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SIM VACUUM COMPARISON FROM 13 Pa UP TO 1,33 kPa

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Abstract. This comparison constitutes the first vacuum comparison among National Metrology Institutes (NMI) within the Interamerican Metrology System (SIM). Each laboratory used its national standards. The Centro Nacional de Metrología (CENAM, Mexico) used a primary standard (static expansion system type) and the Instituto Nacional de Metrologia, Normalização e Qualidade Industrial (INMETRO, Brazil) used a secondary standard (comparison system type). CENAM had the role of coordinator and pilot laboratory. The comparison started in December 2004 and finished in March 2005. The objective of the comparison was to estimate the level of agreement for the realization of the quantity and the uncertainty associated to its measurement.

One transducer (Capacitive Diaphragm Gauge) was used as transfer standard; the comparison range was selected from 13 Pa to 1,33 kPa. The relevant aspects of the measurement protocol are summarized in the paper but were widely developed in the comparison reference documents. The gas used for the comparison was nitrogen. The measurements started at the lowest pressure and the pressure was increased up to the maximum range. The analysis of measurements comparability between the laboratories is included here. This bilateral comparison has been entered within the SIM data base as a pilot comparison SIM.7.26 P.

Keywords: Comparison, vacuum, capacitive diaphragm gauge, static expansion system, SIM.

1. INTRODUCTION

In the frame of the technical cooperation between the Centro Nacional de Metrología (CENAM, Mexico) and the Instituto Nacional de Metrologia, Normalização e Qualidade Industrial (INMETRO, Brazil) and within the Sistema Interamericano de Metrologia (Interamerican Metrology System, SIM) [1 and 2], the first vacuum comparison was performed

A capacitive diaphragm gauge (CDG) was used as transfer standard for the comparison. The pressure target points measured were the following: 13 Pa, 40 Pa, 133 Pa, 400 Pa, 1 330 Pa.

In addition to the quantity being measured, an important value for the determination of the degree of equivalence is the uncertainty of the generated pressure in the reference standard used by each laboratory. This value was considered the responsibility of each participant laboratory and had to be reported as part of the comparison final results [3, 4 and 5]. CENAM had the role of coordinator and pilot laboratory. The comparison started in December 2004 and finished in March 2005.

2. SCOPE OF WORK

The objective of this comparison was to determine the degree of equivalence between the measurements in absolute pressure (vacuum) performed by CENAM and those by INMETRO in the medium and low vacuum range from 13 Pa to 1,33 kPa (0,1 torr to 10 torr), using the normalized error equation as the equivalence parameter [6 and 7].

This bilateral comparison has been entered within the data base of the Sistema Interamericano de Metrología (Interamerican Metrology System, SIM) as a pilot comparison SIM.7.26 P.

2.1 Transfer standard (TS)

A CDG was used as transfer standard for the comparison. Each laboratory used its controller to operate the CDG. The characteristics of the CDG, according to the manufacturer, are included in Table 1.

Table 1. Transfer standard data.

Transducer Type:	Capacitive Diaphragm Gauge
Range:	0,1 torr to 100 torr (13 Pa to 13,3 kPa)
Units:	torr
Accuracy Class:	0,05% of the Reading
Manufacturer:	MKS
Model:	690A12TRA
Serial number:	96143181A
Code number	MN003

2.2 Comparison dates

The dates of the calibrations performed by the laboratories are shown in Table 2.

Table 2. Dates of the calibrations by the NMIs.

National Metrology Institute	*Calibration date*	*Standard used*
INMETRO	2004-12-03	Capacitive Diaphragm Gauge
CENAM	2004-12-13	Static Expansion System
INMETRO	2005-01-14	Capacitive Diaphragm Gauge

2.3 General Guidelines and Procedure

The following main calibration considerations were agreed upon before the comparison:

a) Each laboratory was to calibrate the CDG at the following 5 nominal target pressures (for nitrogen pressure) in ascending order: 13 Pa, 40 Pa, 133 Pa, 400 Pa, 1 330 Pa.
b) Each target pressure had to be generated at least 3 times. This meant that after a measurement at the target point, the system was pumped down to residual pressure conditions and the same point re-generated. In total 5 × 3 = 15 points were measured and were considered as one calibration sequence.

3. PARTICIPATING LABORATORIES' STANDARDS

Table 3 lists the two participating laboratories and characteristics of their reference standards.

Table 3. Participating laboratories' standards.

Laboratory:	CENAM	INMETRO
Standard Type:	Static Expansion System	Capacitive Diaphragm Gauge
Character of standard:	Primary	Secondary
Traceable to:	CENAM	PTB
CMCs listed in the BIPM data file:	No. Only the secondary standards are listed	Yes

The CENAM's pressure standard (SEE-1) is based on the static expansion method (also called, serial expansion method). The SEE-1 system is used to measure medium and high vacuum within the range from 1 x 10^{-5} Pa and up to 1 x 10^{3} Pa in absolute pressure [3 and 5]. It consists of 4 known volumes, two small volumes of nominally 0,5 L and 1 L and two expansion chambers, 50 L and 100 L, nominally. With this primary standard the Boyle–Mariotte law is applied: a known small volume of gas at a known relatively high pressure is expanded into a previously evacuated larger volume (under isothermal conditions). This generates a pressure drop which depends on the initial and final volumes' ratio.

The CENAM's pressure standard was subjected to a peer review by PTB staff and participated successfully in a bilateral comparison with PTB [4], according to the guidelines of BIPM. This comparison was later listed as key comparison SIMEuromet.M.P-BK3 in the BIPM data base. The key comparison SIM-Euromet.M.P-BK3 was directly connected to and followed the same procedure and used the same transfer standards as the comparison Euromet.M.P-K1.b [8].

INMETRO's pressure standard is based on the comparison method. The system is used to measure low and medium vacuum within the range from 1 x 10^{-4} Pa up to 1 x 10^{5} Pa in absolute pressure. It consists of two capacitive diaphragm gauges, one having a range up to 1,3 x 10^{3} Pa and another with a range up to 1,3 x 10^{5} Pa.

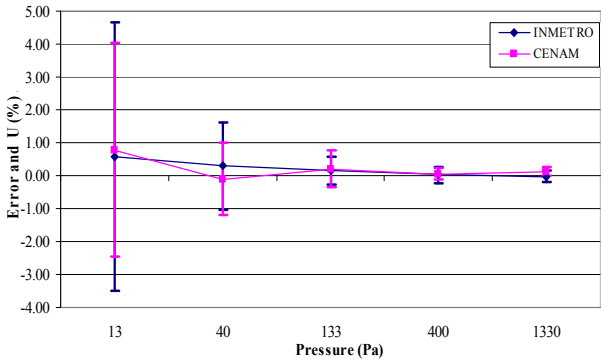
4. RESULTS

Table 4 shows the summary of the measurements made by CENAM and INMETRO, for the calibration of the TS.

Table 4. Summary of measurements results.

	INMETRO		CENAM	
Pressure Pa	Error %	%U $k = 2$	Error %	%U $k = 2$
13	0,56	4,1	0,78	3,3
40	0,31	1,4	-0,10	1,1
133	0,16	0,43	0,21	0,55
400	0,02	0,24	0,05	0,18
1330	-0,03	0,18	0,13	0,14

Graph 1 shows the errors found by each laboratory and its corresponding uncertainty for each target pressure.



Graph 1. INMETRO and CENAM measurements results. The least squares best-fit lines have been superimposed over each laboratory's measurement results.

5. DISCUSSION

The degree of equivalence between the results of the measurements made by both laboratories was evaluated using the normalized error according to Equation 1.

$$e_n = \left| \frac{E_{INMETRO} - E_{CENAM}}{\sqrt{(U_{INMETRO})^2 + (U_{CENAM})^2}} \right| \quad (1)$$

Where,

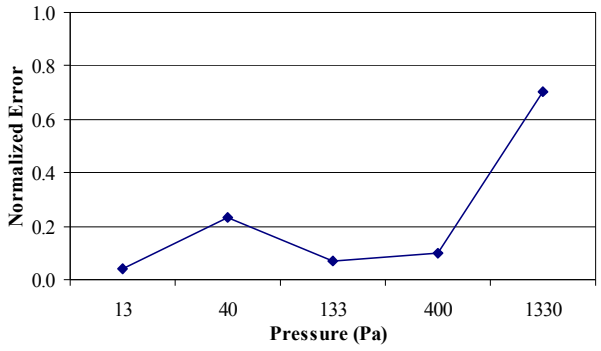
e_n - normalized error calculated at each calibration pressure,
E_{CENAM} - CENAM's estimated error,
$E_{INMETRO}$ - INMETRO's estimated error,
U_{CENAM} - CENAM's estimated expanded uncertainty,
$U_{INMETRO}$ - INMETRO's estimated expanded uncertainty.

The results of the normalized error equation application are shown in Table 5.

Table 5. Normalized error equation degree of equivalence between INMETRO and CENAM.

Pressure Pa	e_n
13	0,04
40	0,23
133	0,07
400	0,10
1330	0,70

The data presented in Table 4 is drawn in Graph 2, for easer understanding of compatibility of measurements.



Graph 2. Graphical representation of the normalized error equation.

This graph provides a better view of the comparison results and of the equivalence of measurements between the two NMIs.

From Table 5 and Graph 2, it is important to notice that no measured pressure point had a value of the normalized error equation greater than 0,7. Also, all but one (1 330 kPa) of the measured target pressure points had values of the normalized error equation below 0,25.

6. CONCLUSIONS

According to the normalized error equation analysis, it can be concluded that a good agreement exists between the measurements carried out by CENAM and those by INMETRO in the compared range of low absolute pressure (vacuum) from 13 Pa up to 1 330 Pa.

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