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Three Technologies Met In The Absolute Pressure Range 0,5 Pa to 3 Pa

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Abstract

The LNE standards around 1 Pa absolute are constituted by a force-balanced piston gauge (FPG), 100 Pa capacitance diaphragm gauges (CDGs), and spinning rotor gauges (SRGs). The key parameters limiting the performance of the devices are presented. The results of a comparison involving at the same time the three instruments are presented and discussed, as well as the capabilities of these instruments in this range. As an example, the repeatability of measurements, and the linearity error in the comparison are less than 2 mPa.

Keywords: Capacitance diaphragm gauge; Spinning rotor gauge; Force Piston Gauge; Thermal Transpiration Effect

1. Introduction

LNE pressure standards around 1 Pa absolute are constituted by a force-balanced piston gauge (FPG), 100 Pa-capacitance diaphragm gauges (CDGs), and spinning rotor gauges (SRGs). If the first one is relatively new, LNE has a long experience in the use of the two others [1]. The pressure scale between 0,5 and 3 Pa is common to the three technologies. At this pressure range, these instruments have qualities and disadvantages inherent in their technology. Most of them are complementary, and their uncertainties are almost identical. Our best measurement capabilities for these instruments are (see figure 1):

(0,013 Pa + 2,3 x 10-5 x P) for the FPG [2],
(0,008 Pa + 1,3 x 10-3 x P) for the CDG,
(7 x 10-6 Pa + 5,0 x 10-3 x P) for the SRG.

In order to ascertain the performances of these instruments in this crucial range and to improve our knowledge concerning their behavior, a comparison involving the three instruments has been carried out in January 2007. The comparison was accomplished by calibrating at the same time one SRG and one CDG with the FPG.

2. Details Of The Comparison

The measurements were carried out with nitrogen. The room temperature was kept at 20 °C within ± 0,2°C. The SRG (type SRG2) and the CDG (type Baratron 698) used for the comparison were connected to the upper chamber of the FPG. They were mounted with tilt adjustment and housed in a polystyrene box.

The comparison was performed within 2 days for 5 cycles by increasing pressure at the nominal pressures of: 0,5 - 0,7 - 1 - 1,5 - 2 - 2,5 - 3 - 3,5 - 4 - 4,5 - 5 Pa.

The pressure was regulated by the FPG pressure contoller (VLPC). The measuring time interval - 20 s - was the same for each instrument.

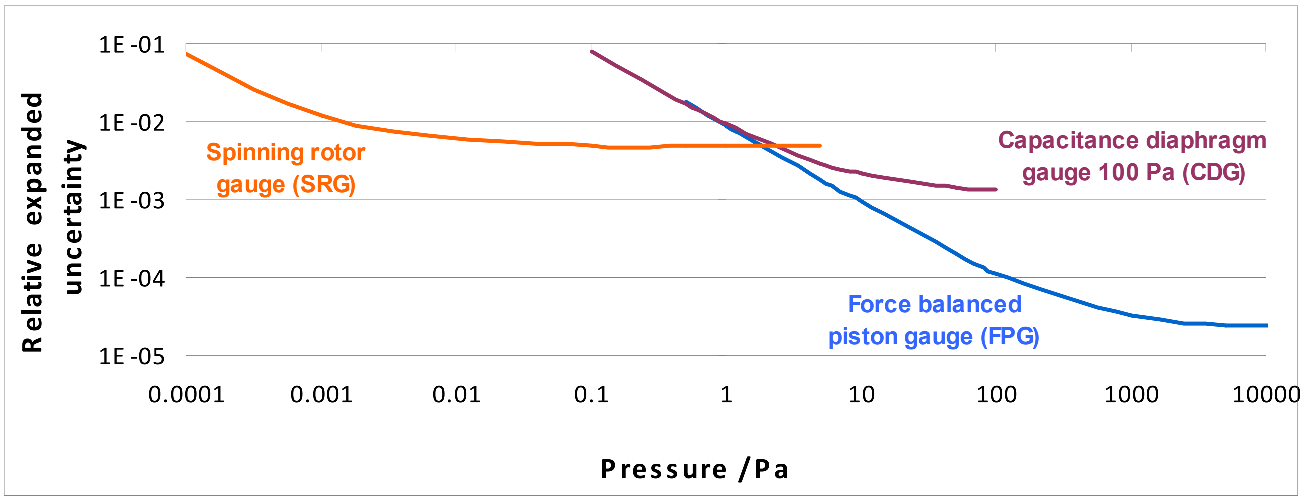

Figure 1. Best measurement capabilities of the standards as a function of pressure

2.1 The Force Balanced Piston Gauge (FPG)

The metrological characteristics of the instrument have been described in reference [3]. Its stability, established to be within a few 10-6, is defined from both its piston-cylinder effective area and its load cell, daily calibrated. But for pressures below 1 x 10-3 of the FPG full scale, the most essential points concern the pressure stability of the associated pressure controller VLPC, the reference pressure (residual vacuum) of the FPG, and the error due to the zeroing of the FPG.

The first point has been achieved by using a new turbo-molecular pump. The pressure fluctuations measured by each instrument during the measuring intervals (20 s) were typically 0,002 mPa for the FPG and 0,001 mPa for the CDG and the SRG, whatever the pressure points were.

Concerning the residual pressure, a second turbo-molecular pump allowed to reach 60 mPa with very low fluctuations of 2 mPa within 2 days. This reference pressure was measured with a 10 Pa-CDG.

The last point is more delicate because it is non measurable. Zeroing the FPG consists in connecting together both chambers with no other pressure influence. Nevertheless, while zeroing, we can notice a slight tendency of the dynamometer, difficult to estimate, but which could indicates that the zero conditions are not perfectly met.

2.2 The Capacitance Diaphragm Gauge (CDG)

CDGs were previously calibrated in differential mode using 2 pressure balances; they are now calibrated by direct comparison with the FPG [3]. In order to improve their zero stability, the CDGs are habitually used heated. At low pressure, the difference of temperature □T - between the CDG and the vacuum chamber where the pressure is measured - generates a significant phenomenon called thermal transpiration. This phenomenon is corrected with a function carried out by Takaishi and Sensui [4]

For pressures less than 5 Pa, the generated effect of thermal transpiration is the main factor when fitting the CDGs output signals. An error of 1°C in the determination of □T causes, below 5Pa, a quasi-linear error of 1,6 x 10-3 x P (which tends to the Knudsen value - see fig 2). This is all the more important as, in that range, the quasi-linear aspect of the thermal transpiration effect makes the error not easily detectable by the SRGs.

A numeric simulation of the thermal transpiration effect shows that an appropriate polynomial fitting can compensate an error in the determination of □T when calibrating the CDG. The generated errors of a few mPa are then insignificant. At low pressure the polynomial fitting cannot compensate it.

Long experience in calibration of CDGs shows different behaviour from one instrument to another. The temperature scale is generally from 37 to 45 °C. The use of CDGs for calibrating SRGs requires consequently the determination of the heated temperature individually. This is achieved with the FPG, of which automated capabilities allow to perform calibrations with a considerable number of pressure targets (about 40) in order to get the best fitting.

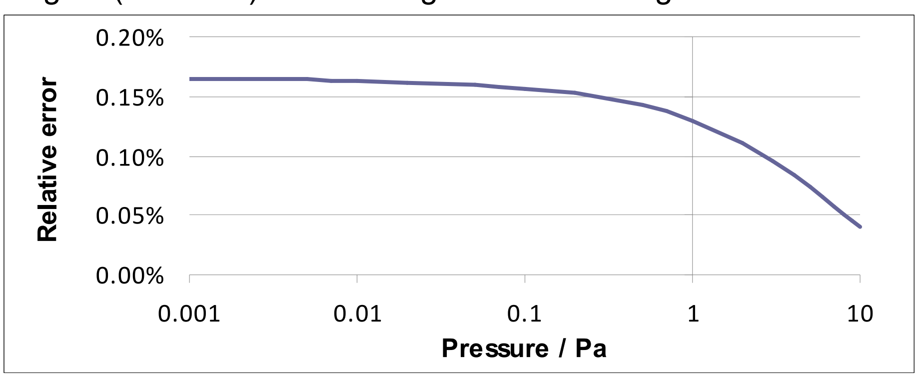

Figure 2. Thermal transpiration effect for □T= 1°C around 45°C for N_2.

2.3 The Spinning Rotor Gauge (SRG)

The accommodation coefficient σ of the SRGs is determined between 0,01 and 3 Pa by direct comparison with a CDG [1]. As noted earlier, an error of a few degrees in the determination of CDG temperature generates an important and undetectable error in the determination of the accommodation coefficient of the SRG. The new means of determining the temperature regulation of the CDG consolidated our assumptions concerning the uncertainties of the accommodation coefficient, which is estimated to be 2,5.10-3.

Another key parameter is the zero-pressure offset of the instrument. It is determined by combining the information issued from the SRG and an ion gauge [1]. The stability of the offset was demonstrated to be within 1.10-5 Pa over several weeks, if the SRG was not switched off. The validity of the method was demonstrated through the comparison EUROMET.M.P-K1.b [2].

3. Results Of The Comparison

3.1 Correction From The Zero-Pressure Offset

As seen earlier, the measurement performances of the FPG in low pressure are limited by the offset of the instrument. This is due to its zero process and the reference pressure measurement. It was decided to correct the FPG pressure for its zero-pressure offset by using the SRG. It was calculated as the intercept, at zero SRG pressure, of a least square regression line between the pressures delivered by both instruments. For its part, SRG zero-pressure offset was determined using an ion gauge just before the comparison. Anyway, its value is 2 decades below the FPG offset.

The FPG zero-pressure offset, for the comparison, was calculated to be 0,005 4 Pa. In so doing, the uncertainty of reference pressure measured with a 10 Pa CDG is estimated to be 5 mPa, and the uncertainty of pressure measured by the FPG, (9 mPa + 2,3 x 10-5 x p).

3.2 Corrections for the Thermal Transpiration Effect

The thermal transpiration effect is calculated by using the equation of Takaishi and Sensui [4]. The corrections consist in calculating the pressure that the CDG and the SRG would measure if they were operating at the same temperature as the FPG. The difference in temperature of the FPG and the other instruments is measured at each target pressure.

A calibration in the whole range of the CDG was performed previously in order to determine its regulated temperature TReg. It was determined to be 45 °C.

3.3 Experimental Standard Deviations

The first indication concerns the experimental standard deviation. It is known that the repeatability of the SRG and the CDG are lower than the FPG in this range of pressure. Figure 3 confirms this, due to the similarity of both curves. We can notice that the maximum experimental standard deviation of 2,5 mPa represents 1,7 X 10-7 of the FPG full scale.

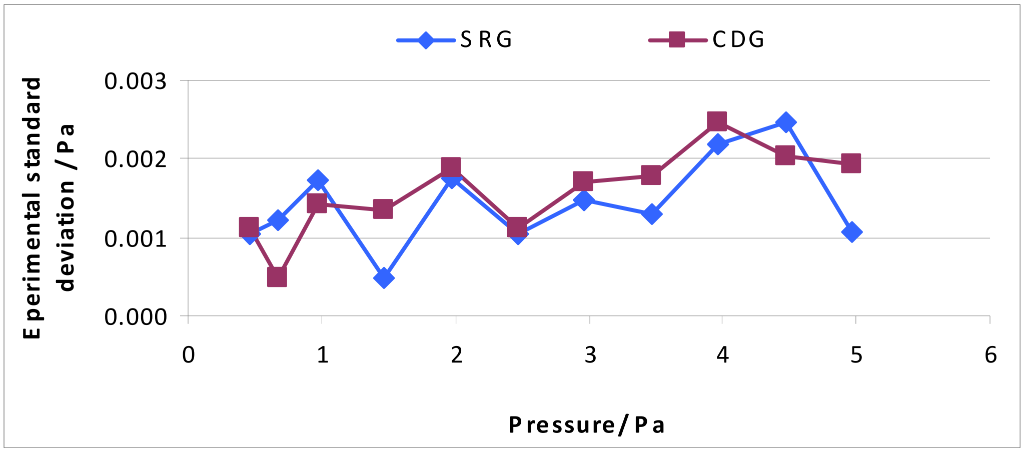

Figure 3. Experimental standard deviations of the SRG and the CDG by comparison with the FPG

3.4 Fitting the Devices

Once corrected for the thermal transpiration effect, the three pressure devices can be considered as linear devices in this range, and so, a linear fitting is adopted.

The results of the fitting show, first of all, that the SRG is not linear any more for pressures above 3,5 Pa. The results from 3,5 Pa to 5 Pa were therefore withdrawn for the SRG. The residuals from the least squares straight lines are given in figure 4. Theses results are very satisfying: all the residuals are within 1 mPa, which is the resolution of the FPG.

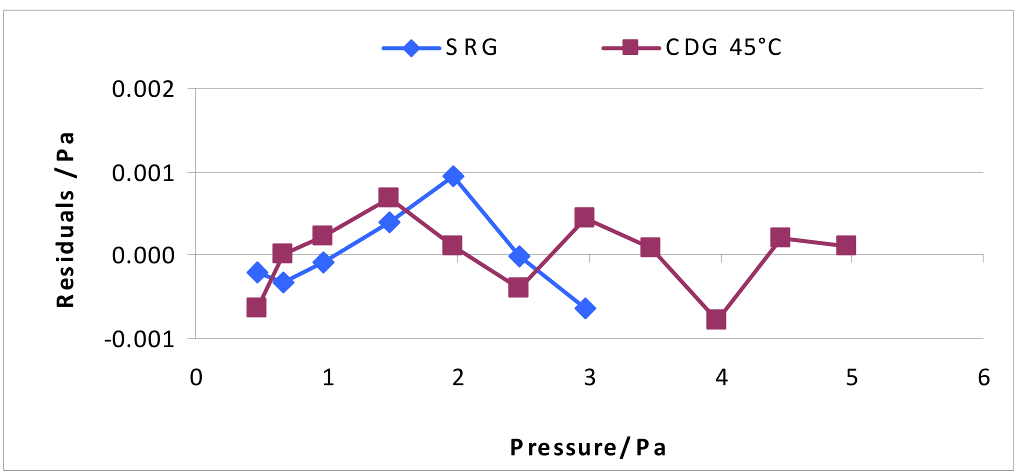

Figure 4. Residuals from the least squares straight lines for the SRG and the CDG by comparison with the FPG

3.5 Comparison with Usual Procedures

In order to validate the comparison, we decided to compare the results of the SRG with those obtained with our two usual calibration procedures. We first calibrated the SRG just prior the comparison with our reference SRG2, and then 6 months later by direct comparison with our reference 100 Pa-CDG. Figure 5 presents the ratio of indicated pressures of the SRG to the standard pressures.

The results are coherent. They confirm a lack of linearity of the SRG above 3 Pa and show an agreement better than 0,2%.

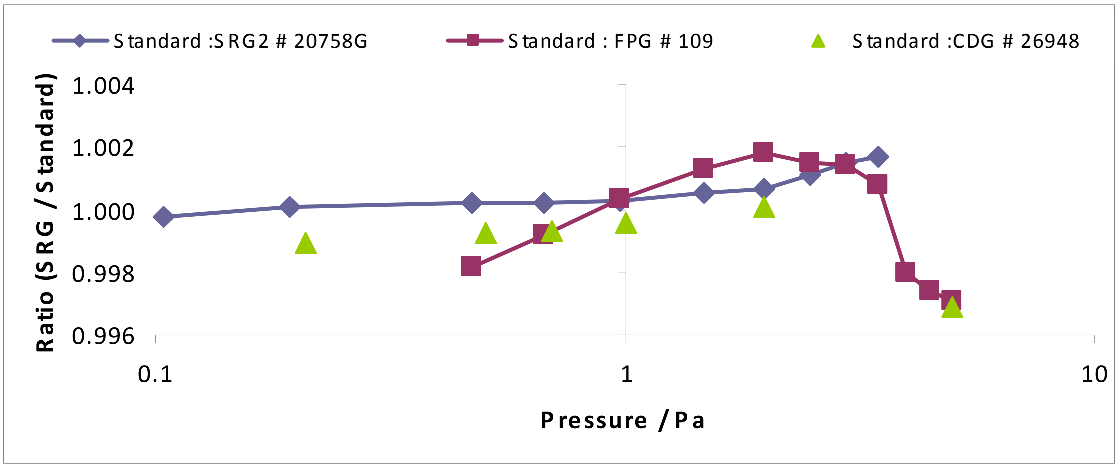

Figure 5. Ratio of indicated pressures of the SRG to standard pressures

The value of the accommodation coefficient of the SRG determined by each standard and the corresponding uncertainty are presented figure 6. As may be seen, the data on the comparisons are well inside the uncertainties, which confirm the validity of the comparison and our assumptions over more than 10 years concerning the behaviour of these instruments in terms of linearity and stability [1].

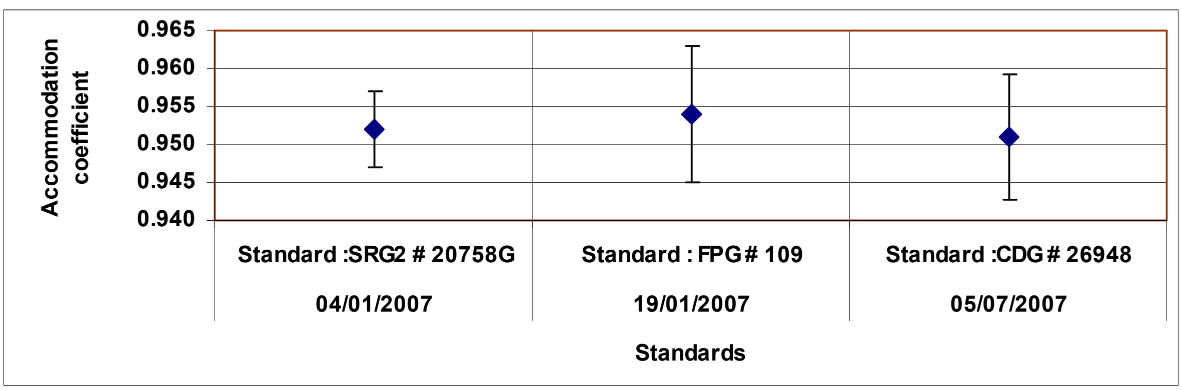

Figure 6. Accommodation coefficient of the SRG determined from three standards. The bars represent the expanded uncertainties.

4. Conclusion

Pressure measurements around 1 Pa can be performed at LNE using three technologies: a force-balanced piston gauge (FPG), 100 Pa-capacitance diaphragm gauges (CDGs), and spinning rotor gauge (SRGs).

A comparison involving the three instruments at the same time showed the complementary of the instruments in terms of linearity, offset, resolution and stability.

The capability of the FPG to measure pressures in this range has been established once corrected for its zero-pressure offset. The errors related to correction for thermal transpiration effect, in particular for the CDG, have been pointed out.

The comparison has been validated with results obtained with usual procedures.

This comparison improved our knowledge concerning the three pressure standards in this range and confirms our methods for the extrapolation to low pressure [1].

References

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