Pa
Modelling errors	$2.4\times10^{-6}\times p$ + 0.013 Pa	$9.9\times10^{-6}\times p$ + 0.0039 Pa	0.0024 Pa
Expanded uncertainty (k = 2)	$U_{10k} = 1.1\times10^{-4}\times p$ + 0.013 Pa	$U_{1k} = 1.5\times10^{-4}\times p$ + 0.0039 Pa	$U_{100} = 2.4\times10^{-4}\times p$ + 0.0059 Pa

drift of CDGs from the characterisation in absolute mode (Figure 8), as the CDGs cannot be rescaled at each calibration cycle. This contribution was estimated to be $1 \times 10^{-4} \times p$. The expanded uncertainty (k = 2), given in Table 4, is about twice as large as that in absolute pressure, except in the range 1 to 100 Pa where CDG100 is not affected by the thermal transpiration effect. The uncertainty contributions of the transfer standard in absolute and gauge pressure are plotted together on the graph of Figure 10.

7. CONCLUSION

A pressure transfer standard between 1 Pa and 10 kPa has been characterised in both absolute and gauge modes. It is based on the stability of the linear correction slope of a resonant silicon gauge (RSG) in the range 5 to 10 kPa, in absolute mode. With a stepwise procedure, this allows one to rescale three capacitance diaphragm gauges (CDGs) of respective full scale 13 kPa, 1.3 kPa and 130 Pa, provided the transducers are compared pairwise at four pressure points. The uncertainty contribution of the transfer standard was estimated to be lower than 1×10^{-4} in the range 100 Pa to 10 kPa, which is at least ten times better than the usual performance of a capacitance diaphragm gauge; it rises to 3×10^{-3} when the pressure falls to 1 Pa, due to the thermal transpiration effect on the CDG 130 Pa.

When used in gauge mode, shortly after the rescaling procedure, the relative uncertainty contribution is about twice higher than that in absolute pressure between 100 Pa and 10 kPa, but slightly better in the range 1 Pa to 100 Pa, where the thermal transpiration effect does not apply.

A comparison between four Laboratories will be held in 2017, in the frame of the EMPIR project 14IND06 with this new transfer standard.

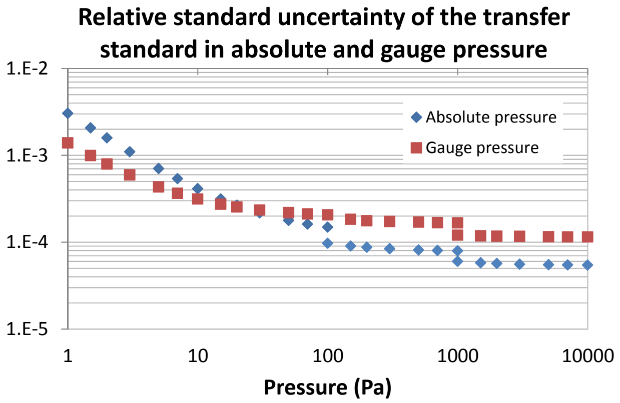

Figure 10. Relative uncertainty (k=1) of the transfer standard as a function of pressure in both absolute and gauge modes.

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Table 4. Uncertainty budget of the transfer standard in gauge pressure.

	CDG10k	CDG1k	CDG100
Expanded uncertainty (k = 2)	$U_{10k} = 2.3 \times 10^{-4} \times p + 0.013$ Pa	$U_{1k} = 3.3 \times 10^{-4} \times p + 0.039$ Pa	$U_{100} = 3.9 \times 10^{-4} \times p + 0.0024$ Pa