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CHARACTERIZATION OF A STATIC EXPANSION STANDARD FOR CALIBRATING MEDIUM AND HIGH VACUUM PRESSURE GAUGES

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Abstract

The Centro de Desarrollo Tecnológico del Gas developed a static expansion system to enable the calibration of medium and high vacuum pressure gauges in Colombia. The system can generate pressures between 0.1 Pa and 100 kPa. The characterization tests included the evaluation of pressure stability and desorption rate, a trueness test, and the analysis of the uncertainty budget of the calibration result. The pressure stability test was successfully completed and showed the positive effect of baking on the final pressure in the system. The trueness test allowed concluding that the calibration results with the system are comparable with those obtained with a reference meter traceable to a national metrology institute. The uncertainty budget analysis indicated the dominance of the pressure of the unit under calibration and of the initial pressure in the small tank in different pressure ranges on the uncertainty of the result. A comparison with a Monte Carlo simulation led to the conclusion that in this situation, the GUM (Guide to the Expression of Uncertainty in Measurement) method is not ideal for estimating the uncertainty of the results.

Keywords: static expansion system, calibration, vacuum, characterization.

1. Introduction

Over time, the need for practical, consistently accurate and wide-range calibration methods for vacuum pressure gauges has increased as a result of the growing importance of vacuum in science and technology [1]. There is a wide range of industrial processes which require pressure measurement in medium and high vacuum, including food packaging, semiconductor manufacturing, metallurgical and chemical processes, and optical and electrical thin-film coating [2]. For many pressure ranges, primary pressure gauges are available, but below 0.1 Pa, no such standards are available. Therefore, for these pressure ranges, static or dynamic expansion systems must be used [3].

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A static expansion system is based on the conservation of the amount of gas stored in the system, and the reference pressure is obtained through static expansions of the gas [2,4]. It requires at least two tanks separated by a valve, with a way to measure the pressure in the first tank and a way to evacuate the second tank. Initially, a given amount of gas is isolated in the first tank, which has a known pressure. The gas is then shared between the two tanks by opening the valve between them. The pressure after expansion is calculated from the initial pressure, the temperature in each tank, and the ratio of the final volume to the volume of the first tank [5]. The static expansion method is typically used for calibration in medium and high vacuum between 1×10^{-5} Pa and 1 000 Pa, because in that range mercury pressure gauges are inadequate and dynamic expansion systems present some characterization problems [1, 6]. Although the static expansion technique can be used to generate calculable pressures as low as 1×10^{-6} Pa by performing multiple successive expansions of the gas in the calibration volume, some authors maintain that the static method presents some practical challenges for calibrations below 1×10^{-4} Pa [5, 7, 8]. It is also important to note that the relative uncertainty increases with each expansion [6].

To characterize a static expansion system, the tank volumes must be calibrated to find the expansion ratios, the traceability of the initial pressure and temperature gauges must be guaranteed, the uncertainty of the results must be estimated, and tests must be performed to confirm the trueness of the results obtained [4, 9]. In particular, it is crucial to determine the volume of the tanks with high accuracy [10, 11]. There are three methods to determine the expansion ratio: the gravimetric method, the gas accumulation method with two reference manometers, and the gas accumulation method with a strictly linear manometer (spinning rotor gauge) [6].

There are numerous physical phenomena which can significantly alter the results of low-pressure measurements [7]. Among the factors which must be considered or controlled are deviation from the ideal gas model, temperature differences in the system, and the interaction of the gas with the internal walls of the system [6, 12]. Regarding the deviation from the ideal gas model, in medium and high vacuum the contribution of the virial coefficient may not be important [7]. The effects of temperature and its corrections on the results in static expansion systems have been the subject of several studies [13]. Multiple temperature measurements and averaging are required since the temperature in the system is neither homogeneous nor constant [14]. Differences of up to 3°C have been reported in large tanks of static expansion systems [13]. Another thermal effect which can occur is thermal relaxation. This effect can cause in the large tank an initial instantaneous increase in the pressure after the expansion and a subsequent fall to the expected value in accordance with the Gay–Lussac thermal effect which states that just after expansion the gas in the initial tank cools down and the gas in the large tank warms up (although the effect is small) [13]. Typically, 15 s are sufficient to reach thermal equilibrium after expansion in large tanks, although in smaller tanks up to 5 min may be required [13]. As for the interaction between the gas and the walls, sufficiently low values of the total degassing rate and the residual pressure limit are required in the calibration chamber [15].

There is a great variety of designs and characteristics in the static expansion systems developed by different institutions. Generally, between 2 and 6 tanks are used. The volumes used in the tanks range from 0.01 L to 0.5 L for the smallest tank in the system, and from 50 L to 233 L for the largest tank [1, 2, 4, 6, 7, 10–12, 14]. The relative expanded uncertainties of the systems range from less than 1% to about 5% [2, 4, 6, 7, 16]. Reported errors can be as high as 15%, although this is not uncommon for measurements in the medium and high vacuum ranges [16]. There are also high differences in reported desorption rates, compounded by the fact that different institutions use different ways of reporting these rates. The commonly reported values are between 1×10^{-6} Pa L s^{-1} and 1×10^{-8} Pa L s^{-1} [2, 4, 6–9, 17].

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To calibrate instruments working in medium and high vacuum ranges, a static expansion system for calibrating absolute pressures in the range of 0.1 Pa to 100 kPa has been developed at the *Centro de Desarrollo Tecnológico del Gas* (CDT de Gas). The system consists of two containers separated by a valve, a pumping system and a nitrogen introduction line, as shown in Fig. 1. The operation of the static expansion system of the CDT de Gas is represented by the mathematical model described in (1), which involves the initial and final volumes, the initial pressure, and the initial and final temperatures of the process.

$$P_f = \frac{P_i V_P + P_{i,G} V_G}{V_P + V_G} \cdot \frac{T_f}{T_i}, \tag{1}$$

where:

P_f – pressure after expansion.
P_i – initial pressure in the small tank.
$P_{i,G}$ – initial pressure in the calibration chamber, whose value will be close to zero.
V_P – small tank volume.
V_G – calibration chamber volume.
T_i – temperature at the beginning of expansion.
T_f – temperature at the end of expansion.

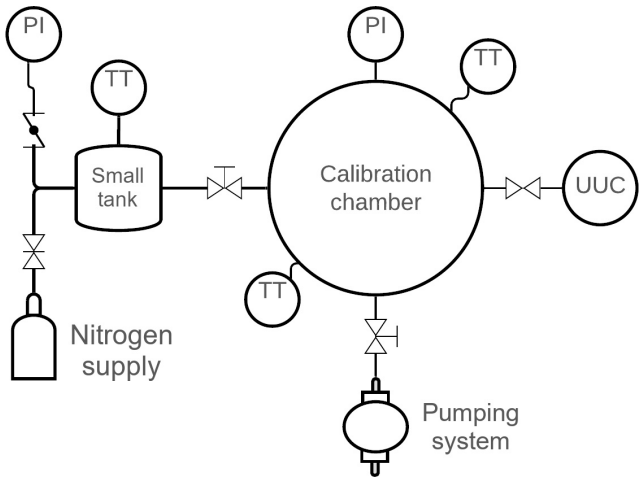

Fig. 1. Static expansion system diagram. PI – pressure measurement. TT – temperature measurement. UUC – unit under calibration.

To evacuate the system, a vacuum pumping system capable of reaching the base pressure, which for simplicity is called zero pressure, is required.

2. System description

The calibration standard consists of two containers of different sizes, with a ratio of approximately 1:16. The volume of every tank was determined by calibration using the gravimetric method. The small tank (initial volume) has a volume of 5.325×10^{-5} m^3 (5.325×10^{-2} L) with a standard uncertainty of 2.3×10^{-8} m^3 (2.3×10^{-5} L), and the large container (calibration chamber) has a volume of 8.079×10^{-4} m^3 (0.8079 L) with a standard uncertainty of 1.9×10^{-6} m^3

(0.0019 L). Thus, with each expansion the pressure is reduced to 6.1% of the initial pressure. The structure of the system is shown in Fig. 1.

The system containers are specialized parts for use in high vacuum. The calibration chamber is a 0.1143 m (4.5 inch) four-way reducing cross with Conflat flanges – flange CF to 0.06985 m (2.75 inch) CF are special flanges to achieve medium and high vacuum). On the other hand, the initial volume is a 0.033782 m (1.33 inch) CF tee pipe fitting. The valves conforming to the standard have a Conflat CF flange system with a metal-to-metal seal between the connections, allowing higher vacuum levels to be achieved.

3. Methodology

For the characterization of the standard, a pressure stability and desorption rate evaluation test of the standard, a trueness test and an uncertainty budget analysis were performed.

3.1. Evaluation of the pressure stability and desorption rate

The evaluation of the pressure stability and desorption rate was assessed by monitoring the pressure behavior in the calibration chamber of the static expansion system for 14 minutes, after evacuating the chamber for 0.5 hours and before performing an expansion. Two factors were varied: one was the number of expansions previously performed and the other was whether the calibration chamber was baked beforehand. The bakeout consisted of heating the calibration chamber for 15 hours at a temperature of 60 °C.

For the desorption rate, defined as the rate of change of pressure with time, its value was estimated by applying central finite difference formulas, and the values obtained were averaged. The formula for estimating the first derivative by central finite differences of a function f evaluated in the value x is (2).

$$f'(x) \cong \frac{f(x+h) - f(x-h)}{2h}, \tag{2}$$

where h is the distance between neighbouring x values on the discretized domain and is called the step size.

The pressure in the small tank is determined using an absolute pressure gauge with a working range between 500 mbar and 1 200 mbar. In the calibration chamber, the pressure gauge included in the pumping package is used to record the initial pressure in the chamber (*i.e.* before the expansion). Additionally, a reference pressure gauge is included in the calibration chamber, which allows the static expansion standard to be used in both calibration procedures. In the first one, the standard is used to generate the pressures and to determine the resulting pressure in the chamber by means of (1). The different pressures achieved are a function of the number of expansions performed and the initial pressure in the small tank before the first expansion. The second calibration method follows ISO 3567 and is based on a comparison between the meter to be calibrated and a secondary reference standard [15]. In this way, the standard is used to generate a certain vacuum level, but the pressure can be increased as required using a controlled leak valve, and the value given by the reference pressure gauge is used as the reference value. This second procedure yields results with greater uncertainty but has advantages in terms of calibration execution time.

Regarding the temperature measurement, it is not convenient to use temperature measuring instruments that are immersed in the gas because of the high vacuum in which they can operate. It is required to measure the temperature at the surface of the containers, and therefore contact

thermometers are used. The pumping system of the standard consists of two pumps in the same station: a mechanical pump which allows reaching the vacuum head, and a turbo-molecular pump which allows reaching a high vacuum. The pump outlet has a 0.1143 m (4.5 inch) Conflat flange.

3.2. Trueness test

This test consisted in defining two target pressure values, and comparing the value calculated by (1) with the value given by the reference meter used in the second calibration method. The reference meter is an MKS 722B Baratron capacitive manometer, model, which was calibrated at the *Centro Nacional de Metrología* (CENAM) in Mexico.

The conclusions of the trueness test are obtained by analyzing the value of the statistic E_n, which is used to evaluate the comparability of two measurements. The formula of the statistic is (3) [12].

$$E_n = \frac{X_{\text{exp}} - X_{\text{ref}}}{\sqrt{U_{\text{exp}}^2 + U_{\text{ref}}^2}}, \tag{3}$$

where X_{exp} is the pressure value yielded by the static expansion standard, and X_{ref} is the pressure value from measurements performed with the MKS Baratron, which has international traceability. The values shown in the denominator (U_{exp} and U_{ref}) are the expanded uncertainties of the static expansion standard and MKS Baratron pressure measurements, respectively. If the absolute value of E_n is less than or equal to 1, it will be concluded that the performance of the static expansion standard is satisfactory. When it is bigger than 1, the value given by static expansion is not consistent with the measurement yielded by the standard.

3.3. Uncertainty budget analysis

An analysis of the calibration uncertainty budget of a meter after different number of expansions in the system was developed. As a calibration measurand, the difference between the pressure reported by the unit under calibration and the pressure calculated by static expansion was calculated, as shown in (4).

$$\Delta P = P_{UUC} - \left(\frac{P_i V_P + P_{i,G} V_G}{V_P + V_G} \cdot \frac{T_f}{T_i} \right). \tag{4}$$

The objective of the analysis was to study the uncertainty contribution of each of the input quantities to the calibration result, and to explore the effect of performing multiple expansions on the relative importance of the different input quantities on the resulting uncertainty. The uncertainty and the uncertainty budget were estimated according to the GUM method, described in the Joint Committee for Guides in Metrology's Guide to the Expression of Uncertainty in Measurement [18]. Applying the GUM method to the measurement model, equation (5) for the estimation of the standard uncertainty is obtained.

$$u(\Delta P) = \sqrt{c_{P_{UUC}}^2 u_{P_{UUC}}^2 + c_{P_i}^2 u_{P_i}^2 + c_{P_{i,G}}^2 u_{P_{i,G}}^2 + c_{V_P}^2 u_{V_P}^2 + c_{V_G}^2 u_{V_G}^2 + c_{T_f}^2 u_{T_f}^2 + c_{T_i}^2 u_{T_i}^2}, \tag{5}$$

where c_x is the sensitivity coefficient for input quantity x and u_x is the standard uncertainty of input quantity x, with x being one of the seven input quantities in the measurement model. Values of u_{V_P} and u_{V_G} were taken from the calibration certificates of the tanks. Uncertainty of T_f, T_i and P_i were estimated by combining uncertainties for repeatability, resolution, traceability and drift.

For the estimation of the uncertainty of P_{UUC}, uncertainties for repeatability and resolution were combined. Uncertainty of $P_{i,G}$ was estimated by combining uncertainties for repeatability and an "operational" source of variability, used to represent the high uncertainty in the measuring of the initial pressure in the calibration chamber. For this source, an uncertainty of 20% of the measured value was assigned.

4. Results and analysis

4.1. Evaluation of the pressure stability and desorption rate

Initially, pressure stability and desorption rate were evaluated as described in Section 3.1. The results of these experiments are presented in Fig. 2.

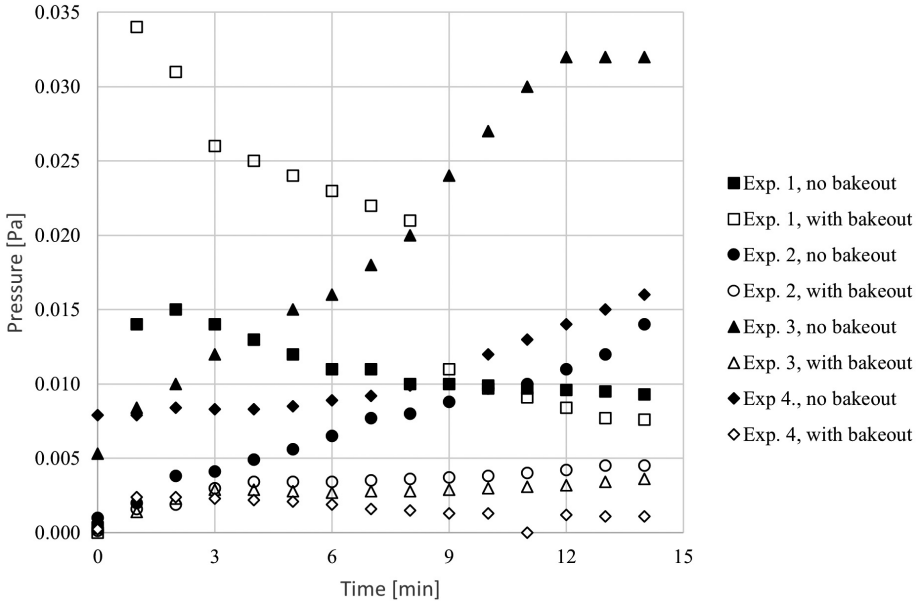

Fig. 2. Pressure stability in the calibration chamber under the different experimentally evaluated conditions.

During the time of the tests, some experiments did not show the expected behavior, i.e. a monotonic increase in pressure as desorption occurred, but rather a rapid initial increase in pressure (between minutes 0 and 1) and then a gradual decrease. This effect could be due to the thermal relaxation effect of the gas [13], or it could be due to the release of surface gas when closing the valve at the beginning of the test which would be consistent with the fact that this increase in pressure and subsequent stabilization at a lower value occurred mainly in the first expansion experiments. From the experimental data obtained, desorption rates were estimated by applying central finite difference formulas. Table 1 presents the average values obtained for the desorption rate under the different conditions studied.

The results of the stability test show that the pressure in the tests with bakeout tends to present a higher stability than the pressure when no bakeout had been performed. This can be attributed to the presence of the gas monolayer inside the walls of the calibration chamber when the equipment

Table 1. Mean outgassing rate (in Pa/s), during 14 minutes after the expansion and subsequent evacuation, in the different evaluated conditions of baking and number of stages of expansion.

	With bakeout	**Without bakeout**
One stage of expansion	-1.2×10^{-5}	2.85×10^{-6}
Two stages of expansion	4.67×10^{-6}	1.47×10^{-5}
Three stages of expansion	3.41×10^{-6}	3.22×10^{-5}
Four stages of expansion	-2.69×10^{-7}	9.74×10^{-6}

had not been baked, because of previously executed calibration processes. A lower final pressure is also evident when the equipment has been baked.

With respect to the evaluation of the desorption rate, the results obtained indicate that bakeout decreases the desorption rate inside the calibration chamber, favoring the stability of the pressure, which is evidenced by lower pressure values which are maintained for a longer period of time. Some of the average desorption rates for the case of bakeout are negative, because of the thermal effects evidenced at the beginning of the test.

4.2. Trueness test

Considering the calibration interval of the MKS instrument performed at CENAM, comparison points were chosen within the range between 0.012 Pa and 93 Pa. The points selected were approximately 10 Pa and approximately 1 Pa. These pressure values can be achieved with the static expansion system at the end of expansions number 3 and 4, starting from an initial pressure of approximately 50 000 Pa. Table 2 shows the data obtained in the test, as well as the average values and estimated uncertainties.

Table 2. Trueness test data. P_{MM}: pressure measurement with the MKS-722B master meter. P_{Exp}: pressure calculated using the static expansion system.

Objective pressure [Pa]	**Reference pressure [Pa]**				**Static expansion pressure [Pa]**			
	Replicate 1	**Replicate 2**	**P_{MM} [Pa]**	**u(P_{MM}) [Pa]**	**Replicate 1**	**Replicate 2**	**P_{Exp} [Pa]**	**u(P_{Exp}) [Pa]**
10	9.16	9.84	9.5	0.577	10.5	11.72	11.11	1.014
1	1.35	1.19	1.27	0.333	1.41	1.29	1.35	1.06

For the expanded uncertainties of each equipment, a level of confidence k=2 was used, corresponding to approximately 95% coverage. Based on the data above, the statistic E_n is calculated. The results are presented in Table 3.

Table 3. Calculated E_n statistics.

Objective pressure [Pa]	E_n
10	0.690
1	0.036

As the E_n statistic is less than 1 in both cases, the test results show that there is comparability between the pressure calculated in the static expansion system and the pressure reported by the pressure measuring instrument with traceability to CENAM.

4.3. Uncertainty budget analysis

Four consecutive expansions were performed with the system, and a pressure gauge was calibrated at the four pressure values obtained. At each of the pressure values, the relative percentage contribution of each input quantity to the uncertainty of the calibration result, which was the difference between the static expansion and values yielded by the unit under test pressure, was determined. Since the expansions are consecutive, the final pressure of each expansion becomes the initial pressure of the next expansion process. The results of the four calibrations are presented in Table 4. Table 5 shows the uncertainty budgets for each calibration.

Table 4. Calibration result in each expansion process and their uncertainties.

	First expansion	**Second expansion**	**Third expansion**	**Fourth expansion**
Final pressure of the expansion [Pa]	3 115	192.9	12.1	1.0
Pressure of the unit under test [Pa]	3 097	185.7	8.6	0.9
ΔP [Pa]	–18	–7.2	–3.5	–0.1
Standard uncertainty of ΔP [Pa]	23	2.1	1.0	1.0

Table 5. Uncertainty budgets for the four expansion processes.

Input quantity	**Relative percentage contribution to uncertainty [%]**			
	First expansion	**Second expansion**	**Third expansion**	**Fourth expansion**
P_{UUC} [Pa]	31.03	58.27	97.76	98.33
P_i [Pa]	49.57	32.35	1.70	1.22
$P_{i,G}$ [Pa]	0.00	0.34	0.40	0.46
V_P [m^3]	0.30	0.14	0.00	0.00
V_G [m^3]	8.83	4.11	0.06	0.00
T_i [K]	5.14	2.40	0.04	0.00
T_f [K]	5.14	2.40	0.04	0.00

The results indicate that the most important sources of uncertainty in calibration using the static expansion system are the pressure of the unit under calibration and the initial pressure in the small tank. In the tests performed, the pressure of the unit under calibration has the greatest influence on the calibration uncertainty at the lowest pressure values, due to the performance of the gauge used. At higher pressures, which correspond to the first expansions, it is the initial pressure in the small tank which dominates the budget. Because the initial pressure in the large tank was considered in the measurement model, some effect of this input magnitude on the calibration uncertainty for the lower pressures can be seen, although the impact is quite limited.

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In this example, the effects of the uncertainties of the tank volumes are relatively small, thanks to the gravimetric calibration of the tanks. As for the temperature, it can be seen that as the amount of substance in the system is lower (*i.e.*, the pressure decreases), its effect on the calibration uncertainty is smaller.

Considering that in the calibrations there are input quantities which dominate the uncertainty budget, particularly at lower pressures, and that this is one of the reasons that can invalidate the assumptions of the GUM method, it was decided to estimate the uncertainty of the calibrations using Monte Carlo simulation, to evaluate the adequacy of the GUM method in the current situation. u_{V_P} and u_{V_G} were assigned normal distributions. Repeatability uncertainties of the different input quantities were assigned t-distributions. For the other sources of variability of the input quantities, uniform continuous distributions were used. The results are presented in Fig. 3.

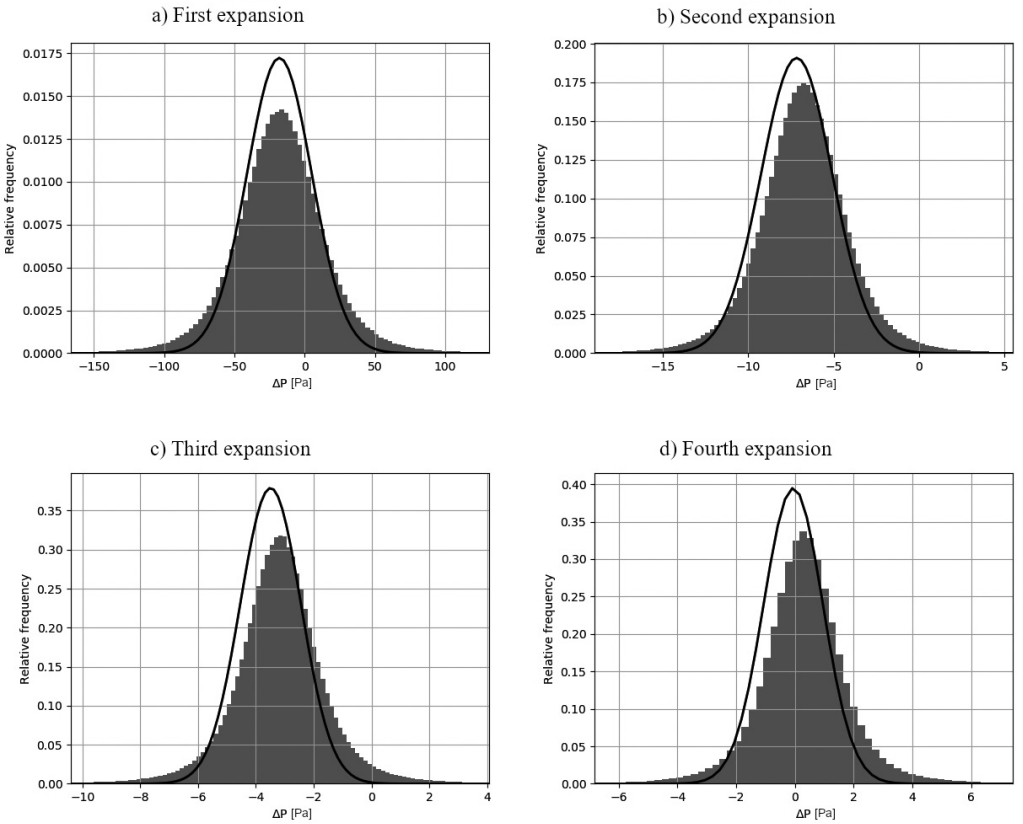

Fig. 3. Comparison between the uncertainties estimated by the GUM method and the Monte Carlo simulation for the four calibration points performed. The black line represents the distribution estimated by the GUM method. The gray histograms are the result of the Monte Carlo simulation.

The results show that the Monte Carlo simulation yields probability distributions with higher standard deviations (*i.e.*, standard uncertainties) for the calibration results. Additionally, it is seen that as the importance of the unit under calibration pressure in the uncertainty budget increases, the Monte Carlo simulation predicts an average value of the distribution of the measurement result which deviates from the value predicted by the GUM method. Considering that Monte

Carlo simulation yields a result consistent with the measurement model, regardless of its degree of nonlinearity or the dominance of the uncertainty of an input quantity [19], it is concluded that a Monte Carlo method, or a GUM method with higher order terms, should be used to estimate the calibration uncertainty.

5. Conclusions

A static expansion system was characterized to calibrate pressure gauges in medium and high vacuum. The desorption rate and pressure stability were evaluated, evidencing the effect of bakeout on the final pressure stability. A trueness test was performed, and it was concluded that the pressure determined by the static expansion system is comparable to that reported by a pressure gauge with guaranteed metrological traceability. Finally, the uncertainty budget obtained by using the static expansion system to calibrate a pressure gauge under real operating conditions was analyzed. The budget indicated the possibility that the GUM method was not suitable for estimating the uncertainty of the calibration results with this static expansion system. A Monte Carlo simulation was applied and compared against the results of the GUM method, and it became apparent that the GUM method is not ideal for estimating the uncertainty in this case.

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