On the stability of capacitance-diaphragm gauges with ceramic membranes

K. Jousten[a)]
Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany

Simon Naef[b)]
INFICON AG, Alte Landstrasse 6, 9496 Balzers, Liechtenstein

(Received 2 July 2010; accepted 29 November 2010; published 4 January 2011)

Capacitance-diaphragm gauges with ceramic membranes or diaphragms have been on the market for about 15 years. The long-term stability of these devices with full scales from 13 Pa to 133 kPa has been tested in the past decade by the calibration of gauges used by the manufacturer as reference gauges on the production line. These reference gauges were calibrated annually on a primary standard. It was found that the reproducibility of these devices depends on their full scale. For 13 Pa, the annual reproducibility near full scale varied between 0.02% and 0.05%, and for full scales of 133 Pa and higher, it varied between 0.005% and 0.03% of full scale. The reproducibility of the ceramic capacitance-diaphragm gauges for full scales of 133 Pa and 1.3 kPa was significantly lower than the uncertainty of a primary standard applying the static-expansion method. [DOI: 10.1116/1.3529023]

I. INTRODUCTION

In the past 15 years, capacitance-diaphragm gauges (CDGs) with ceramic membranes, also called diaphragms, have become popular for pressure measurements in many different applications such as etching, chemical-vapor deposition, physical-vapor deposition, thin-film deposition, and for use as transfer standards. INFICON, a well-known manufacturer of such gauges, started a cooperation with the Physikalisch-Technische Bundesanstalt (PTB) in 2000 in order to qualify their CDGs as reference standards and to ensure the measurement specifications of the production line. CDGs with full scales (FSs) from 13.3 Pa up to 133 kPa were calibrated annually at PTB. Since typically four CDGs were calibrated at the same time, changes common to all gauges gave PTB the chance to check the reproducibility of their primary standard as well.

II. CAPACITANCE-DIAPHRAGM GAUGES WITH CERAMIC MEMBRANES

Capacitance-diaphragm gauges have been established as the most precise commercial vacuum-measurement device suitable for process applications. The measurement principle is based on the deflection of a diaphragm under a differential pressure across it.

Instead of having an all-metal-based sensor design, INFICON's ceramic-diaphragm gauge technology is based on sintered alumina (Al_2O_3). The diaphragm and entire housing are made of $>99.5\%$ pure Al_2O_3.

The diaphragm is bonded with the housing using a glass solder. The upper housing, the getter dome, and the diaphragm form a reference cavity, which is kept in a low vacuum range by a nonevaporable getter. The lower housing with the diaphragm forms the measurement cavity. The electrodes are located on the reference vacuum side and form the capacitor (see Fig. 1).

A capacitance-diaphragm gauge measures the pressure difference between the reference cavity and the measurement cavity. The membrane bends if pressure is applied; the deflection is directly related to the applied pressure. The two extreme cases are shown in Fig. 2. Figure 2(a) shows the full-scale pressure deflection of the membrane, and Fig. 2(b) shows the "zero" deflection of the membrane.

Beyond the full-scale pressure, the membrane is supported by the upper housing of the sensor cell that results in good overpressure resistance.

The deflection within the zero pressure and the full-scale pressure is described as fixed circular plates under a uniform load. The deflection is proportional to the pressure p as long as the deflection is significantly less than the membrane thickness. The diaphragm deflection ω in the center of the diaphragm can be calculated by the following equation:[1]

$$\omega(p) = \frac{p \cdot r^4 \cdot 3 \cdot (1 - \upsilon^2)}{16 \cdot E \cdot t^3} \left[m = \frac{\mathrm{Pa} \cdot m^4}{\mathrm{Pa} \cdot m^3} \right], \tag{1}$$

where ν is the Poisson ratio (unitless), E is the Young modulus, t is the thickness of the diaphragm, and r is the free radius of the diaphragm.

The typical deflection at full-scale pressure is adjusted to a few micrometers, which, in relation to a several hundred micron-thick membrane, is only about 1%. The sensitivity to pressure is varied by the diaphragm's thickness.

In the center of the upper housing, a measurement electrode is located. The counter electrode is on the membrane. The distance change due to pressure change is measured by the resultant capacitance change,[2]

[a)]Electronic mail: karl.jousten@ptb.de
[b)]Electronic mail: simon.naef@inficon.com

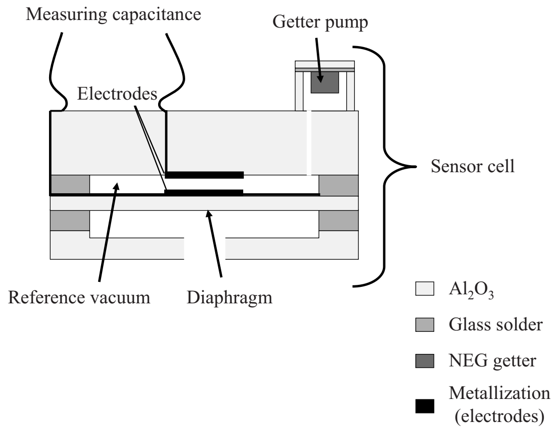

FIG. 1. (Color online) Structure of INFICON ceramic-diaphragm gauge.

$$\Delta C_{\text{meas}}(\omega) = \frac{\varepsilon_0 \cdot A}{d_0 + \omega} - C_0 \left[F = \frac{\text{As}}{V} = \frac{\text{As} \cdot m^2}{Vm \cdot m} \right], \quad (2)$$

where d_0 is the distance at zero pressure, ε_0 is the dielectric permittivity, A is the area of the housing electrode, ΔC_{meas} is the resultant capacitance change, and C_0 is the capacitance at zero deflection.

The electronics measures the capacitance change due to pressure change and scales the raw signal into a linear 0–10 V signal output. The relation of the indicated pressure p_{ind} and voltage measured at signal output voltage U and for full scale FS is given by[3]

$$p_{\text{ind}}\ (\text{Pa}) = \frac{U\ (\text{V}) \cdot \text{FS}\ (\text{Pa})}{10.0\ \text{V}}. \quad (3)$$

Besides the heat controller and the internal power supply, the electronics processes the raw capacitance reading through five defined stages. The first stage is the capacitance measurement, which is based on a sigma-delta capacitance/voltage converter. The second stage consists of prescaling of the raw signal.

A feedback loop for linearization is the third stage since the capacitance measurements add some nonlinearity caused by the hyperbolic relationship of distance to capacitance (see Fig. 3).

The fourth stage is the temperature compensation. The temperature compensation applies to zero (temperature effect on zero) and the span (temperature effect on span). Finally, the output stage changes the output impedance and contains all protection devices against electromagnetic compatibility and reverse polarity.

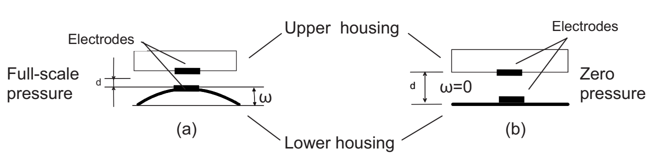

FIG. 2. Schematic diaphragm bending.

The tested reference gauges were of the standard 45 °C thermally stabilized ceramic-diaphragm gauge type (INFICON CDG045) with full scales from 13.3 Pa up to 133 kPa.

III. CALIBRATION

The CDGs were calibrated annually by standards for low and medium vacuums at the PTB with nitrogen as the test gas. For CDGs with full scales up to 1.3 kPa, two primary standards were used, namely, the SE2 static-expansion system and the FRS5 pressure balance (Furness Control, Inc.).

In the static-expansion system, SE2 pressures are established by expanding a known gas amount from a small volume into much larger volumes. The pressure in such a system can be calculated and compared to the indication of a vacuum gauge to be calibrated.[4,5] SE2 covers the range from 0.1 Pa to 1.3 kPa.[4,6]

In the FRS5 pressure balance, the pressure is measured by the force acting on a piston, where the lower side of the piston is in high vacuum.[7] The force is measured by a force meter. The FRS5 can accurately measure pressures from 30 Pa up to 11 kPa,[8] but in this investigation, it was used only for calibrations up to 1.3 kPa.

Beyond 1.3 kPa, a 7010 quartz Bourdon spiral was used as the secondary standard for calibration. The 7010 is traceable to the mercury manometer of PTB.[9]

The voltage output 0–10 V of the CDGs was measured by a calibrated digital voltmeter. Typically, four CDGs were calibrated at the same time. The value in volts was converted to the pressure unit in millibar (1 mbar=100 Pa) by a respective multiplier, which was 1.33322 times full scale in torr divided by 10. The calibration range covered 3 decades from 0.1% to 100% of full scale, where the target points were set to (0.1%, 0.5%, 1%, 5%, 10%, and 20% through 100%).

The measurand to be determined was the error of reading e defined as

$$e = \frac{p_{\text{ind}} - p_{\text{cal}}}{p_{\text{cal}}}, \quad (4)$$

where p_{ind} is the pressure indicated by the CDG as described above and p_{cal} is the pressure defined by the primary standard or measured by the secondary standard of PTB.

Four gauges for each full scale were shipped under atmospheric pressure conditions. Between recalibrations, two CDGs of each full scale were stored in a cabinet under atmospheric pressure conditions as a backup, whereas the other two pieces of each full scale were used for the calibration of the production tools at INFICON.

For economical reasons, only one measurement series was taken for each CDG. An additional uncertainty was added to cover the repeatability of the measurements. An example of a calibration curve for a CDG with a full scale of 1.3 kPa (10 torr) is shown in Fig. 4.

The uncertainties related to the calibration are as follows:

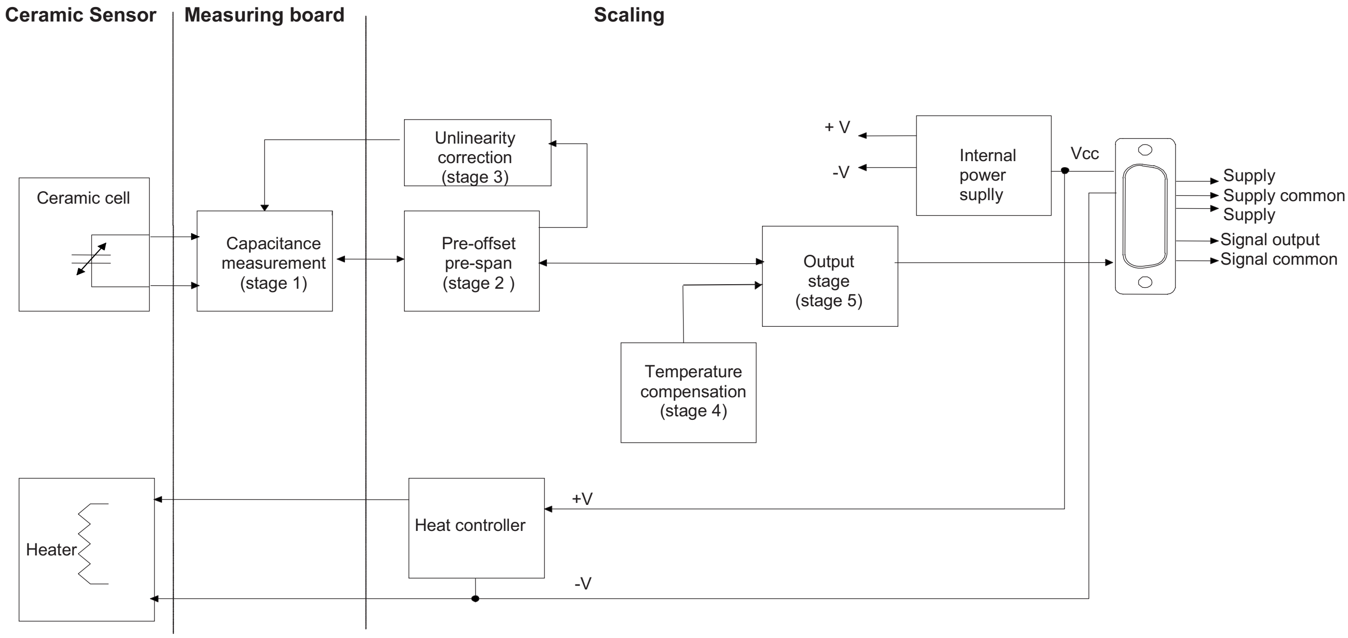

FIG. 3. Electronics concept of heated ceramic-diaphragm gauge.

(1) the uncertainty of the calculated pressure in the respective primary standard or the measurement uncertainty of the secondary standard,
(2) the repeatability of the measurements, and
(3) the measurement uncertainty of the CDG under calibration.

The relative uncertainties u of the calculated pressures in the primary standard SE2 ranged from 0.094% (standard uncertainty, coverage interval is about 68%, $k=1$, k being the so called coverage factor as a multiple of the standard uncertainty) at 0.1 Pa to 0.074% at 1.3 kPa. For the FRS5, the values ranged from $u=0.072\%$ at 30 Pa to 0.0029% at 1.3 kPa. The u of the 7010 was between 0.019% at 1.4 kPa and 0.014% at 130 kPa.

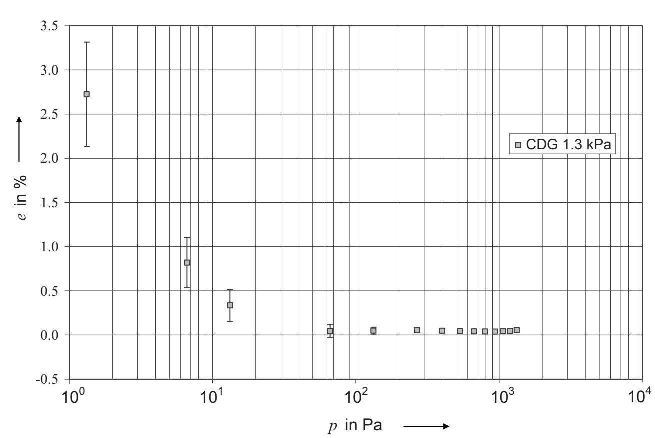

FIG. 4. Calibration curve of a capacitance-diaphragm gauge with full scale of 1.3 kPa. e is the relative error of indication. Calibration below 30 Pa was performed by the static-expansion system at PTB above 30 Pa by use of a pressure balance. At higher pressures, the uncertainty bars for the 95% confidence interval are smaller than the size of the symbols.

The repeatability of the measurements $(k=1)$ with SE2 was considered to be 0.08% for pressures $p<10$ Pa and 0.03% for 10 Pa$\leq p<1.4$ kPa and with 0.01% for the measurements with the 7010. These values were estimated from repeated measurements on the same day of several CDGs of the same type. The repeatability is both due to the reference standard and the device under calibration, but in the case of SE2, dominated by the primary standard. Since the newly established FRS5 had a better repeatability than the SE2, a new investigation was conducted, which is described in the following section.

During calibration, the uncertainty due to the CDG itself was mainly caused by the drift and measurement of the offset. Typically, this amounted to a few parts of 10^5 of the full-scale reading and dominated the total uncertainty in the lowest of the three calibration decades. The digitizing error of the digital voltmeter could be neglected.

IV. REPEATABILITY

To assess the repeatability of the ceramic CDG with 1.3 kPa (10 torr) full scale, three identical devices $(j=12,22,23)$ were repeatedly (16 times, $l=1,\ldots,16$) calibrated at the same time by the FRS5 at 30 Pa and 1.2 kPa. Between each measurement, the pressure was pumped down to below the resolution limit of the device. The 16 calibrations were performed one after the other within 3 h. For each CDG j, the mean error of reading $\bar{e}_j$ and the experimental standard deviation as estimate of the repeatability were determined,

$$s_j=\sqrt{\frac{1}{15}\sum_{l=1}^{16}(e_{lj}-\bar{e}_j)^2}. \quad (5)$$

The results are given in Table I. At 30 Pa, the mean repeatability of the three gauges is 0.027%, which is practically

TABLE I. Repeatability of 16 calibrations of three ceramic capacitance-diaphragm gauges with full scale of 1.3 kPa against an FRS5 pressure balance at two different pressures p of 30 and 1200 Pa. The repeatability is defined in Eq. (5).

p (Pa)	Device 12 (%)	Device 22 (%)	Device 23 (%)	Mean (%)
30	0.020	0.029	0.032	0.027
1200	0.0025	0.0016	0.0081	0.004

identical to the one with SE2. At 1200 Pa, however, it is much lower, at 0.004%.

The values in Table I include both the repeatability of the FRS5 and of the CDGs. If, however, several gauges are calibrated at the same time, it is possible to distinguish between the repeatability of the FRS5 reference standard and the CDGs by employing a statistical model. This is currently being investigated, and the results will be reported elsewhere.

V. LONG-TERM INSTABILITY

The long-term instability was evaluated near full scale for all types of CDGs and additionally at 5 Pa for the CDGs with full scale at or below 133 Pa and at 7 Pa for the 1.3 kPa full scale.

To quantify the long-term instability for the annual recalibrations, we use the following two approaches:

(1) the experimental standard deviation s about the all-time mean $\bar{e}$ of a gauge and
(2) the mean of absolute (non-negative) changes between recalibrations $\bar{\Delta}$.

The two quantities are defined as

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(e_i - \bar{e})^2}, \qquad (6)$$

$$\bar{\Delta} = \frac{\sum_{i=1}^{n-1}|e_{i+1} - e_i|}{n-1}, \qquad (7)$$

where e_i is the error of reading in year i and n is the total number of annual recalibrations.

When n is not too small and the changes are purely random, the two values are very similar. When a systematic drift is observed over several years in addition to random variations, $\bar{\Delta}$ will be smaller than s.

It was evident after the recalibrations (Fig. 5) that the uncertainty of the calibration with the SE2 static-expansion system was too high to evaluate the long-term stability of the devices with a full scale of 1.33 kPa directly. In addition to the four ceramic sensors by INFICON, a check standard of PTB was calibrated, which was an Inconel-type CDG by MKS Instruments with the same full scale. All five CDGs were calibrated at the same time each year and showed similar systematic changes from year to year. Although the changes from year to year are within the measurement uncertainty, they indicate a common effect which was the generated calibration pressure p_{cal} in SE2. For this reason, it was more complicated to estimate the long-term instability of the CDGs with full scale 1.33 kPa and below with the calibration at SE2.

To estimate the long-term stability in this case, it is necessary to distinguish between the influence of the primary standard and the influence of possible annual shifts of the indication of the gauges under calibration. Normally, it is not possible to distinguish between the two effects. In this case, however, when four gauges are calibrated at the same time, an estimate is possible; although the gauges were all of the same type, it can be expected that long-term shifts are different from gauge to gauge. Hence, it can be assumed that the shift of the mean value of all gauges can be attributed to p_{cal}, which can vary within its uncertainty, and the scatter of a single gauge around this mean shift can be attributed to the specific gauge. So, we calculated the mean value of all error of readings in year i and $\bar{e}_i$ by

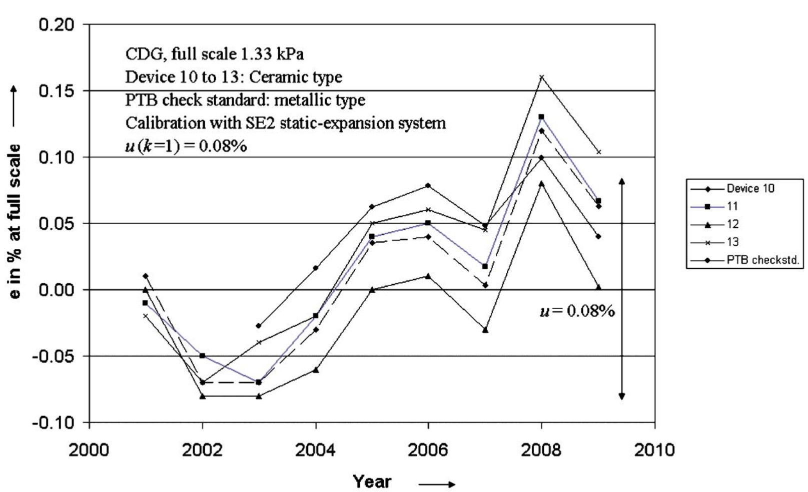

FIG. 5. (Color online) Error of reading at full scale for CDGs with full scale of 1.3 kPa for 8 years calibrated with the SE2 primary standard. Standard measurement uncertainty u (k=1) was 0.08%.

TABLE II. Apparent long-term instability of ceramic CDGs with full scale 1.33 kPa in percent of pressure near full scale. Different equations defined in the third column were applied. The recalibration period was one year; data were observed within 9 years. The results with the FRS5 as standard are based on four successive years only. The rows with SE2 as standard and s and $\bar{\Delta}$ calculated by Eqs. (6) and (7) do include the uncertainty of the standard and are not suitable to characterize the CDGs' long-term instability.

Standard	Quantity	Equation	Device 10	Device 11	Device 12	Device 13	Mean
SE2	s	Eq. (6)	0.062	0.062	0.051	0.074	0.062
	$\bar{\Delta}$	Eq. (7)	0.050	0.049	0.050	0.046	0.049
SE2	s_j	Eq. (12)	0.010	0.015	0.009	0.010	0.011
	$\bar{\delta}$	Eq. (13)	0.009	0.010	0.009	0.012	0.010
FRS5	s	Eq. (6)	0.009	0.005	0.009	0.012	0.009
	$\bar{\Delta}$	Eq. (7)	0.013	0.006	0.012	0.008	0.010

$$\bar{e}_i = \frac{1}{N}\sum_{j=1}^{N} e_{ij}, \tag{8}$$

where e_{ij} is the error of reading of gauge j in year i and N is the number of gauges. The shift from year i to $i+1$ for the mean is denoted by

$$d_{i+1} = \bar{e}_{i+1} - \bar{e}_i, \tag{9}$$

and for a single gauge with

$$\delta_{i+1,j} = e_{i+1,j} - e_{ij}. \tag{10}$$

If no scatter due to the gauges occurs,

$$\delta_{i+1,j} - d_{i+1} = 0 \quad \text{all } j. \tag{11}$$

So, the root-mean-square deviation

$$s_j = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n-1}(\delta_{i+1,j} - d_{i+1})^2} \tag{12}$$

gives a reasonable estimate of the long-term instability of the calibrated CDG j. Similar to Eq. (7), we can also observe the quantity

$$\bar{\delta}_j = \frac{\sum_{i=1}^{n-1} |\delta_{i+1,j} - d_{i+1}|}{n-1}, \tag{13}$$

which is the average change per year over the recalibration period. A more rigorous treatment would need to consider the random variations of d_i as well, but since we are interested in a rough estimate only and have already carried out eight recalibrations, this is of minor significance.

Table II shows the results of the two approaches according to the Eqs. (6), (7), (12), and (13), respectively. When the reproducibility of the SE2 apparatus of about 0.058% (standard deviation of d_i) is considered by the Eqs. (12) and (13), the long-term instabilities for the CDGs are much lower and more realistic.

In 2006, the realization of the pressure scale above 30 Pa was improved by establishing the FRS5 pressure balance.[8] The four CDGs with full scale 1.33 kPa were calibrated by the FRS5, in addition to the calibration at SE2. The observed changes from year to year for a single gauge decreased by a factor of 5, and there were no clear common systematic changes for all four gauges (Fig. 6). The results from these calibrations directly give realistic values for the long-term instability of the CDGs. In the case of full scale 1.33 kPa,

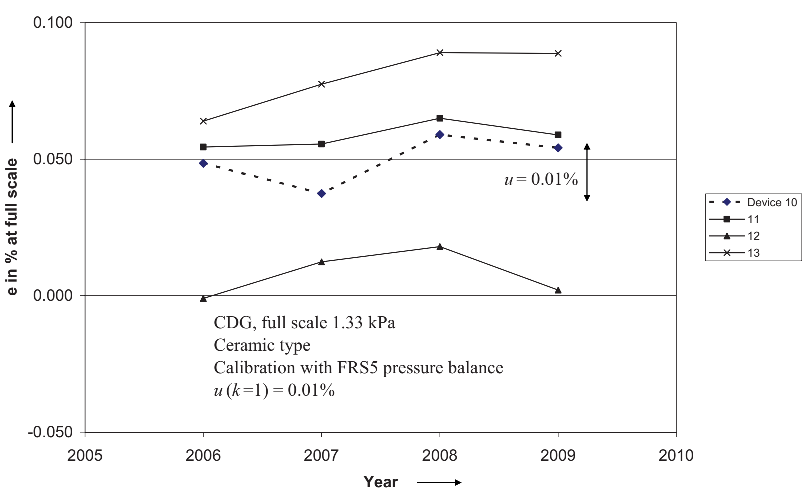

FIG. 6. (Color online) Error of reading at full scale for CDGs with full scale of 1.3 kPa for 4 years calibrated with the FRS5 pressure balance. Standard measurement uncertainty u $(k=1)$ was 0.01%.

TABLE III. Long-term instability of ceramic CDGs with full scales from 13 Pa to 133 kPa in percent of pressure near full scale. For each full scale, the values for four or five devices are given (in total of 21 different devices). The equation of the quantity for the long-term instability is given in the third column. The recalibration period was one year; data were generally observed within 9 years. The results for the 1.33 kPa full scale CDGs are based on four successive years only. For two CDGs with a full scale of 133 kPa (devices 3 and 4), data were ignored when recalibration showed a change of about −1% in 2 years (device 20) or 1 year (device 21).

FS	Quantity	Equation	Device 1	Device 2	Device 3	Device 4	Device 5	Mean
13 Pa	s_j	Eq. (12)	0.040	0.038	0.021	0.042	0.041	0.036
	$\bar{\delta}$	Eq. (13)	0.037	0.029	0.014	0.033	0.031	0.029
FS	Quantity	Equation	Device 6	Device 7	Device 8	Device 9		Mean
133 Pa	s_j	Eq. (12)	0.014	0.015	0.012	0.009		0.013
	$\bar{\delta}$	Eq. (13)	0.009	0.011	0.009	0.008		0.009
FS	Quantity	Equation	Device 10	Device 11	Device 12	Device 13		Mean
1.33 kPa	s	Eq. (6)	0.009	0.005	0.009	0.012		0.009
	$\bar{\Delta}$	Eq. (7)	0.013	0.006	0.012	0.008		0.010
FS	Quantity	Equation	Device 14	Device 15	Device 16	Device 17		Mean
	s	Eq. (6)	0.009	0.011	0.015	0.016		0.013
13.3 kPa	$\bar{\Delta}$	Eq. (7)	0.007	0.008	0.010	0.011		0.009
FS	Quantity	Equation	Device 18	Device 19	Device 20	Device 21		Mean
	s	Eq. (6)	0.006	0.017	0.040	0.015		0.019
133 kPa	$\bar{\Delta}$	Eq. (7)	0.005	0.021	0.026	0.010		0.016

they should be similar to the ones calculated from above. The last two rows in Table II show that this is indeed the case.

Table III summarizes the observed values of s [Eq. (6)] and $\bar{\Delta}$ [Eq. (7)] or s [Eq. (12)] and $\bar{\delta}$ [Eq. (13)], respectively, over 9 years for CDGs from 13 Pa to 133 kPa full scale, except for CDGs with 1.33 kPa full scale, where only 4 years serve as the database for the reason described above.

The lowest long-term instability was 0.005% (device 11 with full scale 1.33 kPa and device 18 with full scale 133 kPa). The highest long-term instability was 0.042% for the 13 Pa full-scale range (device 4).

Two of the four ceramic CDGs with full scale 133 kPa showed a change of −1% after 6 (device 20) and 7 (device 21) years, respectively, of recalibrations between successive years. These data were ignored in Table III. These two devices had an accessible potentiometer for the full-scale adjust which were mistakenly misadjusted. Both devices could be readjusted and recalibration was resumed.

In Table IV, the values of s and $\bar{\delta}$ at a pressure of 5 Pa are shown for CDGs with a full scale of 13 and 133 Pa. In the case of 13 Pa full scale, these values are a factor of 1.6 higher than the ones at full scale, whereas in the case of 133 Pa, they are larger by a factor of about 3.5. The reason is that the variability of the offset due to drift and its measurement already affected the value at 5 Pa for the 133 Pa full scale. This is also true for the values of s and $\bar{\Delta}$ at a pressure

TABLE IV. Long-term instability of ceramic CDGs with full scales of 13 Pa and 133 Pa in percent of pressure at 5 Pa. s_j and $\bar{\delta}$ are defined in Eqs. (12) and (13). The recalibration period was 1 year; data were observed within 8 years (13 Pa) or 9 years (133 Pa).

Full scale	Quantity	Device 1	Device 2	Device 3	Device 4	Device 5	Mean
	s_j	0.085	0.062	0.025	0.058	0.060	0.058
13 Pa	$\bar{\delta}$	0.072	0.052	0.018	0.047	0.044	0.047
Full scale	Quantity	Device 6	Device 7	Device 8	Device 9		Mean
	s_j	0.022	0.071	0.040	0.038		0.043
133 Pa	$\bar{\delta}$	0.020	0.057	0.030	0.030		0.034

TABLE V. Long-term instability of ceramic CDGs with full scales of 1.33 kPa in percent of pressure at 7 Pa. s and $\bar{\Delta}$ are defined in Eqs. (6) and (7). The recalibration period was 1 year; data were observed within 9 years.

Full scale	Quantity	Device 10	Device 11	Device 12	Device 13	Mean
	s	0.124	0.097	0.131	0.098	0.113
1.33 kPa	$\bar{\Delta}$	0.144	0.095	0.162	0.083	0.121

of 7 Pa for the 1.33 kPa full scale (Table V), where 7 Pa is only 0.5% of full scale. In this case, Eqs. (6), (12), (7), and (13), respectively, delivered very similar values. This indicates that the uncertainty of the primary standard did not play a significant role at pressures of 0.5% of full scale.

VI. DISCUSSION

This is the first investigation of the long-term stability of CDGs with a ceramic diaphragm. Previous investigations published data on the long-term stability of CDGs with a metal diaphragm.[10,11] In the study by Hyland and Tilford,[10] the authors found changes in the error of indication for recalibrations near full scale between 0.05% and 0.86% for full scales of 133 kPa, 0.07%–0.24% (13.3 kPa), 0.1%–1% (1.33 kPa), and 0.1%–2% (133 Pa) with unequal recalibration periods between 0.4 and 4.5 years. In the study by Grosse and Messer,[11] similar changes of 0.03%–0.25% for 133 Pa and 0.09%–0.34% for 1.33 kPa full scale were reported for several gas species, but only one gauge each.

These values for metal CDGs seem to indicate that the long-term instability near full scale is higher than that of ceramic CDGs. This is also the experience of the vacuum metrology laboratory at PTB gained from repeated customer calibrations. Since the long-term instability is the dominant uncertainty for measurements and calibrations with CDGs in many cases, PTB will publish these data on metal CDGs in the near future. Despite the better long-term stability near full scale of the ceramic CDGs, it must be mentioned, however, that the accuracy of high-resolution CDGs with a metal diaphragm below 1% of full scale is significantly better, compared to the CDGs with a ceramic membrane.

In comparison with resonance silicon gauges,[12] the long-term shifts of ceramic CDGs are about a factor of 3 higher for pressures below 1.3 kPa and a factor of 10–100 higher for pressures up to 100 kPa. Similar to ceramic CDGs and static-expansion systems, resonance silicon gauges seem to be so stable that their true long-term instability at 100 kPa cannot be determined with the uncertainty of a high-level mercury primary standard.

VII. CONCLUSION

The technology of ceramic CDGs has gained a great deal of maturity, and the long-term instability is within 0.01%–0.02% (full scale $\geq$133 Pa), which is so low that the uncertainties of the primary standards of the static-expansion type are too high to observe the true long-term instability directly. Near full scale, ceramic CDGs can be recommended as very stable transfer standards. It must be noted, however, that the CDGs tested here were stored, transported, and used under relatively pure conditions with either atmospheric air around them or nitrogen as the test gas. The results of this investigation are not representative for CDGs in contaminating process environments.

ACKNOWLEDGMENTS

The careful acquiring of data by Christian Buchmann and Marco Schulz of PTB, discussions with Clemens Elster of PTB, and Per Björkman of INFICON are gratefully acknowledged.

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