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SIM vacuum comparison from 133.3 Pa to 13.33 kPa

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Abstract

This comparison constitutes the second part of the first vacuum comparison among National Metrology Institutes (NMI) within the Interamerican Metrology System (SIM). Each laboratory used its national standards for the compared range. The Centro Nacional de Metrología (CENAM, Mexico) used a reference standard (comparison system type) and the Instituto Nacional de Metrologia, Normalização e Qualidade Industrial (INMETRO, Brazil) used a similar reference standard (comparison system type). 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 133.3 Pa up to 13.33 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, 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), a second part of the first vacuum comparison was performed [1]. This second part compared the measurements carried out by both countries by means of its secondary reference standard systems (comparison systems).

A capacitive diaphragm gauge (CDG), belonging to INMETRO, was used as transfer standard for the comparison. The pressure target points measured were the following five: 133.3 Pa, 400 Pa, 1.33 kPa, 4 kPa and 13.33 kPa.

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 [2, 3]. CENAM had the role of

coordinator. 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 low vacuum range from 133.3 Pa to 13.33 kPa (1 torr to 100 torr), using the normalized error equation as the equivalence parameter [1, 2, 3].

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 13.33 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
CENAM	2004-12-13	Capacitive Diaphragm Gauge
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: 133.3 Pa, 400 Pa, 1.33 kPa, 4 kPa, and 13.33 kPa.
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:	Capacitive Diaphragm Gauge	Capacitive Diaphragm Gauge
Character of standard:	Secondary	Secondary
Traceable to:	CENAM	PTB
CMCs listed in the BIPM data file:	Yes	Yes

The pressure standard used as reference by CENAM is based on the comparison method. The traceability of the references CDGs used in this system is to the static expansion system from CENAM [4, 5], which has had a successful bilateral comparison with PTB [6]. That bilateral comparison with PTB is linked to a Euromet comparison [7]. The reference system used for this comparison measures low and medium vacuum within the pressure range from 1 x 10^{-4} Pa up to 1 x 10^{5} Pa in absolute pressure. For the range of the comparison, it consists of four capacitive diaphragm gauges (CDGs), one having a range up to 1.3 x 10^{2} Pa, another with a range up to 1.3 x 10^{3} Pa, another 1.3 x 10^{4} Pa and the fourth with a range up to 1.3 x 10^{5} Pa.

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. For the range of the comparison, 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.

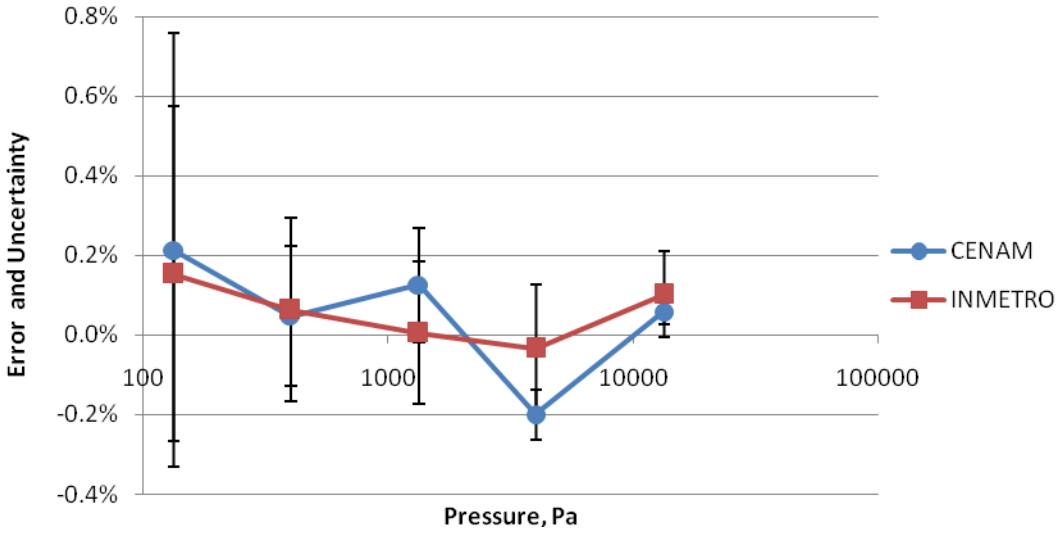
4. Results

Table 4 shows the summary of the measurements made by CENAM and INMETRO, for the calibration of the transfer standard (TS). The results are presented in percentage of the reading and referred to the pressure target point.

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

Table 4
Summary of measurements results

	INMETRO		CENAM	
Pressure, Pa	Error, %	U, % (k = 2)	Error, %	U, % (k = 2)
133	0.15	0.42	0.21	0.55
400	0.06	0.23	0.05	0.18
1 333	0.01	0.18	0.13	0.14
4 000	-0.03	0.16	-0.20	0.06
13 330	0.10	0.11	0.06	0.03



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 equation according to Equation 1.

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

Where,

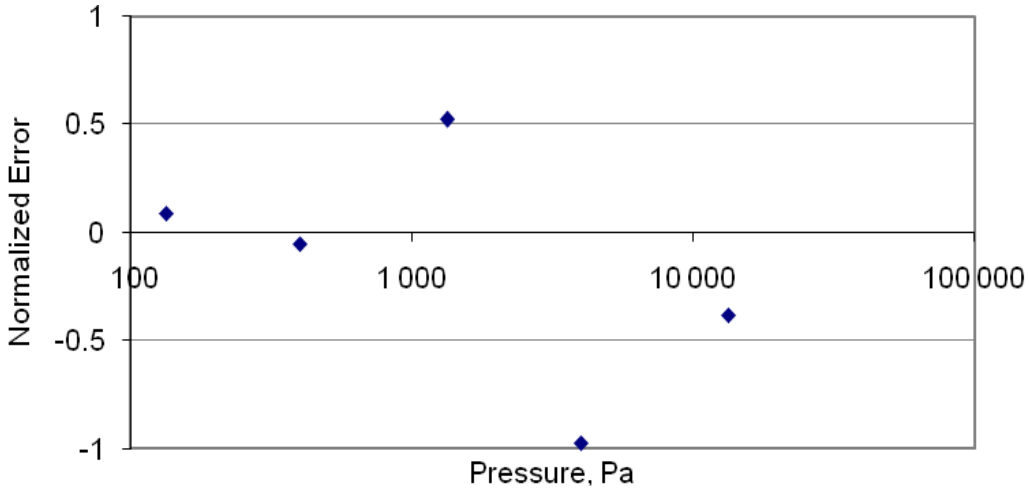
e_n - Normalized error calculated at each calibration pressure,
E_{CENAM} - CENAM's estimated error,
$E_{INMETRO}$ - INMETRO's estimated error,
U_{CENAM} - CENAM's estimated expanded uncertainty,
$U_{INMETRO}$ - INMETRO's estimated expanded uncertainty.

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
133	0.09
400	-0.05
1 333	0.52
4 000	-0.97
13 330	-0.38

The data presented in Table 5 is drawn in Graph 2, for easer understanding of the compatibility of measurements between the two participating laboratories.



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 1 (absolute value). Also, all but one of the measured target pressure points (4 000 kPa) had values of the normalized error equation below 0.6 (absolute value).

6. Conclusions

According to the normalized error equation analysis used for this comparison, it can be concluded that a good agreement exists between the measurements carried out by CENAM and those performed by INMETRO, in the compared range of low absolute pressure (vacuum) from 133.3 Pa up to 13.33 kPa. From the results, we can conclude that excellent agreement was found in most measured pressure points, only one pressure target point had a value greater than 0.6 (absolute value) of the normalized error equation; at 4 000 Pa a value of -0.97 was calculated.

We understand that better analysis of the comparison results can be achieved if a more detailed comparison procedure is used (including the full analysis of the transfer standard used). This comparison, as a pilot exercise, has given us the confidence that the measurements carried out in both laboratories are comparable for these secondary reference standard systems used in this measurement range in both countries.

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