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A high-accuracy working standard for absolute pressure from 5 kPa to 130 kPa

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Abstract. We describe a calibration device for absolute pressure ranging from 5 kPa to 130 kPa whose relative uncertainty contribution ($k = 1$), including stability, repeatability and linearity is better than 1 Pa. The device is composed of a capacitance diaphragm gauge (CDG) and a resonant silicon gauge (RSG). The good long-term stability of the calibration slope of the RSG disseminated to the CDG which in turn allows one to master the offset of the RSG, is the reason for such a low uncertainty contribution. The metrological characterisation of this working standard is presented.

Keywords: absolute pressure / calibration / working standard / capacitance diaphragm gauge / resonant silicon gauge

1 Introduction

Mastering absolute pressure from around 100 kPa down to values as low as 5 kPa is important in various areas such as aeronautics and clean-room technology for semi-conductor manufacture and pharmaceuticals. It is then crucial for calibration services to provide pressure calibrations with low uncertainties in this range. The EMPIR project 14IND06 is aimed at developing methods in the intermediate pressure-to-vacuum range, especially in absolute pressure mode.

In previous work [1], we presented a resonant silicon gauge (RSG), full scale 130 kPa that exhibits a good long-term stability in its calibration slope and which was used to rescale a capacitance diaphragm gauge (CDG) of full scale 13 kPa, and stepwise two other CDGs of full scale 1 kPa and 100 Pa full scale respectively. In the present study, we focus on the RSG and the CDG 10 kPa. The latter, once rescaled by means of the RSG, is used to master and correct the drift in the offset of the RSG. The rescaling operation is a current way to enhance the uncertainty of a transfer standard, by estimating its drift, during the course of some metrological comparisons between national metrology institutes [2,3]. As an example, in the key comparison CCM.P-K4.2012 [3], the calibration procedure requires one compare at a pressure of 10 kPa, the measurement of a CDG of 10 kPa full scale and that of a RSG 100 kPa full scale. Since the particular RSG 100 kPa used in this comparison has a much better stability than the CDG 10 kPa, their direct comparison at 10 kPa allows one to calculate a correction coefficient which is applied to the output signal of the CDG 10 kPa in the lowest pressure range (not covered by the RSG 100 kPa): this is called the rescale procedure. In the work described in this paper, the rescale procedure is slightly different: as our RSG is poorly stable for a single pressure point but highly stable however as far as its calibration slope is concerned, several comparison points are needed to calculate the correction coefficient for the CDG. The metrological characterisation of both of these commercially available instruments and the associated procedures for their use and operation as working standards in the range 5–130 kPa with a standard uncertainty of 1 Pa is considered in this paper. For comparison, we have plotted in Figure 1 the best capabilities of French accredited calibration services in the aforementioned range [4], where the standard used to achieve this capability is given: a piston-cylinder device (which can be either a pressure balance or a digital piston gauge, or a liquid column standard) or a digital manometer.

Once calibrated and used correctly, this working standard could offer a performance equivalent to that of the best devices shown in Figure 1.

The paper describes first the preliminary observations we have made of the metrological performances of our absolute secondary pressure standards. Thereafter, the method for rescaling the 10 kPa full scale CDG, which in turns allows one to master the offset of the RSG, is presented. From the performance of the gauges and their characterisation, the contribution in uncertainty of the working standard is assessed.

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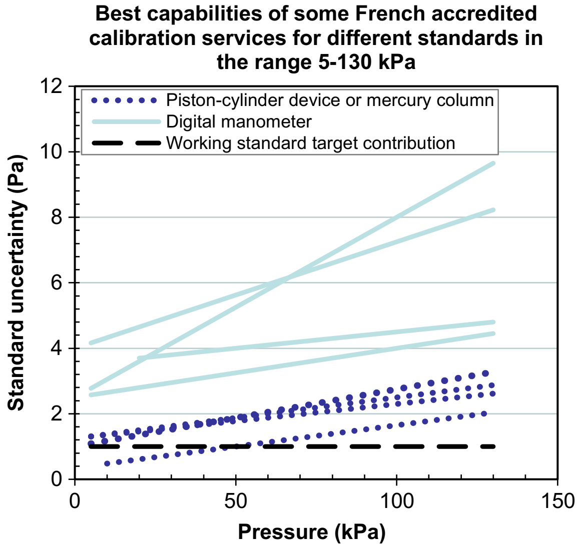

Fig. 1. Uncertainty ($k = 1$) of some French accredited calibration services. The uncertainty is achieved by means of either a piston/cylinder or liquid column device (dotted lines) or a digital manometer (solid lines). The maximum uncertainty contribution of the working standard in this study is represented with the dashed line.

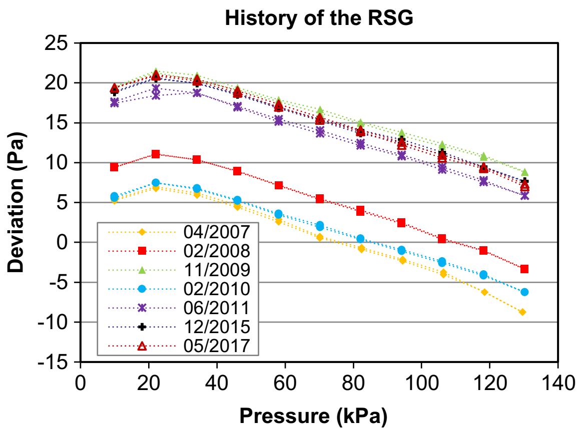

Fig. 2. Several calibrations of the upper range of the RSG by means of an absolute pressure balance. One can see at a glance, in the upper pressure range (30–130 kPa), that the RSG signal is linear and the slope of an applied straight correction line would be stable over time.

2 Metrological performances of the standard pressure gauges

2.1 Resonant silicon gauge of full scale 130 kPa

Ten or so years ago, a RSG (Druck type DPI142[1]) was acquired by the vacuum department of LNE-LCM for daily calibrations in the pressure range 10–130 kPa. From the successive calibrations of the RSG with an absolute pressure balance, the main drift of the RSG characteristic was found to be that of the offset, the slope remaining stable (Fig. 2).

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

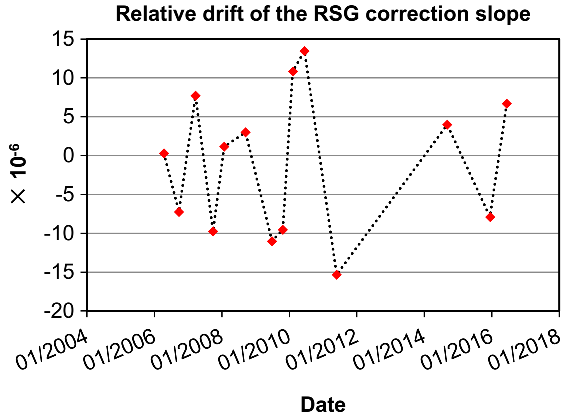

Fig. 3. Relative drift in the slope correction coefficient of the RSG between a current calibration and the previous one.

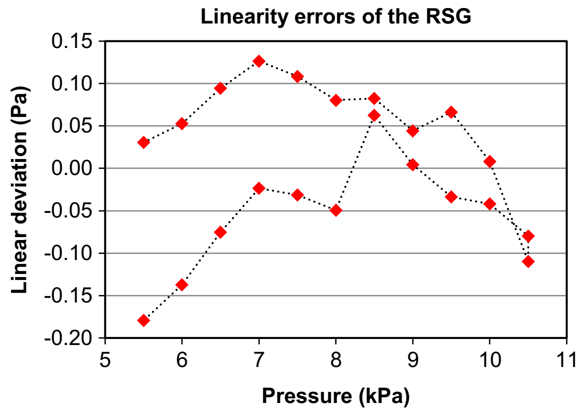

Fig. 4. Linearity deviation from a regression line of the lower range of the RSG.

Figure 3 shows the scattered drift of the correction slope, determined by means of an unweighted simple least-squares fitted straight line. The drift is estimated to be (-1.0 ± 6.6) ppm per year.

As one can see from Figure 1, the main drawback of this RSG is the lack of linearity between 10 kPa and 35 kPa which leads to a modelling error of about 2.5 Pa. We shall see in Section 4 how this issue can be dealt with (Fig. 4).

As the nominal range of the sensor is 3.5–130 kPa, it appeared appealing to calibrate it in the low pressure range from 5 kPa to 10 kPa. The calibration with the force-balanced piston gauge (Fluke FPG 8601) of LNE-LCM has shown a good linearity of the RSG in this range as illustrated in Figure 3. Associated with the low drift of its correction slope, the RSG can then be applied to check and rescale the calibration function of our working standard CDG 13 kPa full scale (Sect. 2.2) used to calibrate customers gauges.

2.2 Capacitance diaphragm gauge 10 kPa

Relative or absolute CDGs from the manufacturer MKS (in particular the 13 kPa full scale model) are currently used as secondary and working standards at LNE-LCM. To

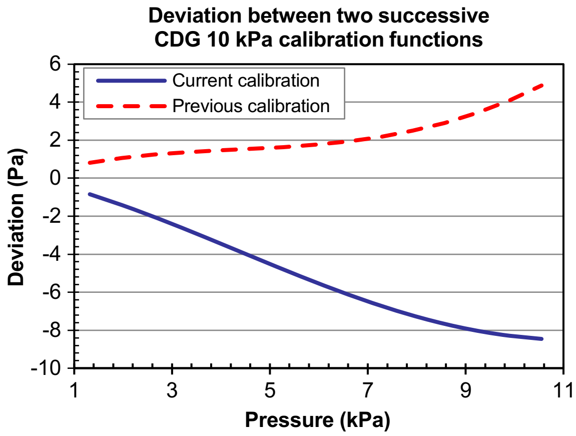

Fig. 5. Plot of the deviation of two calibration functions of a CDG 13 kPa full scale, in absolute pressure mode. The two calibrations are spaced by about 12 months. The deviation is the difference between the CDG calibration function and a linear function $g(U-U_0)=a(U-U_0)$, where a is an arbitrary coefficient.

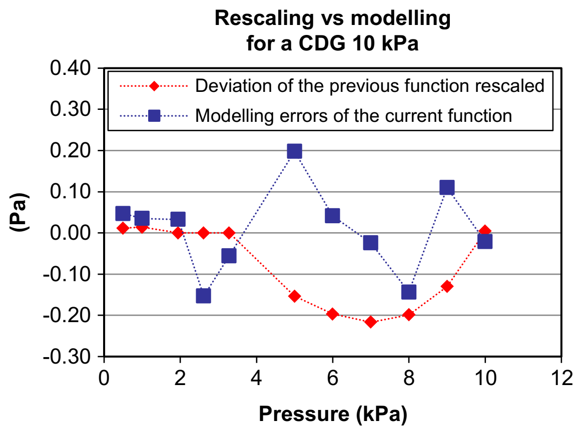

Fig. 6. Difference between the calibration function of a 10 kPa full scale CDG obtained in one case by modelling the calibration data (f_t), and in the other case by applying a correction factor k_{CDG} to the previous calibration function ($k_{CDG} \times f_{t-1}$). Here k_{CDG} is the slope coefficient of the least squares fit to a straight line that is used to estimate 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: ($f_t - p_{FPG}$).

enhance the gauge resolution, it is generally preferable to treat the analogue output U (0–10 V), rather than the digital one. Thus the calibration function is expressed using an equation of the form:

$$p = f(U - U_0), \tag{1}$$

where U_0 is the output signal of the gauge when zero pressure is applied (which in absolute mode corresponds to a pressure lower than a tenth of the gauge resolution) and $f(U-U_0)$ is a polynomial function of up to fourth order. As the CDG is controlled at a temperature of 45 °C, a thermal transpiration correction [5] is applied to calculate the reference pressure p. We then applied the empirical

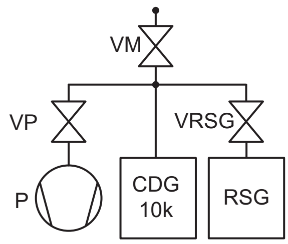

Fig. 7. Set-up of the working standard.CDG10k: capacitance diaphragm gauges MKS type 690 with an effective full scale of 10 kPa; RSG: resonant silicon gauge Druck type DPI142 (3.5–130 kPa); P: vacuum pump; VM: isolation valve of the working standard; VRSG: isolation valve of the RSG; VP: isolation valve of the pump.

function of Takaishi-Sensui [5]; one can find in the literature other work on this correction [6,7]. In absolute mode, the polynomial function and the gauge temperature are determined from a calibration using the FPG, for pressures between 1 kPa and 10 kPa.

From the numerous calibrations of CDGs performed, it was stated that for the mean term, the shape of a calibration curve does not change much, as one can see at a glance from Figure 5; consequently, it is possible to estimate the new calibration function f_t by applying a linear correction to the previous one, f_{t-1}. Let us denote by f'_t the function determined by the linear correction (k_{CDG} being the linear correction factor), as f_t and f_{t-1} are the calibration functions obtained from the CDG successive calibrations. We have:

$$f'_t(U - U_0) = k_{CDG} \times f_{t-1}(U - U_0), \tag{2}$$

where k_{CDG} is the slope coefficient of the least-squares-fitted straight line used to estimate 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[2]. This is the method used to rescale a CDG. From the example of Figure 5, with quite a 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 6 as a function of the pressure.

On this same graph are plotted 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 FPG 8601. 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 3.0×10^{-5} in relative value.

3 Experimental set-up

The metrological features of the instruments, described in Section 2, make possible the rescaling of the CDG of 10 kPa (effective) full scale, starting with a comparison between the CDG 10 kPa and the RSG between 5 kPa and 10 kPa.

[2] A large part of the CDGs used at LNE-LCM exhibits a significant non linearity between 80% and 100% of the full scale so we don't use them in this range.

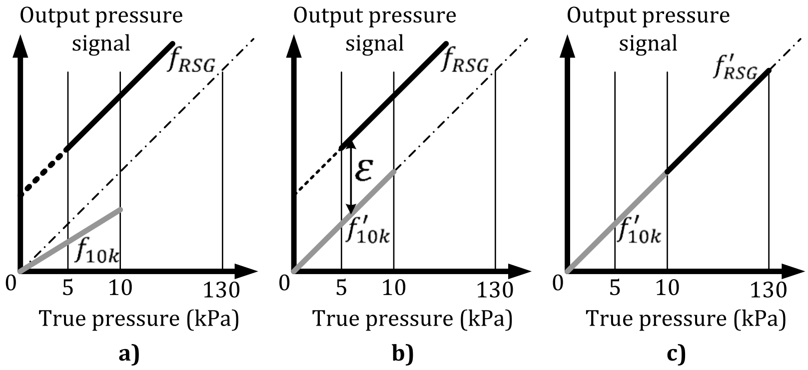

Fig. 8. Procedure to correct the working standard.The straight line passing through the origin with a slope equal to unity represents the output signal of the ideal instrument.Respective drifts of the RSG (in offset) and CDG (in slope) are voluntarily exaggerated for a greater clarity.

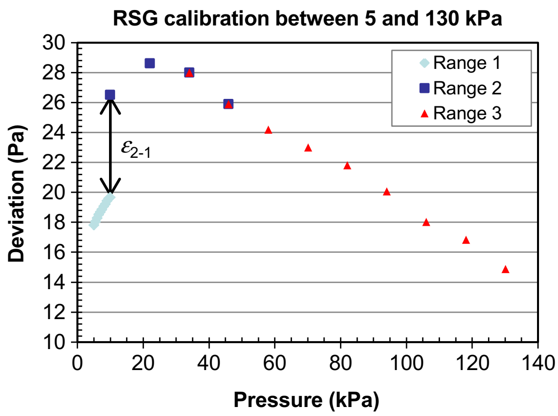

Fig. 9. Deviation of the RSG between 5 and 130 kPa.The whole range is split into three (1 to 3) to model the corresponding calibration functions: range 1 lies from 5 to 10 kPa, range 2 from 10 to 46 kPa and range 3 from 35 to 130 kPa.

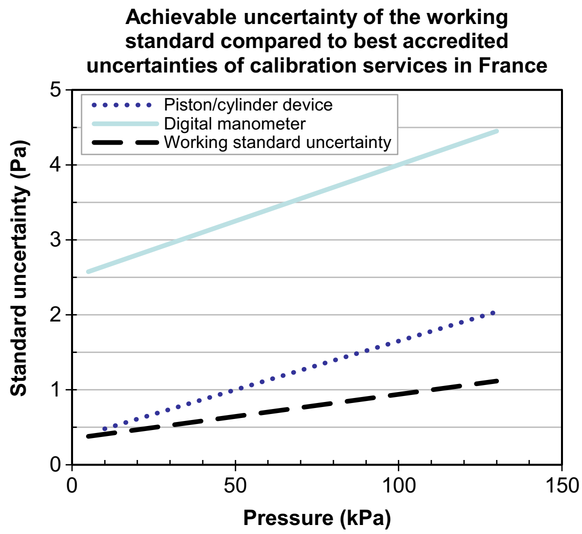

Fig. 10. Final uncertainty of the working standard together with the best accredited uncertainties of calibration services in France and the corresponding apparatus employed.

Once rescaled, the CDG 10 kPa is used to determine the offset of the RSG. The experimental set-up of the transfer standard is described in Figure 7.

The CDG10k is an MKS Instruments 690 absolute pressure transducer connected to an MKS 670 electronics package. The vacuum pump, whose ultimate pressure is lower than 0.01 Pa, is used to determine the zero U_0 of the CDG10k.

4 Procedure to use the working standard

4.1 Principle

This working standard is actually based on the simultaneous operation, in a common pressure range, of a standard with a very stable linear correction, i.e. the RSG and a standard for which the zero can be routinely determined, namely the CDG 10 kPa. The associated metrological properties of the instruments lead to a low uncertainty contribution of the working standard. The procedure to correct the output signal of the CDG and the RSG is illustrated in Figure 8. Each graph has the true pressure along the horizontal axis and the output pressure signal given by the calibration function of each instrument along the vertical axis. On these graphs, the ideal instrument output signal is the straight line passing through the origin with a slope equal to unity. Let us denote f_{RSG} the calibration function of the RSG and f_{10k} that of the CDG: both output signals given by the functions are coinciding with the ideal instrument output signal at the time of their respective calibration. Sometime later (graph a, Fig. 8), the RSG has drifted in offset: the output signal still has a slope equal to unity but no longer crosses zero. As for that of the CDG, it still crosses zero, as U_0 is determined each time of using, but the slope has drifted slightly. Several measurements performed at the same pressure levels between 5 kPa and 10 kPa allow one to determine the linear correction k_{10k} (according to Eq. (2)) *via* a least-squares analysis. The corrected calibration function f'_{10k} is:

$$f'_{10k} = k_{10k} \times f_{10k}. \quad (3)$$

The mean difference ε between the RSG output signal and that of the CDG10k given by f'_{10k} at the aforementioned measurement points allows one to correct in turn the

Table 1. Modelling errors of the RSG for three different pressure ranges.

Calibration function	Range (kPa)	Maximum modelling error (Pa)
f_1	5–10	0.18
f_2	10–46	0.33
f_3	35–130	0.37

function (f_{RSG}). The corrected function (f'_{RSG}) is:

$$f'_{RSG} = f_{RSG} - \varepsilon. \quad (4)$$

The new output signal of the RSG calculated with f'_{RSG} coincides again with that of the ideal instrument, between 10 kPa and 130 kPa.

4.2 Application to LNE-LCM instruments

The RSG is calibrated in two steps to cover the range from 5 kPa to 130 kPa (Sect. 2.1): the first range 10–130 kPa with an absolute pressure balance and the second range 5–10 kPa with the force balanced piston gauge FPG 8601. A difference of a few Pascals in the deviation at 10 kPa from the two calibrations is observed, since the offset of the RSG drifts slightly (Fig. 9). Moreover, the RSG shows a non-linearity for pressures around 25 kPa (Sect. 2.1). In Figure 9 which shows the deviation at the calibration points over the entire range, one can see that f_{RSG} cannot be expressed by means of a single function. Consequently, the RSG range has been split into three: f_1 denotes the function for pressures between 5 kPa and 10 kPa obtained from the calibration with the FPG 8601 and is linear; f_2 covers the non-linear part of the RSG characteristic between 10 and 46 kPa, obtained from the calibration with the absolute pressure balance and is a second-order polynomial; finally f_3 is a linear function for pressures between 35 kPa and 130 kPa obtained from the aforementioned calibration.

We have denoted by ε_{2-1} the offset drift during the short time between the calibration with the pressure balance and the FPG:

$$\varepsilon_{2-1} = f_2(10\,\text{kPa}) - f_1(10\,\text{kPa}). \quad (5)$$

The CDG10k rescaling (Sect. 4.1) is performed using the functionf_1 of the RSG. The mean difference betweenf'_{10k} and f_1 at different chosen measurement points is thus denoted by ε_1.It is used to obtain the actual output signal of the RSG by means of the corrected functions f'_1, f'_2 and f'_3:

$$f'_1 = f_1 - \varepsilon_1, \quad (6)$$

$$f'_2 = f_2 - \varepsilon_{2-1} - \varepsilon_1, \quad (7)$$

$$f'_3 = f_3 - \varepsilon_{2-1} - \varepsilon_1, \quad (8)$$

The working standard is used as follows: valves VM and VRSG are closed, valve VP opened and the pump P switched on. After two hours of pumping, the CDG10k output voltage U_0 is recorded. The pump is then switched off and Valve VP is closed. At the same time, the calibration chamber connected to the other side of the valve VM is filled up to the first pressure calibration level. Valve VM is opened to start a calibration. Once the pressure reaches 5 kPa, Valve VRSG is opened and some common measurement points for CDG and RSG are performed during the course of the calibration. These operations are repeated for each calibration cycle (three cycles are recommended), after which data are post-processed to determine the values of k_{10k} and ε_1 for each calibration cycle.

Table 2. Rescaling coefficient of the CDG10k and drift in the offset of the RSG.

	k_{10k}	ESD(k_{10k})	ε_1 / Pa	ESD(ε_1) / Pa
Cycle 1	0.99968	3.7×10^{-5}	9.68	0.14
Cycle 2	0.99966	3.7×10^{-5}	10.01	0.14
Cycle 3	0.99965	2.7×10^{-5}	10.29	0.10

5 Characterisation of the working standard

5.1 Linearity and hysteresis errors of the RSG

From the functions f_1, f_2, and f_3 used to model the RSG and the actual calibration pressure, the linearity errors have been calculated. Since the RSG is calibrated by increasing and decreasing pressure levels, the determined linearity errors also include the hysteresis error of the RSG. A closer analysis shows that the main error is due to the hysteresis effect (as one can observe Fig. 3, for the range 5–10 kPa), with a maximum model error summarised in Table 1, depending upon the range considered.

5.2 Rescaling of the CDG and offsetting of the RSG

The characterisation of the working standard took place during the study of a low pressure transfer standard [1], as well as in the framework of the EMPIR project 14IND06 [8]. The CDG10k used was a differential one; however, its behaviour is analogous to that of an absolute CDG and so has similar performances. Three calibration cycles were made in which six common measurement pressure readings equally distributed between 5 kPa and 10 kPa were taken (namely 5, 6, 7, 8, 9 and 10 kPa).

The correction factor k_{10k} is the slope coefficient of the least-squares-fitted straight line that estimates $f_1(p_{RSG})$ as a function of $f_{10k}(U - U_0)$. At each pressure level i, we calculate:

$$\varepsilon_{1i} = f_1(p_{RSGi}) - k_{10k} \times f_{10k}(U_i - U_0). \quad (9)$$

The mean drift in offset of the RSG from its calibration function f_1 is then given by:

$$\varepsilon_1 = \frac{1}{6}\sum_{i=1}^{6}\varepsilon_{1i}. \quad (10)$$

The values of the rescaling coefficient k_{10k}, the offset deviation ε_1 and their respective experimental standard deviation ESD(k_{10k}) and ESD(ε_1) are shown in Table 1. We

Table 3. Uncertainty budget in the determination of ε_1.

Uncertainty component	Value
Calibration with the FPG 8601	$1.5\times10^{-5}\times p$
RSG slope stability	$6.6\times10^{-6}\times p$
Rescaling coefficient determination: ESD(k_{10k})	$3.7\times10^{-5}\times p$
Experimental standard deviation: ESD(ε_1)	0.14Pa
Ambient temperature (±3 K)	$2.0\times10^{-6}\times p$
Combined uncertainty $u(\varepsilon_1)$ ($k=1$)	0.22Pa for $p=10$ kPa

Table 4. Uncertainty budget in the contribution of the working standard.

Uncertainty component	Value
f_1 offset correction: $u(\varepsilon_1)$	0.22Pa
RSG slope stability	$6.6\times10^{-6}\times p$
Linearity and hysteresis	0.21Pa
Ambient temperature (±3 K)	$2.0\times10^{-6}\times p$
Combined uncertainty ($k=1$)	$4.9\times10^{-6}\times p+0.28$ Pa
Non applied correction: offset modelling between f_2 and f_3	0.13 Pa
Working standard contribution uncertainty $u_{cont}(p)$ ($k=1$)	$u_{cont}=4.9\times10^{-6}\times p+0.35$ Pa

can observe that the offset deviation ε_1 slightly drifts between the cycles, so it is relevant to take into account each individual cycle rather than a mean value calculated on the three cycles.

Note that the modelling errors of the CDG10k (Sect. 2.2) are lower than the maximum value of ESD(k_{10k}).

5.3 Temperature influence

As the overall performance of the transfer standard is based on the correction slope of the RSG, it is important to check to what the extent it is affected by temperature. To determine the temperature coefficient, the RSG was placed in a climate controlled chamber at temperatures successively of 20 °C, 15 °C, 25 °C then 20 °C once more 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 $(-5.5\pm3.8)\times10^{-7}$ K^{-1}. For a difference of 3 K, which is a huge tolerance for an accredited Laboratory, the temperature effect never exceeds 2×10^{-6} in relative value.

6 Uncertainty budget

We first calculate the uncertainty contribution of the working standard. Following the procedure described Section 4, we determine the rescaling coefficient k_{10k} of the CDG10k from the slope of the corrected signal of RSG, and then the RSG offset deviation ε_1. Table 3 lists the uncertainty components of ε_1. The calibration uncertainty for the gauge FPG 8601 is $1.5\times10^{-5}\times p$ ($k=1$) while the drift in the RSG correction slope is $6.6\times10^{-6}\times p$ (Sect. 2.1). We assume that the modelling errors of both CDG10k and RSG are already taken into account in the maximum value of the experimental standard deviations ESD(k_{10k}) and ESD(ε_1) respectively (*cf.* Tab. 2). Furthermore, the RSG linearity error is used to calculate the uncertainty contribution of the working standard (Tab. 4). Finally, the temperature effect is $2\times10^{-6}\times p$ (Sect.5.3) for 3 K.

The uncertainty in the function f_1 offset correction $u(\varepsilon_1)$ is maximal at 10 kPa and equal to:

$$u(\varepsilon_1)=0.22\ \text{Pa}. \tag{11}$$

To calculate the contribution in uncertainty of the working standard, u_{cont}, we combine with $u(\varepsilon_1)$ at 10 kPa, the overall linearity and hysteresis error of the RSG on its whole range (the maximum value is 0.37 Pa from the Tab. 1 with a rectangular distribution), the RSG slope stability (Tab. 3) and the temperature effect. The RSG pressure is calculated either with the function f_2 or f_3 at 35 kPa and 46 kPa (overlap area); the maximum discrepancy between f_2 and f_3 is 0.13 Pa is considered as an unapplied correction i.e. an intrinsic systematic error correction term to account for the slight mismatch that is present in overlapping pressure scale regions and which is added to the expanded uncertainty ($k=2$) at the final stage. In Table 4, only the standard uncertainty is specified.

We have calculated the uncertainty u_{WSt} in using the working standard. For that purpose, it suffices to combine its contribution u_{cont} with the best capability of the LNE-LCM for the range 10–130 kPa, u_{CMC}, that is:

$$u_{CMC}=3.5\times10^{-6}\times p+0.05\ \text{Pa}. \tag{12}$$

We then obtained:

$$u_{Wst} = 5.9 \times 10^{-6} \times p + 0.35\,\text{Pa}, \qquad (13)$$

which leads to a maximum uncertainty of 1.1 Pa at 130 kPa. For comparison with the existing capabilities of French calibration services Figure 10 shows u_{Wst} together with the best capability obtained with a pressure balance and a digital manometer respectively. We can state that the working standard studied here could disseminate the lowest level of uncertainty in pressure in the range 5–130 kPa, which is at least four times better than that of the best digital manometer.

7 Conclusion

The experimental study of two pressure instruments with a common pressure range and complementary metrological features has allowed us to produce a high-accuracy working standard in the absolute pressure range from 5 kPa to 130 kPa, with an uncertainty lower than 1 Pa ($k = 1$). The instruments are a commercially available CDG and RSG, of full scales 13 kPa and 130 kPa respectively. In the range 5–130 kPa, the RSG has the advantage that its linear correction remains stable over many years. By comparing the CDG with the RSG in the range 5–10 kPa, the CDG can be rescaled. Since it is straightforward to measure the zero signal of the CDG (given a suitable pumping unit), the latter in turn allows one to master the drift in the offset of the RSG. The uncertainty analysis based on a strict procedure leads to an uncertainty contribution of the working standard ($k = 1$) which rises from 0.37 Pa to 0.99 Pa over the range 5–130 kPa.

The mathematical tools used to model the output signals of the instruments and calculate the different corrections are basic (polynomial functions, unweighted least-squares fitting) and available in most spreadsheets software. Consequently, it is straightforward for an experienced calibration service in pressure metrology to apply the method described in this paper once it has identified the most suitable instruments for the purpose.

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