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Realization of the medium and high vacuum primary standard in CENAM, Mexico

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Abstract
A medium and high vacuum primary standard, based on the static expansion method, has been set up at Centro Nacional de Metrología (CENAM), Mexico. This system has four volumes and covers a measuring range of 1×10^{-5} Pa to 1×10^{3} Pa of absolute pressure. As part of its realization, a characterization was performed, which included volume calibrations, several tests and a bilateral key comparison. To determine the expansion ratios, two methods were applied: the gravimetric method and the method with a linearized spinning rotor gauge. The outgassing ratios for the whole system were also determined. A comparison was performed with Physikalisch-Technische Bundesanstalt (comparison SIM-Euromet.M.P-BK3). By means of this comparison, a link has been achieved with the Euromet comparison (Euromet.M.P-K1.b). As a result, it is concluded that the value obtained at CENAM is equivalent to the Euromet reference value, and therefore the design, construction and operation of CENAM's SEE-1 vacuum primary standard were successful.

1. Introduction

The Centro Nacional de Metrología (CENAM), the Mexican National Metrology Institute, designed a static expansion system as a vacuum primary standard. This was developed in a project within the framework of a technical cooperation between Germany and Mexico. The vacuum section from the Physikalisch-Technische Bundesanstalt (PTB), Germany, helped CENAM in the establishment of this primary standard. The measurement range for this newly established system is 10^3 Pa down to 10^{-5} Pa.

1.1. Measurement principle

The primary standard realizes the static expansion method [1], by which gas is introduced into a previously evacuated volume V_0 up to a pressure p_0, high enough to be measured with high accuracy. After measuring p_0, the gas is expanded into a previously evacuated volume V_f, which is much larger than V_0.

The pressure p_0 will be reduced by the volume ratio between the initial and final volumes, which can be calculated from

$$f = \frac{V_0}{V_0 + V_f}. \qquad (1)$$

When the ideal gas law is applied, including a first order approximation for real gas behaviour, the pressure in the calibration volume is determined from equation (2):

$$p_f = p_0 f \frac{T_f}{T_0} \frac{1 + B_f p_f/(RT_f)}{1 + B_0 p_0/(RT_0)}, \qquad (2)$$

where T_0 is the gas temperature in the initial volume, T_f the gas temperature in the final volume, $R = 8314$ Pa L (mol K)$^{-1}$ and $B_{0,f}$ the virial gas coefficients at the conditions in the initial and final volume.

1.2. System description

The Mexican static expansion system (SEE-1) consists of four volumes (see figure 1). The four volumes as described in

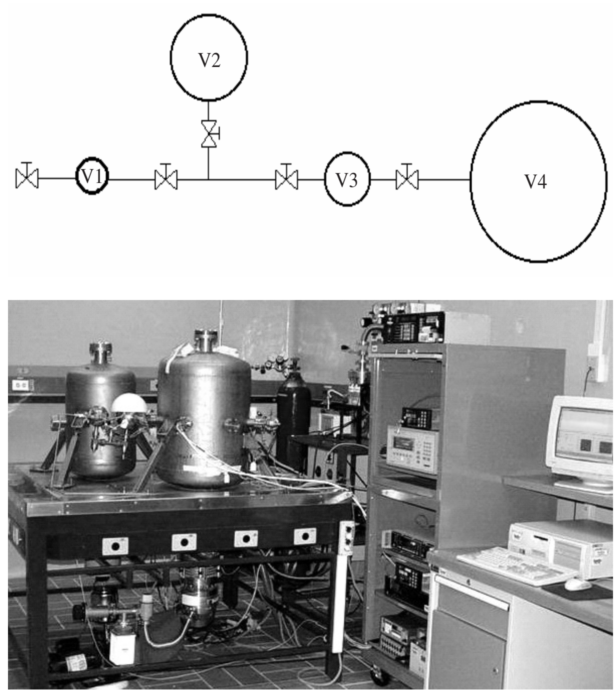

Figure 1. Mexican static expansion system, SEE-1: schematic and photograph.

Table 1. SEE-1 volumes with their nominal and measured volumes, and their standard uncertainties in litres.

Identification	Nominal volume/L	Measured volume/L	Uncertainty/L
V_1	0.5	0.520 43	±0.000 12
V_2	50	51.056 7	±0.007 2
V_3	1	0.992 68	±0.000 12
V_4	100	96.881	±0.016

table 1 are used to obtain different expansion paths. The calibration volume is V_4, to which the units under calibration are connected. The four volumes allow various expansion paths which are listed in table 2. V_x is the volume occupied by the fittings and valves between the volumes.

Various pressure ranges can be obtained with the SEE-1 by combining the expansion paths, as shown in table 3.

2. Expansion ratio determination

The expansion ratios are the most important parameters in any static expansion system and have to be determined with high accuracy. The SEE-1 expansion ratios were determined by two different methods as follows.

2.1. Gravimetric method

By the gravimetric method, the unknown volume is first measured empty and then filled with distilled water. The mass of distilled water is measured and, from its density at the measured water temperature, the volume occupied by water can be determined.

$$V = \frac{m_{\mathrm{H_2O}}}{\rho_{\mathrm{H_2O}}}\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{std\ mass}}}\right)\left(1 - \frac{\rho_{\mathrm{air}}}{\rho_{\mathrm{H_2O}}}\right)^{-1}(1 - \alpha(t - 20)). \tag{3}$$

The weighting process is repeated typically ten times [3–5]. The volume values and expansion ratios are listed in tables 1 and 2, respectively.

2.2. Spinning rotor gauge method

The spinning rotor gauge (SRG) method is performed by evacuating the system and determining the offset of the SRG. Then, an initial pressure is established in the initial volume (V_0). An expansion into the final volume (V_f) is performed. Once the expansion has been done, the deceleration ratio is measured (DCR_1) and the final pressure at the volume V_0 is measured. The volume V_f is evacuated again and the retained gas at volume V_0 is expanded; the deceleration ratio reading is taken (DCR_2). Under isothermal conditions and with a linearized deceleration ratio $\mathrm{DCR}_2/\mathrm{DCR}_1$ the expansion ratio can be determined as described in [6]. Table 2 shows the values found for the expansion paths by the SRG method.

2.3. Difference in gravimetric and SRG methods

The maximum relative difference between the two methods was below -0.005. Table 4 shows the difference between the two methods for each expansion path.

The average values between the two methods (given in table 2) were chosen as the working values. The uncertainty is the combined uncertainty of the two methods plus their difference (as a rectangular distribution).

3. SEE-1 uncertainty budget

In static expansion systems, the system's residual pressure is the main cause for the lower end range limit. This lower end pressure is related directly to the system outgassing. In the SEE-1 the residual pressure, after a bake-out at 300 °C for 48 h, is 10^{-8} Pa, with a pressure increase of 4.81×10^{-11} Pa s^{-1}. This corresponds to a specific outgassing of 4.05×10^{-13} Pa L s^{-1} cm^{-2}. The pressure rise means that, within the time scale of a calibration (5 min), the residual pressure rise will be 1.44×10^{-8} Pa. This is 0.144% of the lowest calibration pressure 1×10^{-5} Pa.

Table 2. Description of expansion paths.

Identification	Expansion path	Value ± uncertainty gravimetric	Value ± uncertainty SRG method	Value used
f_A	$V_1 \to V_1 + V_x + V_2$	$0.010\,0465 \pm 2.7 \times 10^{-6}$	$0.010\,0927 \pm 3.0 \times 10^{-6}$	$0.010\,069 \pm 4.6 \times 10^{-5}$
f_B	$V_1 \to V_1 + V_x + V_2 + V_3$	$0.009\,8576 \pm 2.6 \times 10^{-6}$	$0.009\,8581 \pm 2.9 \times 10^{-6}$	$0.009\,8578 \pm 3.9 \times 10^{-6}$
f_C	$V_3 \to V_3 + V_4$	$0.010\,0989 \pm 2.0 \times 10^{-6}$	$0.010\,0943 \pm 3.0 \times 10^{-6}$	$0.010\,0966 \pm 5.9 \times 10^{-6}$

Table 3. Expansion paths required according to the nominal pressure range.

Nominal pressure range/Pa	Expansion paths
10^{-5} to 10^{-3}	f_A, f_A, f_B, f_C
10^{-3} to 10^{-1}	f_A, f_B, f_C
10^{-1} to 10^{1}	f_B, f_C
10^{1} to 10^{3}	f_C

Table 4. Comparison of the expansion ratio found by the two different methods. The difference is equal to the value determined by the gravimetric method minus the value determined by the SRG method (see table 2). For the relative difference this value is divided by the gravimetrically determined value.

Expansion path	Difference	Relative difference
f_A	-4.62×10^{-5}	-4.60×10^{-3}
f_B	-5.0×10^{-7}	-5.07×10^{-5}
f_C	4.6×10^{-6}	4.55×10^{-4}

A typical uncertainty budget for the SEE-1 is shown in table 5.

For table 5,

$$p_f = p_0 f \left(\frac{T_f}{T_0}\right) \mathrm{cor} + p_{\mathrm{res}}, \tag{4}$$

and

$$\mathrm{cor} = \frac{1 + B_f(p_0 f/RT_f)}{1 + B_0(p_0/RT_0)}. \tag{5}$$

4. Measurement validation

A comparison between PTB and CENAM was performed with the purpose of identifying possible deviations of the generated pressure in SEE-1 against the internationally validated standard of PTB and of checking the assigned uncertainties of the pressures in SEE-1. This comparison [7] was performed according to the CIPM guidelines and was assigned by SIM-Euromet.M.P-BK3. It links in the whole range (3×10^{-4} Pa to 0.9 Pa) to the Euromet.M.P-K1.b comparison, where the same transfer standards and the same procedures were used [8]. For the value at 0.9 Pa, it also links to the key comparison CCM.P-K4, in which PTB participated. Therefore, the degree of equivalence of CENAM's SEE-1 standard could be determined against the Euromet standards that took part in Euromet.M.P-K1.b and to the international standards at 0.9 Pa that were compared in the CCM.P-K4.

The comparison consisted in the determination of the accommodation coefficients σ of two SRGs and their respective uncertainties at eight target pressure points (3×10^{-4} Pa, 9×10^{-4} Pa, 3×10^{-3} Pa, 9×10^{-3} Pa, 3×10^{-2} Pa, 9×10^{-2} Pa, 3×10^{-1} Pa, 9×10^{-1} Pa). Assuming stability of the transfer standards the generated pressures in the two standards could be compared. The stability was checked by two measurements at PTB before and after transportation to CENAM (hand-carried in both directions). The results of these measurements were compatible with the assumption that the transfer standards did not change their σ values due to the transportation.

It appeared that for the same calculated pressures the CENAM generated pressures were about 0.3% higher than the pressures generated at PTB (the values of σ were lower). Figure 2 shows the accommodation coefficients corresponding values for the SRGs at each measuring point. Figure 3 shows the relative pressure differences between CENAM and PTB primary standards.

The CENAM primary standard SEE-1 was equivalent ($E_n < 1$) to the EUROMET reference values over the whole pressure range compared. In most cases $E_n < 0.5$. E_n was calculated from

$$E_n = \frac{p_{\mathrm{CENAM}} - p_{\mathrm{Eur}}}{2\sqrt{u^2(p_{\mathrm{CENAM}}) + u^2(p_{\mathrm{Eur}})}}, \tag{6}$$

where p_{Eur} and $u(p_{\mathrm{Eur}})$ are the EUROMET reference value and its standard uncertainty, respectively.

The CENAM primary standard SEE-1 was also equivalent to the CCM reference value at 0.9 Pa.

5. Conclusions

CENAM has established an internationally validated vacuum primary standard for the calibration of vacuum gauges. The system is shown in figure 1.

As from the end of year 2004, the SEE-1 started to serve as a link to other SIM national laboratories to verify the compatibility of their measurements.

Table 5. Example of SEE-1 uncertainty budget at a calibration pressure p_f of 0.9 Pa.

	Influence quantity		Standard uncertainty	Degrees of freedom	Sensitivity coefficients		Associated covariance	100× impact
	Value	SI unit	$u(x_i)$	v_i	Equation $\partial p/\partial x_i$	Value	$\partial p/\partial x_i \cdot u(x_i)/$ Pa	$(\partial p/\partial x_i \cdot u(x_i))^2/(u_c(p))^2$
p_0	9060	Pa	0.679 5	50	$f(T_f/T_0)$cor	9.952×10^{-5}	0.000 0676	0.30
f	9.9496×10^{-5}	—	$1.092\,5 \times 10^{-7}$	50	$p_0(T_f/T_0)$cor	9062.1697	0.000 9901	57.0
T_0	294.44	K	0.19	5	$p_0 f(1/T_0)$cor	0.003 0615	0.000 5817	19.70
T_f	294.51	K	0.19	5	$p_0 f(T_f/T_0^2)$cor	0.003 0623	0.000 5818	19.70
cor	1.000 0017		0.000 266	5	$p_0 f(T_f/T_0)$	0.901 6491	0.000 2399	3.3
p_{res}	1×10^{-8}	Pa	1×10^{-9}	5	1	1	1×10^{-9}	0.0
p_f	0.901 651	Pa			Combined standard uncertainty $u_c(p) =$		0.001 31	100

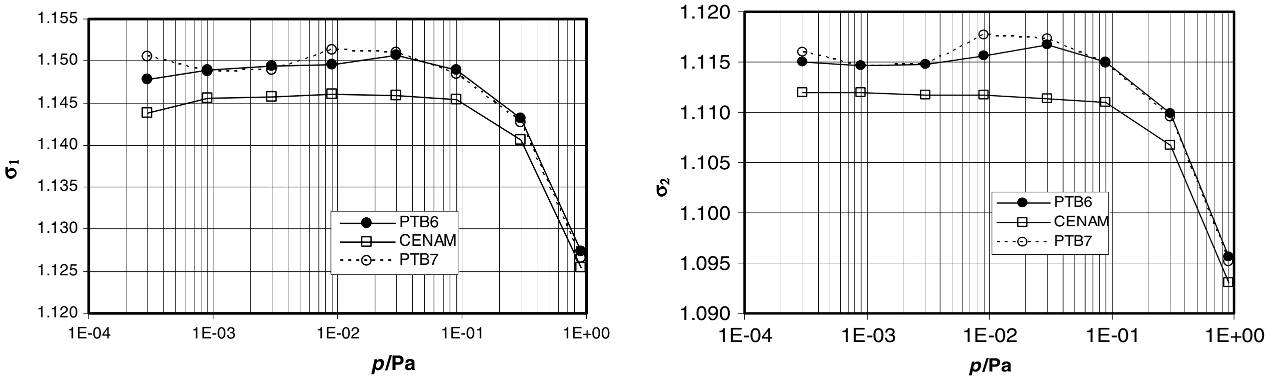

Figure 2. Results of accommodation coefficient measurements for rotor 1 and rotor 2 for the comparison with SIM-Euromet.M.P-BK3.

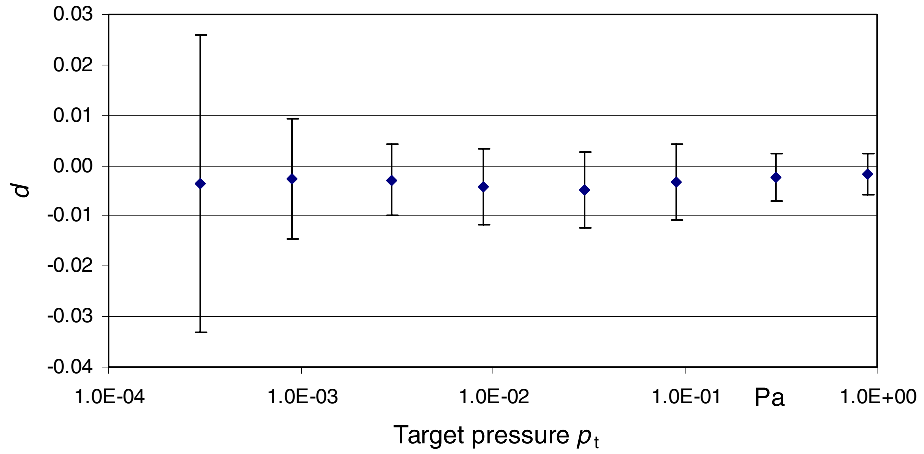

Figure 3. The relative difference $d = (p_{\mathrm{CENAM}}/p_{\mathrm{PTB}}) - 1$, for the two pressures generated in the two primary standards, as a function of the target pressures in this comparison. Overlap of uncertainty bar with $d = 0$ means equivalence of the two standards.

(This figure is in colour only in the electronic version)

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