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Transfer Gauges for the Pressure Range From 100 kPa Down to Ultra High Vacuum

Mercede Bergoglio, Anita Calcatelli
Istituto di Metrologia "G.Colonnetti", IMGC-CNR
Torino, Italia

Abstract

Primary metrological laboratories have been and are involved in the characterisation of vacuum gauges to be used as transfer gauges at various levels (comparisons or dissemination). In the present paper, some of the main characteristics of the most popular gauges in the whole pressure range from the atmosphere down to ultra-high vacuum are presented and discussed together with a short presentation of the primary standard systems available at IMGC- CNR.

1.Introduction.

In the last years, and in connection with the need for international comparisons and calibrations for secondary laboratory accreditation, accurate metrological characterisations of transducers from atmospheric pressure down to high vacuum were performed in several metrological laboratories. From point of view only some of the commercially available gauges show an interesting behaviour and can be considered as suitable transfer gauges. For the considered range there are essentially three types of transfer gauges: mechanical gauges for the (10^5-0,1) Pa range; momentum transfer gauges (spinning rotor) for the (1 – 10^{-4}) Pa range; ion gauges for pressures lower than 10^{-2} Pa.

Accurate metrological characterisations consist in performing several calibration cycles made in different days with reference to primary standard systems. Consequently, correction factors of the gauges are defined; they may be constant over the whole considered pressure range or a function of pressure. Their uncertainty is evaluated taking into account short term repeatability and stability (when available). Calibration results are given in the calibration certificates which then constitute a library of stored data which can be continuously compared to give useful information on the stability of the considered gauges over wide time intervals (up to several years).

The mentioned gauges must be calibrated against primary standard systems in turn based on different physical principles which, at IMGC-CNR, are the following: pressure from

100 kPa to 1000 Pa: interferometric manometer [1]; pressure from 1000 Pa to 10^{-1} Pa: static expansion system [2,3]; pressure lower than 10^{-1} Pa: continuous expansion system [3,4,5].

Those systems are shortly described with their uncertainty values. Among the secondary gauges only those showing good metrological characteristics are considered, namely: pressure from 100 kPa Pa to 1 kPa resonant structure gauges-RSG or capacitance diaphragm gauges- CDG [6,7]; pressure from 1000 Pa to 10^{-1} Pa: capacitance diaphragm gauges-CDG [6,7,8]; pressure from 10 Pa 10^{-3} Pa: spinning rotor gauges-SRG [9,10]; pressure lower than 10^{-3} Pa: ion gauges-IG [11,12].

The gauges are calibrated in nitrogen atmosphere if there are not special requests

2. Primary Standard Systems

The whole considered pressure range can not be covered by only one system, but depending on the range three devices based on different principles have been realised at IMGC.

a) pressure from 10^5 Pa to 1000 Pa

The primary pressure standard of IMGC in the barometric range up to 120 kPa is a laser interferometer manometer (called Hg5), essentially made of an U-tube placed in a temperature-controlled water bath. The vertical displacement of mercury menisci is measured by a single-beam interferometer. The two vertical laser beams are normally reflected by cube corner reflectors carried by very light weight floats (thin glass disks) placed on both mercury menisci.

b) pressure range from 1000 Pa to 0. 1 Pa

In this range the standard is the static expansion system, which is equipped with three volumes having 10^{-2} L, 0,5 L and 50 L capacities. The largest volume is the calibration chamber. The different expansion ratios (R_i) were measured, and are periodically determined, by application of the well known multiple-expansion method. The inlet pressure values between 1 kPa and 100 kPa are measured by secondary transfer standards directly traced to the mercury manometer. The base pressure is in the range of 10^{-6} Pa; when necessary the system can be baked. The generated pressure is given by $p=(p_0/R_i)Fc$, where Fc is a factor taking into account temperature gradient ($Fc= T_V/T_v$ where T_V and T_v are the temperatures of the calibration chamber and of the in-let chamber respectively

c) pressure lower than 0.1 Pa

For pressures lower than 0.1 Pa at IMGC a continuous expansion system of fixed conductance and variable gas flow-rate (Q) is used. The generated pressure is evaluated by the relationship $p=(Q/S_{eff})F_c = [Q/(C/(1+C/S_p)] F_c = [Q/(C (1-p_2/p_1)] F_c$, where p_1 and p_2 are the pressures in the calibration and pumping chamber respectively, C is the conductance between the two chamber, S_p is the pumping speed of the pumping system and Fc is the factor taking into account the temperatures; it is given by $F_c = (T_{ch}/T_Q)\ (T_r/T_{ch})^{1/2}$ (where T_{ch} is the temperature of the calibration chamber and consequently of the conductance, T_Q is the temperature of the flow meter, T_r is the temperature at which the conductance has been calculated (in our case 293,15 K)).The measurement of the gas flowrate Q ($Q= p_o\ (A\ \Delta L/\Delta t)$ is based on the constant pressure and variable volume method. The value of $\Delta L/\Delta t$ at pressure limit for the molecular regime is situated at $9x10^{-2}$ Pa which is considered the higher pressure limit of the device.

The temperatures of the various parts of the system are measured by PRTs which are all traced to the each gas flow rate level is evaluated by the slope of the straight line L=f(t).The high

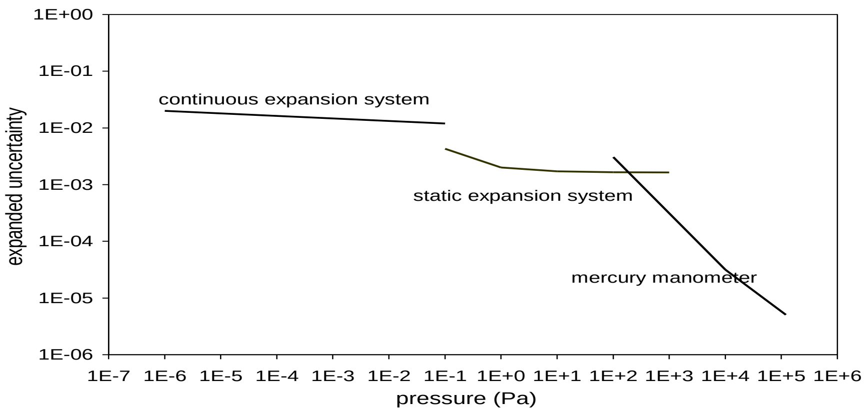

Figure 1. Expanded uncertainty versus pressure for the IMGC primary vacuum systems

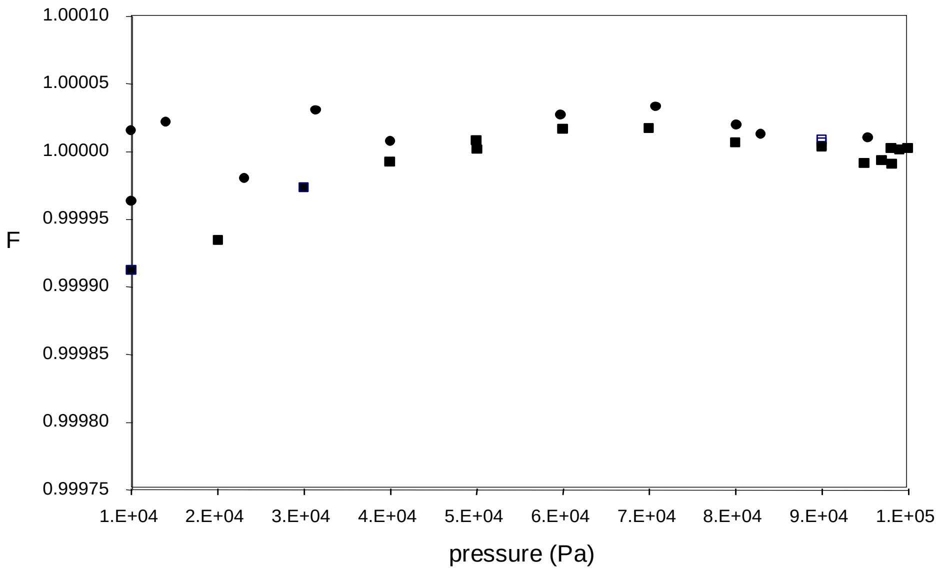

Figure 2. Correction factor versus pressure for a RSG, ■first calibration ● after one year

IMGC Temperature Division while the inlet pressure (p_0) of the static and dynamic systems are directly (133 kPa f.s) or indirectly (133 Pa f.s.) traced to the manometer.

Figure 1 summarizes the expanded uncertainty values for the primary IMGC systems in the considered range (from 100 kPa down to 10^{-6}.

Their standard uncertainty, evaluated at IMGC by considering all the components related to resolution, to the computational model and to the primary systems resulted to be 0.3 Pa at 10 kPa and of 2 Pa at full scale. Their hysteresis is negligible down to 10 kPa while is 0.2 Pa in the (10 –1) kPa range. Figure 2 gives an example of RSG calibration curves

The behaviour of the CDG the correction factor is linear with pressure for the (100 – 10) kPa range and the hysteresis is of the order of 3 Pa at 10 kPa and less at full scale. If in the period between calibrations the gauges are accurately used and preserved (with both sides at the same pressure) their output drift is about 35 Pa at the full scale and 5 Pa at 10 kPa.

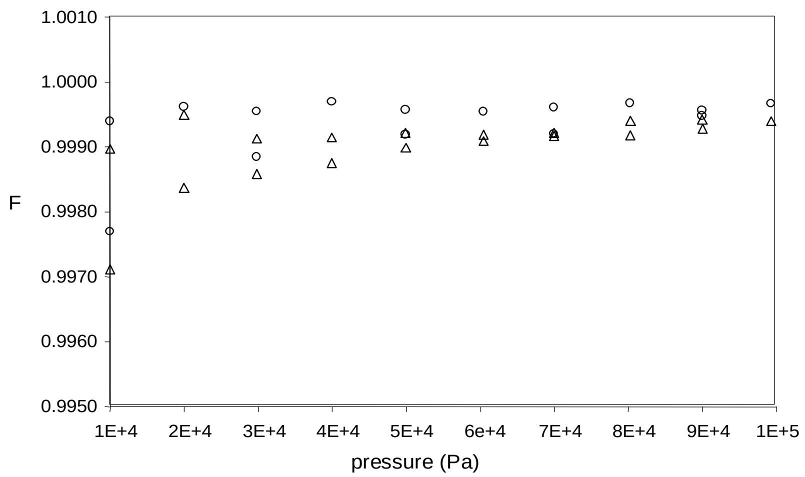

Figure 3. Typical behaviour of the correction factor versus pressure over two calibration cycles for a CDG in subsequent days

d) pressure range from 1000 Pa to 0. 1 Pa

In this pressure range CDGs are still used, and they are normally characterised with static expansion system only at increasing pressure; consequently, it is advisable that they are used in the same conditions. These gauges are generally available with their own heating system which stabilises their temperature at about 45°C; therefore, if they are used with the heater on, thermal transpiration must be taken into account [6]. The correction factor does not show a linear behaviour with pressure; various fitting procedures were applied [7] but a continuous function such as

$$F = \left[(a - d)/(1 + (x/c)^b) \right] + d \qquad (1)$$

proved to be the most reliable. In the above equation x represents the gauge reading. Under ideal conditions, at low pressure (molecular regime) F tends to be equal to the square root of the ratio of the calibration chamber and gauge temperatures; while, in the viscous regime (P>100 Pa) it tends to 1.

These two limits should coincide with the limits of the fitting functions which are, respectively, a (x=0) and d (x=∞). A possible difference between the values of the two sets of constants and their ideal condition values is due to the initial calibration when the gauge scale was drawn.

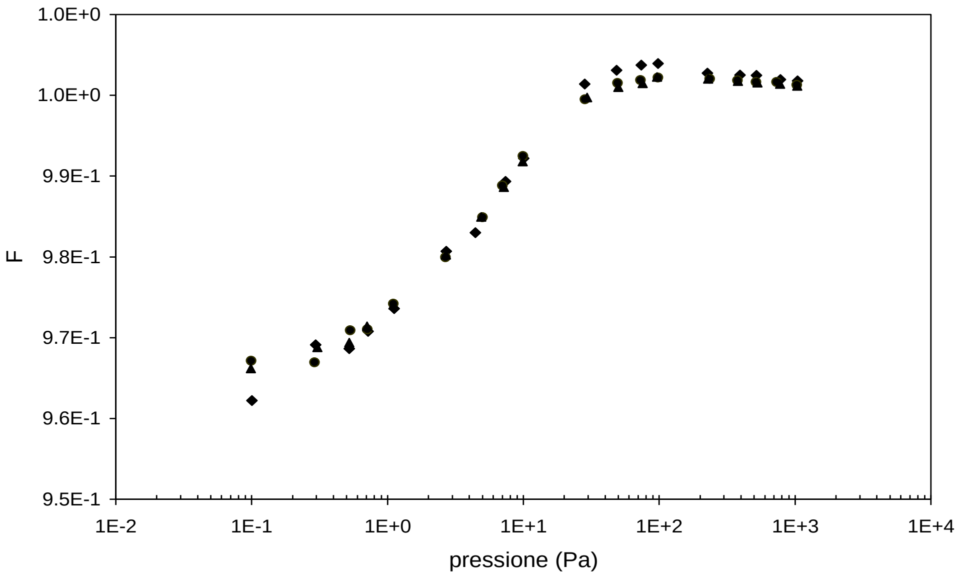

Figure 4. Typical behaviour of the correction factor versus pressure for several calibration cycles

Once the gauges are calibrated and their calibration factors defined, together with the best curves fitting the experimental data, their metrological characteristics may be defined, including the uncertainty values which take all the factors into account: resolution, short and long-term repeatability, output stability, standard deviation of the fitting curve and uncertainty of the primary device. For the best gauges the relative expanded uncertainty ranges from 5 10^{-3} at 0.1 Pa to 2 10^{-3} at full scale and the stability, over a period of three years, is 0.8 Pa at 1 kPa.

e) Pressure lower than 0.1 Pa

For pressures in the (1 - 10^{-4})Pa range SRGs are the most reliable gauges; they are generally calibrated at IMGC against the continuous expansion system for pressures from 10^{-4} Pa 10^{-1} Pa and in the static expansion system for pressure higher than 10^{-1} Pa. SRGs show the interesting characteristic of being linear over a wide pressure interval, namely from 10^{-4} Pa to 1 Pa, as it is shown in figure 5. For pressures higher than 10^{-1} Pa the signal from the SRGs becomes increasingly not linear with increasing pressure up to a saturation [10]; that is connected to the increases of the temperature and to the gas viscosity. But even at pressures slightly higher than 1 Pa the gauges show good repeatability of the calibration curves [9].

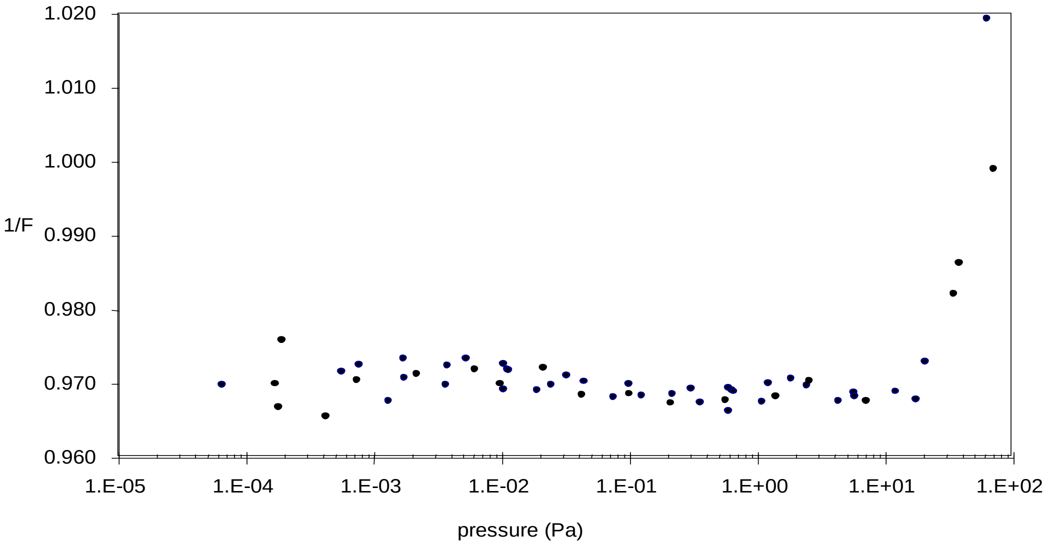

Figure 5. Typical behaviour of the gauge coefficient for SRG as function of the pressure

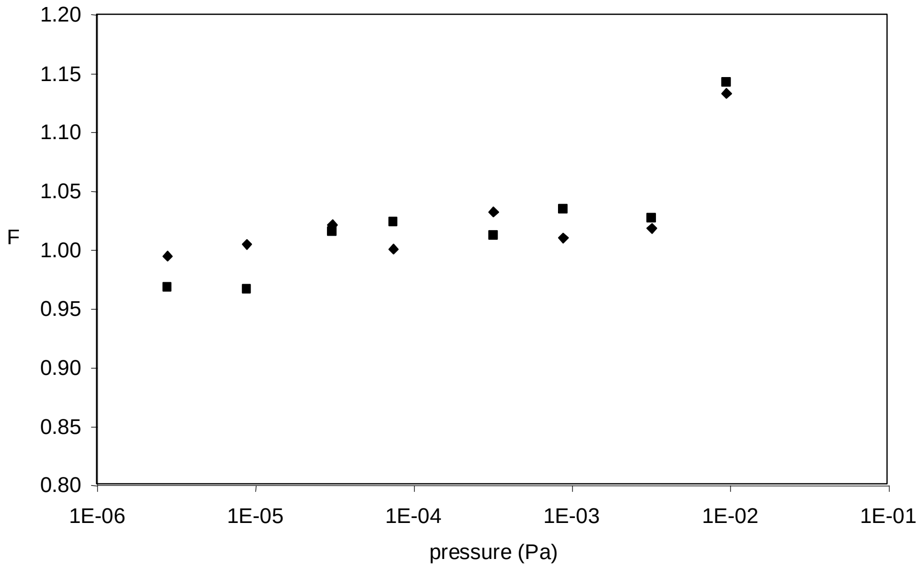

Figure 6. Correction factor versus pressure for an extractor ion gauge in two subsequent days

These gauges are considerably stable, their stability being even better than 1% in the wide linearity range but it is strongly related to the conditions of the rotor surface. The manufacturers mostly for what concerns the constancy of the frequency and the possibility of checking it continuously improve sRGs.

Nowadays realisations easy to be used while preserving interesting metrological characteristics [13] are available. The relative standard uncertainty is $2x10^{-2}$ at $1x10^{-4}$ Pa and $3x10^{-3}$ at 10 Pa, including the stability over a period of six months.

For pressures lower than 10^{-4} Pa and down to ultra-high vacuum hot cathode ion gauges are the most diffused measuring devices, at various levels, from research to industrial laboratories. When they are used as transfer gauges they must be equipped with very stable control units or with especially built units to maintain constant the voltages to the electrodes during their operation.

In addition, both for calibration as well as for normal operation, if they are exposed to the atmosphere they need to be subjected to special conditioning procedures to be used at their best operating possibility. In this way their short term repeatability can be a few parts per cent.

Results of calibration cycles of one commercial gauge (extractor type) are shown, as an example, in figure 6.

3. Conclusions

The combined uncertainty of the considered resonant gauge is 0,3 Pa at 10 kPa and 2 Pa at full scale; for the capacitance membrane gauges the most important uncertainty component is due to the drift of the calibration curves, which may be 10 Pa at full scale (100 kPa), but also 35 Pa in the less good realisations. In the lower pressure range (10 kPa-1 kPa) the zero drift strongly affects the final uncertainty value. For the (1 kPa – 0.1 Pa) range membrane gauges show a stability (the main component of the uncertainty) of 0.8 Pa at 1 kPa and 0,005 Pa at 0,1 Pa over a period of one year. Generally, in that pressure range the membrane gauges are used with their heating system on and show a non-linear behaviour of the calibration factor with pressure.

For (10 Pa e 10^{-4} Pa) pressure range only spinning rotor gauges are available as transfer gauges that show the interesting characteristic of constant gauge factor (1/F) up to 1 Pa while it becomes strongly dependent on the pressure above 1 Pa even if their repeatability is still good.

For pressure lower than 10^{-4} Pa only ion gauges are available of both cold and hot cathode type, but only hot cathode vacuum gauges have been used in several cases for comparisons or in the accredited laboratories. The repeatability and stability of the commercially available gauges may be even of the order of several percent.

Aknowledgements

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Contact Person for Paper:
Dr. M. Bergoglio
m.bergoglio@imgc.to.cnr.it