

## VACUUM PRESSURE STANDARDS INTERCOMPARISON BETWEEN CERN AND LNLS

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In November 1992, the vacuum standards of CERN and LNLS have been intercompared. Reference vacuum gauges from Laboratório Nacional de Luz Síncrotron (LNLS) Campinas Brazil and CERN Vacuum Group have been compared [1] and used for calibration of ionization gauges [2] and residual gas analysers [3].

### 1. INTRODUCTION

An ultra-high-vacuum (UHV) gauge cross-comparison was used for calibrating ionisation gauges and residual gas analysers (RGA) for use on the LNLS calibration system against CERN ionisation gauges and RGA standards. It was also used to gain experience on calibration systems.

### 2. MATERIAL

#### 2.1 GAUGES

Several gauges and the associated power supplies were provided by LNLS: one SVT ionisation gauge model 303 (303#2), one spinning rotor gauge VM 211 from Leybold (SRG) and one RGA QMG 420 from Balzers. They were compared with CERN gauges: one SVT ionisation gauge model 305, serial number 722, one SVT ionisation gauge (303#1), one SRG gauge VM 211 from Leybold and one residual gas analyser MassTorr-DX from Vacuum Generator.

#### 2.2 CALIBRATION SYSTEM

The gauges were mounted on an all-metal bakeable vacuum system equipped with a leak valve connected to a gas injection system, a turbo-molecular (TM) pump with isolation valve and a 60 l/s sputter ion pump.

#### 2.3 OPERATION

The vacuum system was evacuated with the turbo-molecular pump and baked at 300°C for 24 hours, the gauges were degassed and the ion pump was started prior to closing the valve between the TM pump and the system. A base pressure of

$2 \times 10^{-10}$  Torr (in nitrogen equivalent or  $4 \times 10^{-10}$  Torr of hydrogen) was reached two days after the end of the bake-out, consisting of hydrogen for about 95% and carbon monoxide, carbon dioxide and methane for the remaining 5%. Pure gas was injected into the system at increased pressure. After a first trial injection in the  $10^{-8}$  Torr range the sputter ion pump is switched off in order not to change the cracking pattern of the gas analysers. The test gas was then injected statically at pressure up to  $10^{-4}$  Torr.

### 3. MEASUREMENTS

#### 3.1 COMPARISON OF THE SPINNING ROTOR GAUGES

Before each gas injection, the two SRG were run at a pressure in the  $10^{-10}$  Torr range for ball deceleration measurement and offset correction. On condition that the balls were not stopped, the offsets of the two units remained stable; otherwise they had to be corrected. The two units gave the same measurements within 2% in the  $10^{-4}$  and  $10^{-5}$  Torr ranges and within 5% in the  $10^{-6}$  Torr range. At pressures below  $1 \times 10^{-6}$  Torr the measurements were not reliable. For calibration of ionisation gauges, the mean value of the two SRG were used.

#### 3.2 ABSOLUTE SENSITIVITY

The absolute sensitivity  $S$  of an ionisation gauge for a given gas is defined by:

$$I_c = P \cdot I_e \cdot S$$

with  $I_c$  the collector current output by the gauges,  $I_e$  the emission current and  $P$  the gas pressure. The LNLS ionisation gauge 303 and associated power supply was checked for nitrogen at pressure between  $2 \times 10^{-8}$  Torr and  $6 \times 10^{-5}$  Torr. The emission current was 5 mA and the grid voltage 195 V. The gauge output current was measured using an electrometer connected to the collector of the gauge. The mean value of the sensitivity for nitrogen was  $14 \pm 0.5 \text{ Torr}^{-1}$ . In order to check the gauge power supply, current was injected

in its electrometer with a calibrated current source. Taking into account the nominal emission current of 5 mA the gauge power supply should be used for direct reading, with a gauge of sensitivity  $19 \pm 0.5$  Torr<sup>-1</sup> for nitrogen. This explains the 70% ratio observed between the two 303 gauges readings.

### 3.3 CORRECTION FACTOR

The correction factor of a gauge for a given gas is defined as the ratio between the real gas pressure and the measurement displayed by the power supply. It was measured for the three ionisation gauges with their own power supplies and for the eight available gases in the  $10^{-6}$  -  $10^{-4}$  Torr range. The reference pressure was the mean of the two SRG gauges (error  $\pm 10\%$ ).

	305	303#1	303#2
H <sub>2</sub>	2.1	2.0	3.1
He	1.9	1.9	2.6
CH <sub>4</sub>	0.7	0.7	0.9
CO	0.7	0.8	1.1
N <sub>2</sub>	0.9	0.9	1.3
Ar	0.6	0.6	0.8
CO <sub>2</sub>	0.6	0.6	0.9
C <sub>2</sub> H <sub>6</sub>	0.4	0.4	0.7

The difference in correction factor of the two 303 gauges with their own power supplies is consistent with the low gain measured on the LNLS power supply electrometer.

### 3.4 RELATIVE SENSITIVITY

The relative sensitivity of a gauge for a given gas is defined as the ratio between the sensitivities for that particular gas and for nitrogen. The three ionisation gauges were measured in injection with the eight available gases. The following relative sensitivity have been deduced using the data of the previous table, measured in the  $10^{-6}$  -  $10^{-4}$  Torr range.

	305	303#1	303#2
H <sub>2</sub>	0.4	0.5	0.4
He	0.4	0.5	0.5
CH <sub>4</sub>	1.3	1.4	1.4
CO	1.2	1.2	1.2
Ar	1.5	1.6	1.5
CO <sub>2</sub>	1.4	1.5	1.4
C <sub>2</sub> H <sub>6</sub>	1.9	2.3	2.2

### 3.5 COMPARISON WITH PREVIOUS CALIBRATIONS

The CERN gauges model 305 are routinely calibrated at low pressure in the  $10^{-9}$  -  $10^{-8}$  Torr range using the dynamic method where a known gas flux is injected in a vacuum system with known pumping speed and a base pressure in the  $10^{-12}$  Torr range. For this measurement, they are operated at the nominal emission current of 4 mA. At pressures higher than  $10^{-6}$  Torr and emission current greater than 20  $\mu$ A the sensitivity of the 305 gauges decreases with increasing pressure; for this reason during a separate run two 305 calibrated gauges were recalibrated for nitrogen at 20  $\mu$ A in the  $10^{-6}$  -  $10^{-5}$  Torr range (high pressure) with the SRG. The mean sensitivities expressed in Torr<sup>-1</sup> obtained with both methods are (error low pressure:  $\pm 5\%$  and high pressure:  $\pm 10\%$ ):

GAUGE SERIAL NUMBER	305#379	305#118
S N <sub>2</sub> at low pressure	36	41
S N <sub>2</sub> at high pressure	41	48

The gauge 305#722 was also calibrated for different gases, the table below gives the sensitivities in Torr<sup>-1</sup> obtained with both methods:

	H <sub>2</sub>	N <sub>2</sub>	Ar	CH <sub>4</sub>	CO	CO <sub>2</sub>
S at low pressure	18	36	42	62	29	58
S at high pressure	19	42	71	63	58	48

### 3.6 ANALYSERS SENSITIVITY

The sensitivity of a RGA for a give gas can be expressed as the ratio of the peak current to the corresponding gas pressure. The peak current of the MassTorr-DX analyser have been measured without secondary electron multiplier during gas injection at pressures between  $10^{-7}$  -  $10^{-5}$  Torr. The mean sensitivities given below are expressed in  $10^{-4}$  A Torr<sup>-1</sup> (error:  $\pm 10\%$ ).

	H <sub>2</sub>	He	CH <sub>4</sub>	CO	Ar	CO <sub>2</sub>	C <sub>2</sub> H <sub>6</sub>
peak	2	4	40	16	28	28	28
S(p)	2.7	1.9	2.0	0.7	1.6	2.1	2.0

The sensitivities decrease by factor of two when the pressure is increased from  $1 \times 10^{-5}$  to  $5 \times 10^{-5}$  Torr; this happens also on the secondary peaks and is attributed to the neutralisation of the ions by the gas in the quadrupole section. The peak

currents of the Balzers QMG 420 analyser from LNLS have been measured during gas injection with the secondary electron multiplier turned on at 1500 V. The mean sensitivities given below are expressed in A Torr<sup>-1</sup> (error:  $\pm 10\%$ ).

gas	H <sub>2</sub>	He	CH <sub>4</sub>	CO	Ar	CO <sub>2</sub>	C <sub>2</sub> H <sub>6</sub>
peak	2	4	40	16	28	28	28
S(p)	6.6	4.3	5.1	7.6	4.1	5.9	10.9

#### 4. CONCLUSION

The comparisons between the CERN and LNLS vacuum standards have shown a good agreement, in particular for spinning rotor gauges. Some discrepancies between the calibration factors, mainly for carbon monoxide, of the model 305 gauge obtained with the SRG and the dynamic method will be investigated.

#### 5. ACKNOWLEDGEMENTS

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#### 6. REFERENCES

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