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Improvement of INRiM static expansion system as vacuum primary standard between 10^{-4} Pa and 1000 Pa.

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Abstract. The INRiM static expansion system has been recently modified in order to improve its performance and extend the working range down to $5 \cdot 10^{-4}$ Pa. A new characterization of the system has been performed and a preliminary estimate of the uncertainty has been evaluated.

1 Introduction

There is a wide range of industrial processes that need vacuum technology from atmospheric pressures down to about 10^{-4} Pa. Vacuum packaging of food, semiconductor devices fabrication, metallurgical and chemical processes, optical and electrical thin-film coating are only few examples of such applications.

In this range, the pressure is measured by standards such as capacitance diaphragm gauges (CDGs) and spinning rotor gauges (SRGs), which must be calibrated using primary standards, including static expansion systems, where the standard pressure is obtained through a static expansion of a pure gas, applying the law of the perfect gases [1-5].

The INRiM static expansion system has been developed in the past [6,7], but it has been recently modified in order to improve its performance and extend the working range down to $5 \cdot 10^{-4}$ Pa. It has been characterised again and it is presented in this paper.

2 Measurement system

The system is made of 304L stainless steel and it is mainly composed by three volumes: a main volume, $V=V_1$, of approx. 68 litres, which represents the measurement volume and two smaller volumes, v_2 and v_3 , of 0.01 litre and 0.5 litre respectively. Figure 1 shows a schematic of the system. The smaller volumes v_2 and v_3 can be singularly or both filled with a pure gas (usually nitrogen) at known pressure (p_0 , inlet pressure), then the gas is expanded in the main volume.

The standard pressure p generated in the system through the gas expansion from a small volume v to the measurement volume V can be obtained by the formula:

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$$P = \frac{p_0}{R} \cdot \frac{T_V}{T_v} \tag{1}$$

where p_0 is the inlet pressure in the volume v before the expansion, T_V the temperature of the measurement volume, T_v the temperature of the small volume v and R is the value of the expansion ratio from one volume to the other.

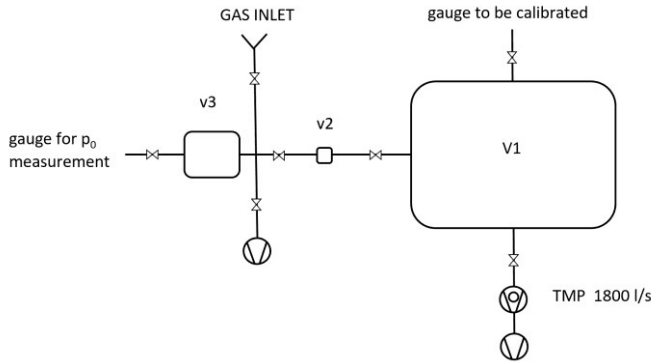


Fig. 1. Schematic of the INRiM static expansion system.

The expansion ratios have been determined using the multiple expansions method, based on the gas accumulation in the measurement volume through successive expansions [7]. The two possible expansion paths are reported in table 1.

Table 1. Expansion paths.

| Volumes involved | Expansion ratio, R |
|------------------------------------|----------------------|
| $v2 \rightarrow V1 + v2$ | $R4 = 6830$ |
| $v2 + v3 \rightarrow V1 + v2 + v3$ | $R3 = 106$ |

The inlet pressure covers a wide range of pressure from 3 Pa to 100 kPa: indeed, above 15 kPa it is measured by using CDGs or a barometer traceable to primary pressure balances, while below 15 kPa it is measured by CDGs calibrated using a Force Balanced Piston Gauge (FPG). FPG is a digital non-rotating pressure balance manufactured by Fluke/DH-Instruments used for gauge and absolute pressures in the range from few pascals up to 15 kPa. The temperature of the three volumes is accurately measured by a set of eight calibrated platinum resistance thermometer: six thermometers are placed on the measurement volume $V1$ and one on each small volume.

The system can be evacuated by means of a pumping system consisting of a mechanical and a turbo pumps with typical residual pressure in the measurement volume below 10^{-6} Pa.

3 Uncertainty budget

A preliminary budget uncertainty of the pressure generated by the renewed static expansion system has been evaluated. According to eq (1), the main components of the pressure uncertainty are the inlet pressure, the expansion ratio, the effect of temperature and the contribution of outgassing.

The uncertainty of the inlet pressure depends on the primary standard used for the calibration of CDGs and barometer. The expanded uncertainty of the inlet pressure is equal to

- $(3 \cdot 10^{-5} p + 0.02)$ Pa, in the pressure range between 3 Pa and 15 kPa
- $(2.4 \cdot 10^{-5} p + 0.5)$ Pa, for pressure higher than 6.4 kPa

as reported on the BIPM database [8].

The uncertainty of the expansion ratio has been evaluated according to ref [7], taking into account the uncertainty due to the measurement repeatability, the temperature variation and the real gas behaviour.

The standard uncertainty of the temperature has been estimated equal to 0.05 K.

The uncertainty contribution due to outgassing has been evaluated after a bake-out of the system up to 180°C for several days, to minimize the residual pressure. A maximum pressure rise of $5.8 \cdot 10^{-6}$ Pa, occurring during each measured pressure point, has been estimated and considered as an uncertainty contribution due to outgassing.

As result, in the pressure range between 0.5 mPa up to 0.09 Pa, the relative expanded uncertainty ranges from $2.4 \cdot 10^{-2}$ to $2.2 \cdot 10^{-3}$, while from 0.09 Pa up to 1000 Pa the relative expanded uncertainty ranges between $2.2 \cdot 10^{-3}$ and $6.8 \cdot 10^{-4}$.

Two examples of uncertainty budget are reported in tables 2 and 3, for a pressure equal to 0.09 Pa and 1000 Pa, respectively.

Table 2. Uncertainty budget for a pressure point equal to 0.09 Pa

| Uncertainty component | Expected value | Standard uncertainty | Sensitivity coefficient | Uncertainty contribution |
|---|----------------|------------------------|----------------------------|--------------------------|
| Inlet pressure | 9.58 Pa | 0.01 Pa | $9.39 \cdot 10^{-3}$ | $9.39 \cdot 10^{-5}$ Pa |
| Expansion ratio | 106.44 | $2.60 \cdot 10^{-2}$ | $-8.46 \cdot 10^{-4}$ Pa | $2.20 \cdot 10^{-5}$ Pa |
| Temperature of the measurement volume V_1 | 295 K | 0.05 K | $3.05 \cdot 10^{-4}$ Pa/K | $1.53 \cdot 10^{-5}$ Pa |
| Temperature of the initial volume v_0 | 295 K | 0.05 K | $-3.05 \cdot 10^{-4}$ Pa/K | $1.53 \cdot 10^{-5}$ Pa |
| outgassing | 0 | $5.8 \cdot 10^{-6}$ Pa | 1 | $5.8 \cdot 10^{-6}$ Pa |
| $u(p)$ when $p = 0.09$ Pa | | | | $2.0 \cdot 10^{-4}$ Pa |

Table 3. Uncertainty budget for a pressure point equal to 1000 Pa

| Uncertainty component | Expected value | Standard uncertainty | Sensitivity coefficient | Uncertainty contribution |
|---|----------------|------------------------|-------------------------|--------------------------|
| Inlet pressure | 106441 Pa | 1.53 Pa | $9.39 \cdot 10^{-3}$ | $1.44 \cdot 10^{-2}$ Pa |
| Expansion ratio | 106.44 | $2.60 \cdot 10^{-2}$ | -9.39 Pa | $2.44 \cdot 10^{-1}$ Pa |
| Temperature of the measurement volume V_I | 295 K | 0.05 K | 3.39 Pa/K | $1.70 \cdot 10^{-1}$ Pa |
| Temperature of the initial volume v_0 | 295 K | 0.05 K | -3.39 Pa/K | $1.70 \cdot 10^{-1}$ Pa |
| outgassing | 0 | $5.8 \cdot 10^{-6}$ Pa | 1 | $5.8 \cdot 10^{-6}$ Pa |
| $u(p)$ when $p = 1000$ Pa | | | | $6.8 \cdot 10^{-1}$ Pa |

4 Conclusions

The renewed INRiM static expansion system is able to generate pressures from 0.5 mPa up to 1000 Pa, thus allowing the calibration of both capacitance diaphragm gauges (CDGs) and spinning rotor gauges (SRGs). The system has been characterized and a preliminary estimate of the expanded uncertainty has been performed. Between 0.5 mPa up to 0.09 Pa, the relative expanded uncertainty ranges from $2.4 \cdot 10^{-2}$ up to $2.2 \cdot 10^{-3}$, while from pressure between 0.09 Pa up to 1000 Pa the relative expanded uncertainty ranges between $2.2 \cdot 10^{-3}$ and $6.8 \cdot 10^{-4}$. In order to validate the system, comparisons with several calibrated sensors are ongoing and an international comparison with another National Metrology Institute is foreseen.

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