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Calibration of capacitance diaphragm gauges with 1333 Pa full scale by direct comparison to resonant silicon gauge and static expansion system

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ABSTRACT
Two capacitance diaphragm gauges (CDGs) with 1333 Pa full scale, with a heated sensor head and an unheated one, respectively, were calibrated by three different methods; direct comparison to a resonant silicon gauge calibrated by a pressure balance, direct comparison to a CDG with 133 Pa full scale calibrated by a static expansion method, and the static expansion method. The calibration results of the three calibration methods show good agreement within their claimed uncertainties. Calibrated higher pressure points of CDGs by the pressure balance and lower pressure ones by the static expansion system are linearly interpolated within the calibrated uncertainty. Here, compensation of the thermal transpiration effect is important when a CDG is used with a heated sensor head.

Section: RESEARCH PAPER

Keywords: pressure, vacuum, standard, calibration, capacitance diaphragm gauge, thermal transpiration effect

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1. INTRODUCTION

Since pressure/vacuum gauges are calibrated at multiple pressure points, interpolation between these points is necessary for practical pressure measurements. In the case that the pressure points are calibrated by a single standard technique with good linearity, the interpolation has high reliability in general. At pressures lower than 10^3 Pa, however, interpolation between pressure points from two different standard techniques is often required. In such a case, the validity of the interpolation should be confirmed. A capacitance diaphragm gauge with 1333 Pa full scale (CDG-10Torr) is used for precise pressure measurements in the range from 1 Pa to 10^3 Pa. For the calibration of the CDG-10Torr at least four candidate techniques are available: pressure balance, static expansion system (SES) [1-5], force-balanced piston gauge [6,7], and oil manometer [5,8,9].

In this paper, the calibration results of the CDG-10Torr based on two different standards are presented. One is a direct comparison to the resonant silicon gauge (RSG), which is calibrated by the pressure balance. The RSG is used as a reliable transfer gauge in the field of pressure and vacuum standards [10,11]. The other is the static expansion system [4]. The calibration results are compared and the validity of the interpolation is discussed.

2. EXPERIMENTAL

2.1. Apparatus

Figure 1 shows the schematic diagram of the static expansion system (SES) and the direct comparison system (DCS) for the calibration of vacuum gauges. These two systems are connected to each other through all metal valves. Two resonant silicon gauges with 130 kPa full scale (absolute) are located on the SES (RSG_{SE}) as reference gauges.
A capacitance diaphragm gauge with 133 Pa full scale (CDG-1Torr) is located between the SES and the DCS, and used as a reference gauge for the DCS. Two capacitance

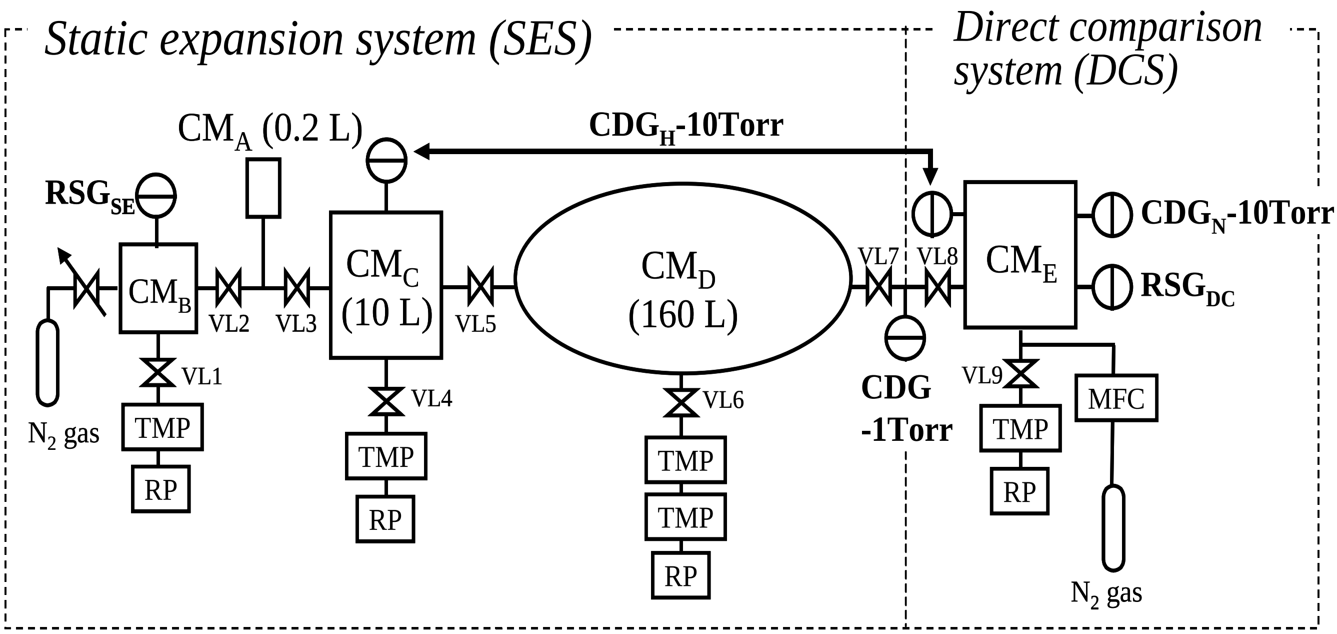

Figure 1. Schematic diagram of the static expansion system (SES) and the direct comparison system (DCS) for the calibration of vacuum gauges.

diaphragm gauges with 1333 Pa full scale were used as test gauges. A high accuracy absolute type capacitance diaphragm gauge with a heated sensor head at a temperature of 45 ^{o}C (CDG_H-10Torr) was tested at both the SES and DCS. Another capacitance diaphragm gauge with unheated sensor head (CDG_N-10Torr) was tested at the DCS only. N_2 gas was used as a test gas.

The calibration procedure of the SES is briefly summarized. The pumping system of the SES consists of turbo molecular pumps (TMP) and rotary pumps (RP). The background pressure before calibration is typically around 10^{-7} Pa. The gas in the initial chamber CM_A was expanded to the chamber CM_C or both chambers CM_C and CM_D depending on the calibration pressure range. To avoid changes in volume and temperature, a reference gauge to measure the initial pressure before expansion is not located on the CM_A. The initial pressure was measured by the RSG_{SE} located on the chamber CM_B by closing both valves VL1 and VL3 and opening the valve VL2. After the initial pressure measurement, static expansion was performed by closing VL2, VL4, VL6, and VL8 and opening VL3 only and/or VL3, VL5 and VL7. The calibration pressure ranges are from 1 Pa to 2000 Pa and from 10^{-4} Pa to 150 Pa at CM_C and CM_D, respectively. Details of the SES are given in Ref [4].

The DCS was constructed based on the ISO 3567 Vacuum gauges – Calibration by direct comparison with a reference gauge [12]. Two reference gauges are located in the DCS. One is the resonant silicon gauge with 130 kPa full scale absolute (RSG_{DC}). The other is a high accuracy absolute type capacitance diaphragm gauge with 133 Pa full scale with a heated sensor head at the temperature of 45^{o}C scale (CDG-1Torr). The CDG-1Torr is used as a reference gauge without detaching the sensor head from the chamber by controlling VL7 and VL8. The pumping system consists of a turbo molecular pump (200 L/s for N_2) and a rotary pump. A static method is adopted for direct comparison. The valve on the TMP (VL9) was closed when the background pressure is lower than 10^{-4} Pa, which is measured by an ionization gauge. The zero points of the CDGs and the RSG_{DC} were measured every time before each calibration. The test gas was introduced to the CM_E by a computer-controlled mass flow controller (MFC) with a full scale of 10 sccm until the pressure in the CM_E has reached the target pressure. The test gauge was calibrated by comparing the reference gauges while the test pressure is kept constant for 300 s.

2.2. Traceability chain in this study

The traceability chain of the pressure in this study is summarized in Figure 2. The RSG_{SE} and RSG_{DC} were calibrated by the pressure balance from 5.0×10^{3} Pa to 1.3×10^{5} Pa. The RSG_{DC} was sometimes calibrated by direct comparison to the RSG_{SE} to check its long-term stability. The CDG-1Torr was calibrated by the SES in the chamber CM_D from 0.1 Pa to 130 Pa. In the SES, both the expansion ratio and the initial pressure at the chamber CM_A, which are important parameters to determine the standard pressure, are measured by the RSG_{SE}.

The CDG_H-10Torr with a heated sensor head was calibrated by three methods; (i) direct comparison to the RSG_{DC} from 100 Pa to 1300 Pa, (ii) direct comparison to the CDG-1Torr from 1 Pa to 130 Pa, and (iii) static expansion

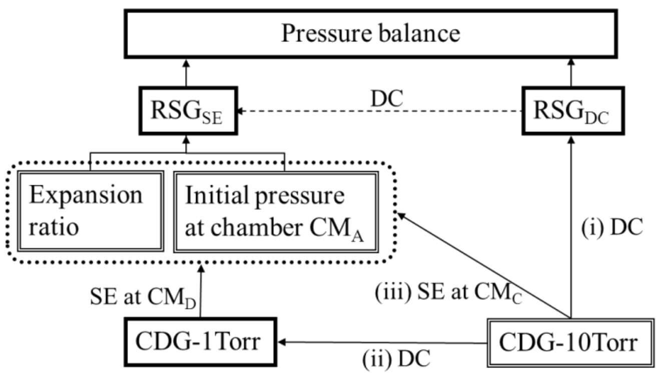

Figure 2. Traceability chain of the pressure in this study. SE and DC mean static expansion and direct comparison. Two capacitance diaphragm gauges with 1333 Pa full scale (CDG-10Torr) were calibrated by (i) DC to RSG_{DC}, (ii) DC to CDG-1Torr, and (iii) SE at CMc.

method at the chamber CM_C from 1 Pa to 1300 Pa. The CDG_N-10Torr with an unheated sensor head was calibrated by using methods (i) and (ii). The direct comparison with the RSG_{DC} was performed by extrapolating the calibration results obtained from 5.0×10^3 Pa to 1.3×10^5 Pa. The procedure of the extrapolation is detailed in section 3.1.

2.3. Compensation of Thermal transpiration effect

The apparent change in the sensitivity of the CDG owing to the thermal transpiration effect was compensated using the Takaishi-Sensui (T-S) equation:

$$\frac{p_1}{p_2} = \frac{Y + \sqrt{T_1/T_2}}{Y+1}, \qquad T_1 < T_2$$
$$Y = AX^2 + BX + C\sqrt{X}, \;\; X = dp_2 \tag{1}$$
$$A = aT^{-2}, B = bT^{-1}, C = cT^{-1/2}, T = (T_1 + T_2)/2$$

p_1 and p_2 are the pressures in the vacuum chamber and in the sensor head (capsule) of CDG that corresponds to the pressure indication of CDG, respectively. T_1 and T_2 are the temperatures of the vacuum chamber and the sensor head of CDG, d is the inner diameter of the connecting tube, and a, b, and c are parameters depending on the gas species to be measured. T_1 was measured befor every calibration. The values of T_2 and d were assumed to be 45^{o}C (318.15 K) and 4.76 mm, respectively. Parameters a, b, and c were equal to 12×10^5 deg^2 mmHg^{-2} mm^{-2} (6.75×10^7 K^2 Pa^{-2} m^{-2}), 10×10^2 deg mmHg^{-1} mm^{-1} (7.50×10^3 K Pa^{-1} m^{-1}) and 14 deg$^{1/2}$ mmHg$^{-1/2}$ mm$^{-1/2}$ (38.3 K$^{1/2}$ Pa$^{-1/2}$ m$^{-1/2}$), respectively. The validities of the T-S equation and these parameters are discussed in [15].

3. RESULTS

3.1. Calibration results of the reference resonant silicon gauges (RSG)

Calibration results of the reference RSG_{DC} and the RSG_{SE} by a pressure balance are shown in Figure 3. The vertical axis is the deviation of the calibrated standard pressure (p_S) from the pressure indication (p_I) of the RSGs. The sensitivity coefficient S of the RSGs is defined as in equation (2) for a pressure range down to 100 Pa,

$$S = (p_I - p_{I0}) / p_S = \Delta p_I / p_S, \tag{2}$$

where p_{I0} is the pressure indication at the background pressure, in other words at zero point, and Δp_I is the difference between p_{I0} and p_I. The $S(RSG_{DC})$ is plotted in Figure 4 with a logarithmic scale of the horizontal axis. The $S(RSG_{DC})$ has a constant value of 0.999987 ± 0.000027. The standard pressure ($p_{RSG\text{-}DC}$) in the DCS from 100 Pa to 1300 Pa is determined by equation (3),

$$p_{RSG\text{-}DC} = \Delta p_I \;/\; S\,(RSG_{DC}). \tag{3}$$

The calibration uncertainty $U(p_{RSG\text{-}DC})$ with a confidence level of 95% (k=2) is estimated by equation (4):

$$U(p_{RSG\text{-}DC})\ [\text{Pa}] = -1.3\times10^{-11}\,\Delta p_I^2 + 5.0\times10^{-6}\,\Delta p_I + 3.0, \tag{4}$$

which is the best fitting curve between Δp_I of the RSG_{DC} and its expanded uncertainty. That means the relative expanded uncertainty of $p_{RSG\text{-}DC}$ from 100 Pa to 1300 Pa is in the range from 3.0% to 0.23%.

3.2. Calibration result of the reference capacitance diaphragm gauge with 133 Pa full scale (CDG-1Torr)

A calibration result of reference CDG-1Torr by SES is shown in figure 5 (a). The vertical axis is the S of CDG-1Torr, which is similarly calculated by eq. (2). The S(CDG-1Torr) increases with decreasing the pressure by thermal transpiration effect because CDG-1Torr has a heated sensor head at the temperature of 45 ^{o}C [10,13-15]. Figure 5 (b) shows the compensated S(CDG-1Torr) by eq. (1) [15,16]. The S(CDG-1Torr) after the compensation by T-S equation has a constant value of 1.0081 ± 0.0014. The relative expanded uncertainty of the calibration from 0.1 Pa to 130 Pa is in the range from 2.8 % to 0.33 % [4].

3.3. Calibration results of two capacitance diaphragm gauges with 1333 Pa full scale (CDG_H-10Torr and CDG_N-10Torr)

The CDG_H-10Torr was calibrated using three methods: (i) direct comparison to the RSG_{DC} from 100 Pa to 1300 Pa, (ii) direct comparison to the CDG-1Torr from 1 Pa to 130 Pa, and (iii) the static expansion method from 1 Pa to 1300 Pa. Table 1 shows the uncertainty budget of (i) and (ii). The expanded uncertainty of (iii) is in the range from 1.0% to 0.26% [4]. As shown in Figure 6(a), the three calibration results for the CDG_H-10Torr are in good agreement within their required uncertainties. The sensitivity of the CDG_H-10Torr, $S(CDG_H$-10Torr), also increases with decreasing pressure by the thermal transpiration effect. After compensation using eq. (1), the $S(CDG_H$-10Torr) also has a linear characteristic within ± 0.2% as shown in figure 6(b).

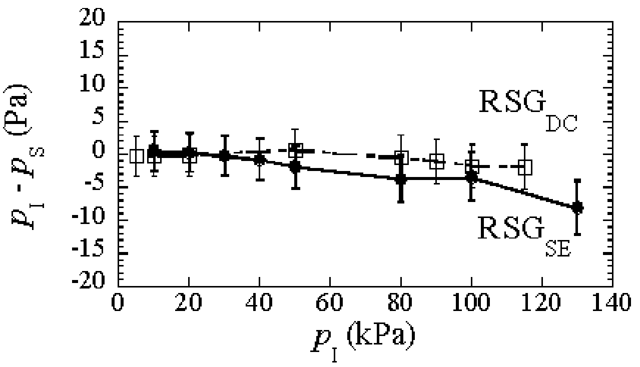

Figure 3. Calibration results ofthe RSG_{SE} and RSG_{DC}.

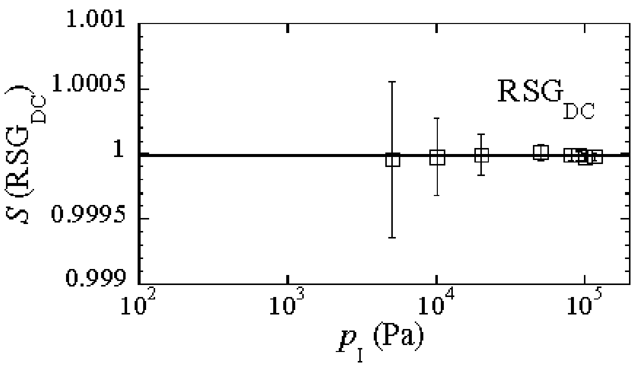

Figure 4. Sensitivity S of the RSG_{DC}.

Table 1. The uncertainty budget of the calibration results of the CDG_H-10Torr by direct comparison. The RSG_{DC} and CDG-1Torr are used as a reference gauge depending on the pressure range.

Reference gauge			(1) RSG_{DC}	(2) CDG-1 Torr
Pressure range (Pa)			100 - 1300	1 - 130
Relative standard uncertainty with k = 1	Reference gauge	Calibration uncertainty	1.5 % – 0.12 % (3.0 Pa / 2)	0.22 % – 0.16 %
		Fluctuation of p_I	0.11 % – 0.01 %	< 0.03 %
		Resolution	0.29 % – 0.02 % (1 Pa / $2\sqrt{3}$)	< 0.02 % (6.7×10^{-4} Pa / $2\sqrt{3}$)
		Temperature	1.3 % – 0.10 % (1.3 Pa)	-
		Attitude	1.5 % – 0.12 % (1.5 Pa)	-
		Compensation of thermal transpiration effect [15]	-	0.2 % / $\sqrt{3}$
		Long-term stability	0.006 % / $2\sqrt{3}$	0.2 % / $\sqrt{3}$
	CDG_H-10Torr	Fluctuation of p_I	< 0.03 %	0.11 % -0.02 %
		Resolution (6.7×10^{-3} Pa / $2\sqrt{3}$)	< 0.002 %	< 0.19 %
	Calibration system	Pressure distribution	0.1 %/ $\sqrt{3}$	0.3 %/ $\sqrt{3}$
		Height	< 0.01 %	
	Reputability		0.29 % – 0.01 %	0.10 % – 0.002 %
	Combined standard uncertainty		2.6 % – 0.24 %	0.40 % – 0.29 %
Relative expanded uncertainty with k=2			5.1 % – 0.48 %	0.80 % – 0.58 %

Table 2. The uncertainty budget of the calibration results of the CDG_N-10Torr by direct comparison. The RSG_{DC} and CDG-1Torr are used as a reference gauge depending on the pressure range.

Reference gauge			(1) RSG_{DC}	(2) CDG-1 Torr
Pressure range (Pa)			100 - 1300	1 - 130
Relative standard uncertainty with k = 1	Reference gauge	Calibration uncertainty	1.5 % – 0.12 % (3.0 Pa / 2)	0.22 % – 0.16 %
		Fluctuation of p_I	0.11 % – 0.01 %	< 0.03 %
		Resolution	0.29 % – 0.02 % (1 Pa / $2\sqrt{3}$)	< 0.02 % (6.7×10^{-4} Pa / $2\sqrt{3}$)
		Temperature	1.3 % – 0.10 % (1.3 Pa)	-
		Attitude	1.5 % – 0.12 % (1.5 Pa)	-
		Compensation of thermal transpiration effect [15]	-	0.2 % / $\sqrt{3}$
		Long-term stability	0.006 % / $2\sqrt{3}$	0.2 % / $\sqrt{3}$
	CDG_N-10Torr	Fluctuation of p_I	< 0.03 %	1.1 % – 0.03 %
		Resolution (3.9×10^{-2} Pa / $2\sqrt{3}$)	< 0.011%	< 1.1 %
	Calibration system	Pressure distribution	0.1 %/ $\sqrt{3}$	0.3 %/ $\sqrt{3}$
		Height	< 0.01 %	
	Reputability		0.26 % – 0.01 %	0.88 % – 0.01 %
	Combined standard uncertainty		2.6 % – 0.24 %	1.8 % – 0.29 %
Relative expanded uncertainty with k=2			5.1 % – 0.48 %	3.6 % – 0.58 %

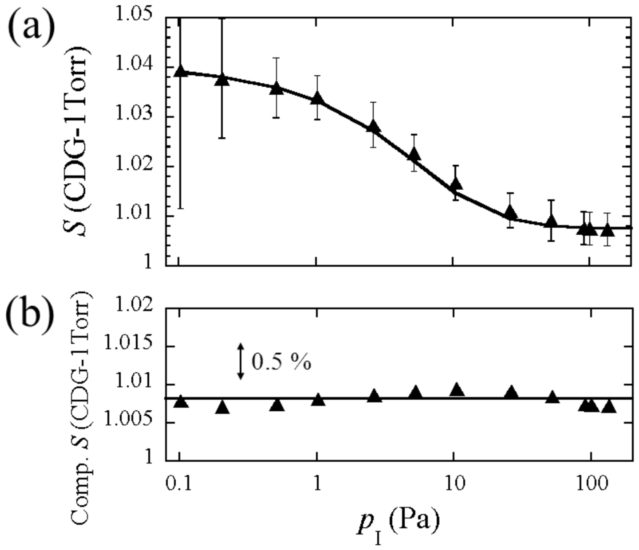

Figure 5. Sensitivity of the CDG-1Torr before (a) and after (b) compensation of the thermal transpiration effect by the Takaishi-Sensui equation.

4. DISCUSSION ON THE REFERENCE GAUGE FOR DIRECT COMPARISON

Calibration by direct comparison is widely used by many users. In the case that the RSG with 130 kPa full scale (absolute) is used as a reference gauge, the lowest calibration pressure may be limited by several hundred Pa if the calibration uncertainty is required to be within several %. The CDGs with 133 Pa or 1333 Pa full scale are useful as a reference gauge for pressures below 100 Pa. In that case, however, the thermal transpiration effect should be compensated if the CDG with heated sensor head is used. A wide calibration pressure range is realized by combining the RSG and the CDG as reference gauges and evaluating the uncertainty arising from the non-linearity of the sensitivity, the correction of the thermal transpiration effect, the resolution, the influence of temperature, attitude, and so on.

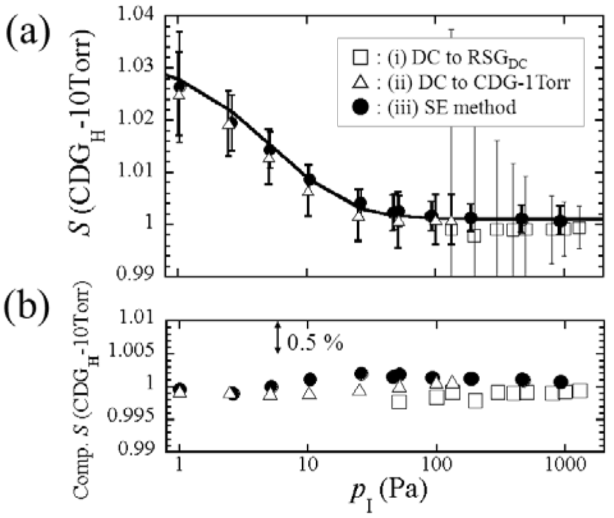

Figure 6. Sensitivity of the CDGH-10Torr with a heated sensor head at 45oC before (a) and after (b) compensation of the thermal transpiration effect by the Takaishi-Sensui equation [15,16].

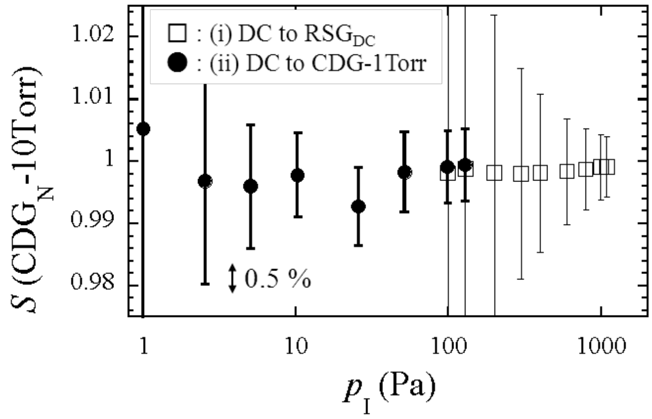

Figure 7. Sensitivity of the CDG_N-10Torr with an unheated sensor head.

5. CONCLUSION

Two capacitance diaphragm gauges with 1333 Pa full scale were calibrated by the following three methods: (i) direct comparison to a resonant silicon gauge with 130 kPa full scale absolute from 100 Pa to 1300 Pa, (ii) direct comparison to a capacitance diaphragm gauge with 133 Pa full scale from 1 Pa to 130 Pa, and (iii) static expansion method from 1 Pa to 1300 Pa. The results by these three methods show good agreement within their claimed uncertainties, which means these calibration methods and the uncertainty analyses are validated. Calibrated higher pressure points of CDGs by the pressure balance and lower pressure ones by the static expansion system are linearly interpolated within the calibrated uncertainty. Here, compensation of the thermal transpiration effect is important when a CDG is used with a heated sensor head.

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