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Methodology Employed to Calibrate Vacuum Sensors in INMETRO – Brazil

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Abstract

The present study it consists of a methodology developed in the Instituto Nacional de Metrologia, Normalização e Qualidade Industrial - INMETRO - (Pressure Laboratory - LAPRE) for the calibration of the vacuum sensors such as: capacitive diaphragm manometers, pirani, spinning rotor gauge and others. The used methodology is based on a program of calibration developed in the INMETRO/LAPRE having as principle the comparative method the transference standards. The method uses the analysis statistics as support. The calibration of these measurers in the LAPRE initiates the national metrologic chain for low pressures. Due to the great amount of instruments to be calibrated in the country it is necessary to spread, to stimulate and to guide the techniques of calibration of these sensors. With the transference of the jobs of calibration of these sensors after the certification of these according to the requisite for laboratories of norm ISO-17025 consequently the traceability and metrologic reliability of the measurements will be evidenced. Additionally the method makes possible also a study at any time of the repetitiveness of the calibrated instrument.

1 . Introduction

The LAPRE carries through calibrations in measurers of high - vacuum through a system Vacuum Gauge Calibration System (VGCS 200) of the MKS Instruments Inc. presented in the Figure 1 in the range 2.7E-6 Pa to 133kPa. In this range of scale the following standards are used: the system Spinning Rotor Gauge (SRG-II) (1E-4Pa to 2Pa), the Ionic Measuring System (Ion Gauge) (2.7E-6Pa to 8.3E-3Pa) and the Capacitive Measuring Systems (CDG - Baratrons) (1E-1Pa a 133 kPa).

The method of comparision between the standard of the LAPRE and the instrument of the customer, is the used one for calibrating the equipment. The gas used in the calibration as manometric fluid is the exteme pure nitrogen (99,999% of pureness). The calibrations of the measurers are carried through in two cycles, and for each point five readings in the standard when is possible the stabilization of the instrument being calibrated. When the stabilization impossibility occurs is carried through the simultaneous reading of the two instruments of the two instruments.

The vacuum chamber shown in Figure 1 presents 08 gates of output, in one of them Spinning Rotor Gauge is connected (SRG-II) and in two others are connected the Capacitive Measuring Systems. The five remaining gates are to be used for the calibration of up to 05 sensors of vacuum simultaneous.

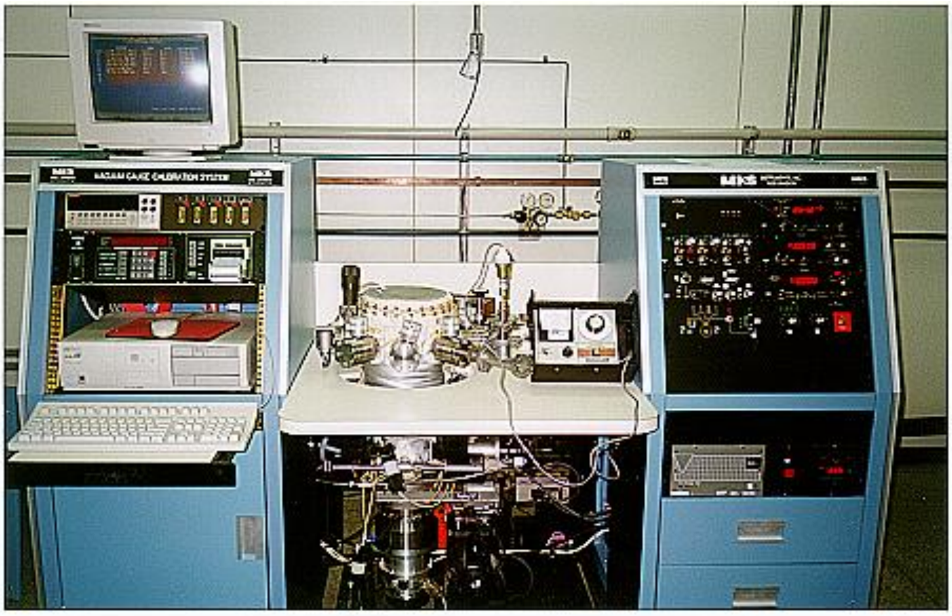

Figure 1. Vacuum Gauge Calibration System (VGCS 200)

2 . Experimental Set Up

Pacings to be observed for the calibration of the vacuum sensors, using the system of calibration of the INMETRO/LAPRE.:

1 the instruments (standard and test) will have to be in the ambient temperature, by at the very least 24h.

2 the sensor will have to be calibrated to a temperature of 20ºC±2ºC in rooms free of vibrations.

3 the customer will have to inform through a document where the sensor is used, to prevent possible contaminations in the system of calibration of the LAPRE.

4 Cleanness of the measurers to be calibrated in accordance with the instructions contained in the manual of the manufacturer. In the case of this not to be available, to enter in contact with the customer and consulting it on the cleanness methodology.

5 After providing the cleanness of the sensor, to check if all the valves of the calibration chamber are closed.

6 Install the to measurer be calibrated, using the appropriate connections in the chamber of calibration and for the prohibition rings, to use plastic gloves to prevent the contamination of the chamber of calibration with molecules of sweat of the hands.

7 the points to be calibrated are distributed in along range of scale of the instrument.

8 Turn on the system of calibration of the vacuum measurers 24 hours before carrying through the calibration and leaving it in the mode pumping until the attainment of a pressure less than 2E-6 Torr.

9 Heat the system of calibration during 12 hours, to remove the humidity contained in the walls of the vacuum chamber.

10 To wait the temperature of the calibration system to enter in thermal equilibrium with the ambient temperature.

11 To verify in controller 290, (component electronic of the calibration system), if it has na increasement of the base pressure and if it had, there is an emptying in the connections.

12 Using a syringe, to inject isopropilic alcohol around of each one of the connections used in the assembly and if it has any brusque change in the value of the pressure indicated in controller 290, this is an emptying point.

13 In case that it has emptying, to retighten all the connections, to repeat item 12 and if to persist the emptying to verify prohibition rings, to change them and to repeat item 12.

14 The rings of steel and copper prohibition can not be reused.

15 To set the zero of the sensor standard in controller 270, (component electronic of the calibration system) to initiate the measurements.

16 The sensors of vacuum of the customer must be set zeros as the instructions of the manufacturer.

17 In the measurement register to write down the values of the vacuum points to be calibrated.

18 To justify the multiplier factor, (1; 0,1 or 0,01), that are in controller 290, (another electronic component of the calibration system) for the range of calibration of the sensorr of vacuum of the customer.

19 To vary slowly the pressure in the potentiometer (set point) in the regulating of stream 244.

3 . Results

To the results gotten in the method they have to be determined the average and the standard deviation and is also made a study of the homogeneity of variances between the repetitivety and the reproducebility among the calibrated points of the instrument.

The results for each reading of the calibration are registered in an in form as the model presented in Table 1.

With the results gotten of Table 1 the corrections of the readings in the standard are effected and is calculated average and standard deviation, these are registered in accordance with the model presented in Table 2.

The expression of measurement uncertainty is in accordance with the Guide for Expression of the Uncertainty of Measurement - ISO GUM 1995[1] and has its components presented in the Table 3[2].

The uncertainties of the standards of the Laboratory of Pressão[3] are presented in Table 4. The uncertainties declared in Table 4 are for 95% reliability and k = 2.

Table 1. Register of the measeurement readings

Instrument reading ( )	Standard reading ( )	Instrument reading ( )	Standard reading ( )	Instrument reading ( )	Standard reading ( )	Instrument reading ( )	Standard reading ( )
1		6		11		16	

Table 2. Register of the averages and standard deviation of readings with the corrections of the standard.

Value indicated in the instrument ( )	Values due to the readings in the standard ( )							
	First Cicle				Second Cicle			
	Loading		Unloading		Loading		Unloading	
	μ_P	S_P	μ_P	S_P	μ_P	S_P	μ_P	S_P

Tabel 3. Components of the uncertainty calculus, an example

Sources of uncertainty	Value (Pa)	Distribution	Divisor	Sens. Coeffic.	Uncertainty (Pa)	Degrees of Freedom
Repetition of the readings of the standard in the point	0,1	normal	√5	1	0,045	4
Standard deviation of the readings of the standard in the point	5,4	normal	√4	1	2,7	3
Standard Certificate of calibration	4	normal	2	1	2	∞
Resolution of the instrument	0,1	rectangular	√3	1	0,058	∞
Combined uncertainty	-	normal		-	3,36	7
Expanded Uncertainty	-	normal k = 2,43 95,45%	-	-	8,2	-

Table 4 . Description of the uncertainty of the Vacuum Equipments in the Pressure Laboratory

Range	Equipment	Maximum Uncertainty
2,7 E –6 Pa a 8,3 E –3 Pa	**Ionic Measuring System**	**6,4 %**
1,0 E –4 Pa a 2,0 Pa	**Spinnig Rotor Gauge**	**0,4 %**
1,1 E-1 Pa a 133,32 Pa	**Capacitive Measuring System 1 torr**	**0,9 %**
1,129 Pa a 1333,22 Pa	**Capacitive Measuring System 10 torr**	**0,6 %**
91,5 Pa a 133,32 kPa	**Capacitive Measuring System 1000 torr**	**0,7 %**

The final presentation of the results for emission of the certificate as is presented in Table 4.

Table 4. Model of Presentation of calibration data in the instrument calibration certificate.

$P_{ind.}$ ( )	Error % Maximum	Standard Deviation ( )	Hysteresis (%)	k to 95,45%	Uncertainty (%)

4. Conclusions

With the implantation of this new methodology of calibration for low pressures, proposed by LAPRE, that is based in the training of the technician and mannering changes, is concluded to be an efficient tool in the calibration of the vacuum sensors, making with that it has an improvement of the exactness of the measures of vacuum and the reduction of the value of the measurement uncertainty.

Also it will act as one technique of measurement, that can be adjusted for the diverse paths of the national industry that use the vacuum technology and that they come consequently to search the traceability of its sensors to the national standard and the metrologic reliability of its Quality System.

The methodology used for the calculation of the uncertainty of type A is relative to the standard deviation of the errors, so that the referring dispersion only proved the readings of the instrument in test and not it variation of the readings of the standard of the LAPRE.

Also graphical accompaniments of measurement uncertainty are effected, in the internal registers of these graphs that show the quantification of the input values, the system of calibration of the INMETRO/LAPRE presents sufficiently steady, since the standard deviation of the measures of the points of the sensory standard, is lesser of that the standard deviation of the measures of the points of the sensors of the customer (test) till now.

This work can be applied any company, laboratory of metrology, or institution of education since that these call INMETRO for the necessary clarifications.

These companies must request to the INMETRO the certification of its laboratories for the implantation of the method of calibration of the vacuum sensors, therefore the decurrent costs with the implantation of the calibration program must be faced as investment, therefore the results with the quality and productivity are significant.

The pressure variations that may appear are easily detected simply by the locking of valves and observing the modifications in the readings of the capacitive manometers.

The standards of transference of the LAPRE must be kept in vacuum when not used to keep them free of contamination before the next use and this precaution helps to in this way minimize the time of beginning of the next calibration, diminishing the gas consumption, magnifying the stability of the equipment.

5 . References

[1].ISO International Organization for Standartization, *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9 1ª ed. 1993.

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