CHARACTERIZATION OF THE MEDIUM AND HIGH VACUUM PRIMARY STANDARD AT CENAM, MEXICO

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Abstract. Static expansion systems are used to generate accurately known pressures in the high and medium vacuum range for vacuum gauge calibration. The SEE-1 is the primary standard for vacuum at the Centro Nacional de Metrología, this system has four volumes, it's measurement range is from 10^{-4} Pa to 10^3 Pa. This article describe a new characterization of the system, basically the determination of the expansion ratios by two methods: the gas accumulation method and the method with a linearized spinning rotor gauge (SRG).

1. INTRODUCTION

The increasing request for high accuracy calibration services from the Mexican industry lead to the establishment of a primary standard for vacuum at The Centro Nacional de Metrología (CENAM), the National Metrology Institute of Mexico. The measurement range for this system is from 10^{-4} Pa up to 10^3 Pa [1]. The system has been in operation for more than 6 years. In order to optimize the system and obtain its best measurement and calibration capabilities a series of improvements have been carried out in the system. Also, a new characterization of the system has been performed. This paper presents the new characterization.

1.1 Measurement principle

Gas is introduced in 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 to a previously evacuated volume V_f, which is bigger than V_0. For an ideal gas under isothermal expansion conditions, since the amount of gas remains the same, the Boyle law applies such that:

$$P_0 \cdot V_0 = P_f \cdot V_f \qquad (1)$$

The pressure at the calibration volume can be determined from:

$$P_f = P_0 \cdot \frac{V_0}{V_0 + V_f} \qquad (2)$$

This pressure change depends on the volume ratio between the initial and final volumes, which can be calculated from:

$$f = \frac{V_0}{V_0 + V_f} \qquad (3)$$

Since ideal gases behavior is not possible to obtain, the real generated pressure has to be obtained including a correction due to this effect [2]. Then, the calibration volume pressure is determined from:

$$P_f = P_0 \cdot f \cdot \frac{T_f}{T_0} \cdot \frac{1 + B_f \cdot P_f / \left(R \cdot T_f \right)}{1 + B_0 \cdot P_0 / \left(R \cdot T_0 \right)} \qquad (4)$$

Where, T_0 is the gas initial thermodynamic temperature, T_f the gas final thermodynamic temperature, R = 8 314 Pa·L/(mol·K) and $B_{0,f}$ the viral gas coefficients.

1.2 System description

The static expansion system has 4 volumes, as described in table 1. These volumes are used to obtain different expansion paths. Figures 1 and 2 show the static expansion system (SEE-1).

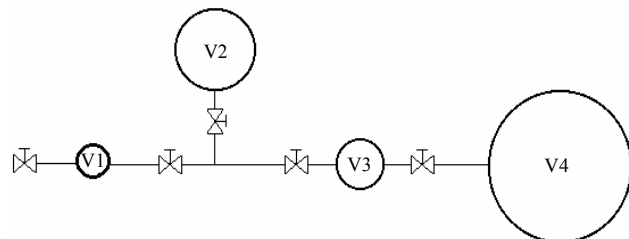| Identification	Nominal volume
V_1	0.5 L
V_2	50 L
V_3	1 L
V_4	100 L

Table 1. *SEE-1 volumes.*

Figure 1. *SEE-1 diagram.*

Figure 2. *Static expansion system at CENAM.*

In the SEE-1 the calibration chamber is V_4. It is possible to perform various expansions before the calibration pressure is achieved. Table 2 shows the different expansion paths in the SEE-1.

Identification	Expansion path
f_A	$V_1 \rightarrow V_1 + V_x + V_2$
f_B	$V_1 \rightarrow V_1 + V_x + V_2 + V_3$
f_C	$V_3 \rightarrow V_3 + V_4$

Table 2. *Expansion paths identification.*

The expansion paths can be combined. Table 3 shows the pressure ranges which can be obtained by the SEE-1.

Pressure range	Expansion paths
10^{-5} Pa a 10^{-3} Pa	f_A, f_A, f_B, f_C
10^{-3} Pa a 10^{-1} Pa	f_A, f_B, f_C
10^{-1} Pa a 10^{1} Pa	f_B, f_C
10^{1} Pa a 10^{3} Pa	f_C

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

2. EXPANSION RATIO DETERMINATION

The expansion ratios are very important parameters in the static expansion systems and have to be determined with high accuracy.

The SEE-1 expansion ratios were determined as part of the characterization by 2 different methods, the gas accumulation method and the SRG method.

a) The gas accumulation method consists in the realization of successive expansions from V_0 to V_f. With the system evacuated, a pressure of 100 kPa is established in V_0 using nitrogen as media; then the gas is expanded into the evacuated V_f, the valve between V_0 and V_f is closed. The pressure of 100 kPa is re-established in V_0 and the gas is expanded again, these expansions are repeated until the

pressure in V_f can be measured with good accuracy. After N expansions under isothermal conditions the expansion ratio is:

$$\overline{\qquad} \qquad (5)$$

b) SRG Method. For this method a spinning rotor gauge is used to measure the pressure, both before and after the gas expansion. Firstly, the SRG offset is determined, the initial pressure is established in V_0 then the gas is expanded into the evacuated V_f; the deceleration rate DCR1 is measured. Then, the valve between V_0 and V_f is closed, V_f is evacuated until it reaches similar values of the offset, the gas retained in V_0 is again expanded and the deceleration DCR2 is measured. Under isothermal conditions and without a linearization of the deceleration rate, the expansion ratio can be determinate from:

$$\overline{\qquad\qquad} \qquad (6)$$

The differences between the two methods is analyzed and included as part of the characterization.

3. RESULTS OF THE CHARACTERIZATION

3.1 SEE-1 degasification

An important limiting factor in the low end of the measurement range of an static expansion system is the degasification. For its quantification a preheat to 200 °C for 36 hours in the system has to be performed at a very low pressure, 10^{-6} Pa, and from there it must be measured the pressure increment in the system with time. The result of this measures are shown in table 4.

Volume	$\Delta P \ s^{-1}$	Specific outgassing	Δp / 5 min
	Pa s^{-1}	Pa·L·s^{-1}·cm^{-2}	Pa / 5 min
V_4	1.26E-08	1.05E-10	3.78E-6
$V_4 + V_3$	1.31E-08	1.06E-10	3.93E-6
V_2	1.01E-08	6.61E-11	3.03E-6
$V_2 + V_1$	1.12E-08	7.10E-11	3.35E-6
$V_2 + V_1 + V_3$	1.36E-08	8.32E-11	4.08E-6

Table 4. *Degasification results.*

The maximum pressure increase is 1.36E-8 Pa·s^{-1}, which corresponds to a specific outgassing of 8.32E-11 Pa·L·s^{-1}·cm^{-2}.

The lowest calibration pressure which can be reached at the SEE-1 is 10^{-4} Pa and the pressure rise for this pressure after 5 minutes is 4.08E-6 Pa, this value corresponds to the 4.08% of 10^{-4}.

3.2 Expansions ratios

The three expansions ratios of the SEE-1 were determined using the gas accumulation [7] and the SRG method [6]. For the gas accumulation method 2 temperature sensors were installed in V_0 and 8 in V_f. In V_f a recently calibrated absolute manometer with a maximum range of 100 kPa was connected. To establish the pressure in V_0 a pressure controller with a range of 200 Pa was used. Firstly, V_f was evacuated, a pressure of 100 kPa was established in V_0 with the pressure controller and its temperature was recorded. Then, the nitrogen was expanded to V_f, after the pressure relaxation the temperature of the sensor on V_f were read and the reading of the absolute manometer taken. For the next expansion the valve between V_0 and V_f was closed and the same expansion was repeated 23 times until the pressure in the absolute manometer indicated around 20 kPa. The expansion ratio was calculated with the next equation:

$$f = 1 - \left(1 + f \cdot \alpha - \frac{P_{fN}}{P_0}\right)^N \quad (7)$$

Where N is the number of expansions to reach the pressure P_{fN} with a pressure P_0 of 100 kPa. α is a factor to correct the temperature gradients between the two volumes and the temperatures changes over time.

$$\alpha = \sum_{i=1}^{N}(1-f)^{i-1}\frac{(T_f)_N - (T_0)_{1+N-i}}{(T_0)_{1+N-i}} \quad (8)$$

To solve equation (7) was necessary to apply an iterative method. Firstly, f was calculated without the temperature correction α, and this value of f was used to calculate α. With these values f was calculated by means of (7), and with this new value of f equation (8) was solved. This calculations were repeated until equation (7) was solved adequately. Equation (7) is valid for an ideal gas, the possible deviations from the ideal gas were corrected with the next equation:

$$f_{corr} = f(1 - B \cdot \rho_m) \quad (9)$$

Where B is the nitrogen second virial coefficient and ρ_m is the gas molar density before the expansion.

$$B = -131.21 + 0.651\,25 \cdot T - 7.636 * 10^{-4} \cdot T^2 \quad (10)$$

$$\rho_m = \frac{P}{R \cdot T} \quad (11)$$

Is necessary to apply other correction to eliminate the additional volume V_{ad} due to the test gauges and its connections to V_f.

$$f_{real} = \cfrac{1}{\cfrac{1}{f_{corr}} + \cfrac{V_{ad}}{V_s}} \quad (12)$$

The SRG method was applied to calculate the expansion ratios. Before the measurements, the SRG offset was determined. For the measures of DCR1 a pressure between 0.4 Pa and 14 Pa was established in V_0 and for the measures of DCR2 a pressure between 4E-3 Pa and 0.14 Pa. Equation (6) is valid under isothermal conditions, to correct the temperature differences is used the equation:

$$f = \left[\left(\frac{DCR1 - OFF}{DCR2 - OFF}\right)\left(\frac{T_f}{T_0}\right)\right]^{-1} \quad (13)$$

Where T_0 y T_f are the temperature in V_0 and V_f respectively. The no linear deceleration with the pressure was corrected by a second order polynomial [6]. In table 5 the results obtained for the gas accumulation method, SRG method and actual values used for each expansion ratio are shown.

Expansion ratio	Gas accumulation method	SRG method	Determined in 2010
f_A	0.010 032 4 ±4.1E-06	0.010 083 5 ±4.4E-06	0.010 058 0 ±5.1E-05
f_B	0.009 844 9 ±4.1E-06	0.009 874 9 ±4.6E-06	0.009 859 9 ±3.1E-05
f_C	0.010 095 7 ±4.1E-06	0.010 133 2 ±4.0E-06	0.010 114 5 ±3.8E-05

***Table 5.** Results from the two methods.*

Table 6 shows the difference between the two methods expressed in absolute and relative values.

Expansion ratio	Absolute difference	Relative difference
f_A	-5.1E-05	-5.1E-03
f_B	-3.0E-05	-3.0E-03
f_C	-3.7E-05	-3.7E-03

***Table 6.** Comparison between the two methods.*

The results obtained when the SEE-1 was established [1] are presented in the table 7. At that time, the expansion ratios were estimated using the gravimetric [3] and the SRG method [4], [6].

Expansion ratio	Gravimetric	SRG	Used at initial operations
f_A	0.010 046 5 ±2.7E-06	0.010 092 7 ±3.0E-06	0.010 069 0 ±4.6E-05
f_B	0.009 857 6 ±2.6E-06	0.009 858 1 ±2.9E-06	0.009 857 8 ±3.9E-06
f_C	0.010 098 9 ±2.0E-06	0.010 094 3 ±3.0E-06	0.010 096 6 ±5.9E-06

Table 7. Expansion ratios estimated at the time of the SEE-1 initial operation.

To compare those results with the new ones, table 8 shows the difference in absolute and relative values.

Expansion ratio	Absolute difference	Relative difference
f_A	-1.16E-05	-1.15E-03
f_B	2.03E-06	2.06E-04
f_C	1.78E-05	1.77E-03

Table 8. Comparison between initial and actual values.

4. MEASUREMENTS ASSURANCE

The results of this characterization were compared with information from a comparison carried out with the PTB´s vacuum laboratory (Germany) at the time of the first operation of the SEE-1 [5]. See table 9.

The last column presents the normalized error equation values (En) for each applied pressure. The criteria of the normalized error equation is that any value equal or minor to 1 is compatible, and any value above should be reviewed or is incompatible.

Nominal pressure	Original values	Original uncertainty	New values	New uncertainty	En
3.00E-04	3.031E-04	2.8E-06	3.030E-04	3.2E-06	0.02
9.00E-04	9.085E-04	8.4E-06	9.082E-04	9.7E-06	0.02
3.00E-03	2.997E-03	1.5E-05	2.999E-03	1.8E-05	-0.10
9.00E-03	9.008E-03	4.3E-05	9.015E-03	5.5E-05	-0.11
3.00E-02	3.000E-02	1.4E-04	3.003E-02	1.8E-04	-0.11
9.00E-02	9.003E-02	4.2E-04	9.011E-02	5.4E-04	-0.11
3.00E-01	2.971E-01	4.4E-04	2.977E-01	1.0E-03	-0.53
9.00E-01	8.935E-01	9.3E-04	8.953E-01	2.9E-03	-0.58

Table 9. Comparison of the values of the SEE-1 with the values from PTB.

5. CONCLUSIONS

The improvements carried out at the static expansion system and the characterization of the system, probe to be adequate and permit to maintain a calibration standard with the most up to date characteristics in Mexico.

The values obtained by means of the normalized error equation on the comparison of the SEE-1 original values (used for the comparison with PTB) and the new values do not exceed 1. Also, 6 out of 8 values are around 0.1, which is a very good result.

ACKNOWLEDGEMENTS.

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