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Establishment of a force balanced piston gauge for very low gauge and absolute pressure measurements at NPL, India

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Abstract: National Physical Laboratory, the National Metrology Institute (NMI) of India is maintaining Primary standards of pressure that cover several decades of pressure, starting from 3.0E-06 Pa to 1.0 GPa. Among which a recent addition is a Force Balanced Piston Gauge, the non-rotating piston type, having better resolution and zero stability compared to any other primary pressure standards commercially available in the range 1.0 Pa to 15.0 kPa (abs and gauge). The characterization of this FPG is done against Ultrasonic Interferometer Manometer (UIM), the National Primary pressure standard, working in the range 1.0 Pa to 130.0 kPa (abs and diff) and Air Piston Gauge (APG), a Transfer Pressure Standard, working in the range 6.5 kPa to 360 kPa (abs and gauge), in their overlapping pressure regions covering both absolute and gauge pressures. As NPL being one of the signatories to the CIPM MRA, the Calibration and Measurement Capabilities (CMC) of both the reference standards (UIM & APG), are Peer reviewed and notified in the Key Comparison Data Base (KCDB) of BIPM. The estimated mean effective area of the Piston Cylinder assembly of this FPG against UIM (980.457 mm²) and APG (980.463 mm²) are well within 4 ppm and 10 ppm agreement respectively, with the manufacturer's reported value (980.453 mm²). The expanded uncertainty of this FPG, Q(0.012 Pa, 0.0025% of reading), evaluated against UIM as reference standard, is well within the reported value of the manufacturer, Q(0.008 Pa, 0.003% of reading) at k=2. The results of the characterization along with experimental setup & measurement conditions (for gauge and absolute pressure measurements), uncertainty budget preparation and evaluation of measurement uncertainty are discussed in detail in this paper.

1. Introduction

The force-balanced piston gauge is a primary vacuum standard developed by Ooiwa A [1] during 1990s. This new non-rotating force-balanced piston gauge is based upon a mass comparator to determine the force applied to a nominal effective area of 980 mm². The major difference from traditional rotating piston gauges is that the FPG measures the force generated from a given gas pressure against a force balanced load cell to which the piston is attached. Here the attachment has two chambers, one for high pressure (p_h) and the other for low/reference pressure (p_r), separated at the middle of the piston and the "lubricating gas flow" enters the chambers at this point. The non-rotating piston is connected to an electronic dynamometer and centered by means of transient gas flow in the tapered gap between piston and cylinder. The load cell is zeroed with high and low pressure chambers connected. In absolute mode, a precision capacitance diaphragm gauge measures the reference pressure at the low pressure chamber of the FPG. Pressure of gas in the upper chamber is adjusted by an automated very low pressure controller (VLPC) that is equipped with two parallel mass flow

controllers for coarse and fine adjustment and a PC, which calculates and keeps generated pressure on a chosen value.

2. Experimental set up

The setup shown in figure 1 is used for our pressure measurements, except for the dashed line used only for gauge mode measurements. The valves V_9 to V_{12} that are associated with the FPG are pneumatically operated for which a continuous supply of 7.0 bar dry air is required; so a separate pneumatic supply is arranged for the same. Since the UIM at NPL, India is a mercury Manometer, a 1.0 torr differential CDG is used as an isolator cum null indicator (CDG#01) to avoid mercury contamination at FPG Piston Chambers as discussed by Hendricks et al [2]. Other added advantages of this isolation CDG is that it keeps away the necessity of thermal transpiration correction arising due to difference in temperatures of UIM and FPG if directly connected and prevented the FPG's VLPC from setting large pressure changes in large volumes of UIM manifold.

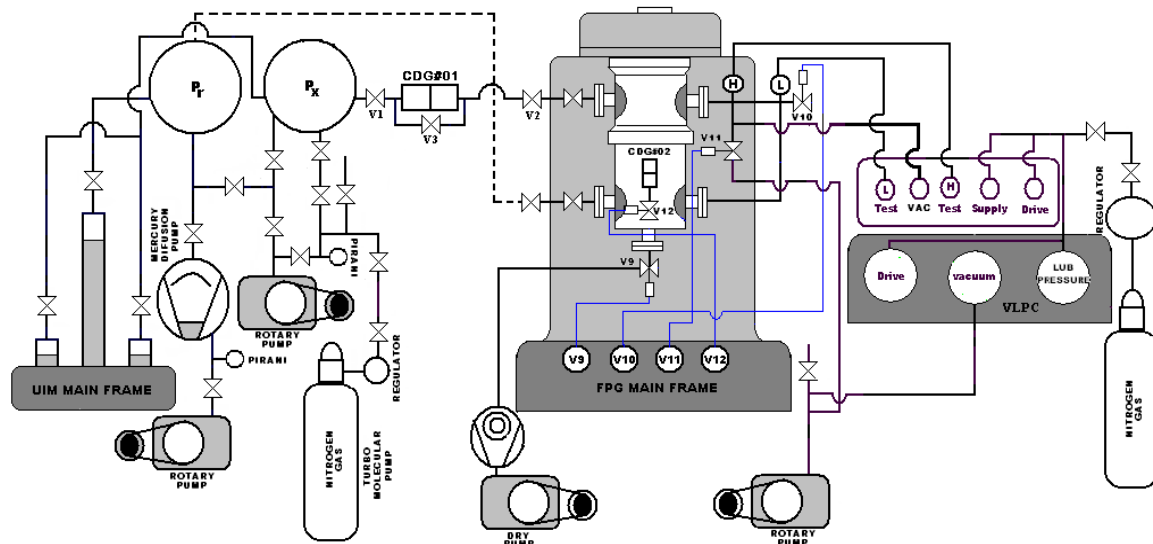


Fig. 1. Measurement Set Up (Dashed line is used only for gauge mode measurements)

All connections are made and secured as shown in figure 1. The UIM and FPG system are initialized for the required mode of measurement. After the whole system attained stabilization, FPG is recalibrated and zeroed. Subsequently the CDG#01 is also zeroed with V_2 , V_3 open and V_1 closed.

With V_3 closed and V_1 , V_2 open condition, both UIM and FPG (VLPC enabled) are pressurized to a selected nominal pressure. After establishing the pressure in the UIM and FPG through continuously monitoring and adjusting the CDG#01 reading close to null, the measurement system was allowed to stabilize. After stabilization, a set of 8 to 10 pressure readings were recorded for FPG, UIM, CDG#01 and drift in FPG zero pressure. Then FPG along with CDG#01 are isolated from UIM (V_1 closed) and zero-pressure readings of both were re-set (V_2 , V_3 open) before proceeding to the next pressure point.

3. Results and discussion

The pressure exerted on the piston of the FPG from the force generated by the mass comparator is given by

$$p_{FPG} = \frac{K_{cal}(N + \delta N_1 + \delta N_2 + \delta N_3)}{A_{0_{20^\circ C}} \{1 + (\alpha_p + \alpha_c)(t - 20)\}} + p_r + p_{head} \quad (1)$$

Where δN_1 , δN_2 and δN_3 are the force correction terms in counts, p_r is the reference pressure at the lower chamber (equal to zero when used in gauge mode), p_{head} is the pressure head, K_{cal} is the

calibration coefficient and N is the load cell reading in counts. The papers by P Delajoud and M Girard [3, 4] may be referred for detailed discussion.

From the data collected, as discussed by Jay Hendricks et al [2] the difference in corrected FPG and UIM reading for absolute and gauge mode are given by equation 2 and 3 respectively as

$$\langle p_{FPG_c} - p_{UIM_c} \rangle = (p_{FPG} - p_{ZC}) - (p_{UIM} + p_{vp_{Hg}} - p_{CDG\#01}) \quad (2)$$

$$\langle p_{FPG_c} - p_{UIM_c} \rangle = (p_{FPG} - p_{ZC}) - (p_{UIM} - p_{CDG\#01}) \quad (3)$$

The plot between measured pressure vs calibration factor (p_{FPG_c} / p_{UIM_c}) of all data collected in both absolute and gauge mode is depicted in figure 2.

3.1. Estimation of effective area of Piston cylinder assembly of FPG

The equation used for effective area estimation in gauge mode is given by

$$A_{0(20^\circ C)} = \left(\frac{m_{cal} g}{N_{cal}} \right) \left(1 - \frac{\rho_{lcal}}{\rho_{mcal}} \right) \left[\frac{(N + \delta N_1 + \delta N_2 + \delta N_3)}{(p_{Std} - p_{head}) \{1 + (\alpha_p + \alpha_c)(t - 20)\}} \right] \quad (4)$$

Where p_{Std} in equation 4 may be UIM measured pressure at 20°C or Piston Gauge measured pressure at 20°C, as in our study both UIM and Air Piston Gauge were used as reference standards. The effective area data thus estimated in the overlapping pressure region (gauge mode) against UIM and Piston Gauge are depicted in figure 3.

The estimated mean effective area of the Piston Cylinder assembly of this FPG against UIM (980.456 mm²) and APG (980.463 mm²) are well within 4 ppm and 10 ppm agreement respectively, with the manufacturer's reported value (980.453 mm²).

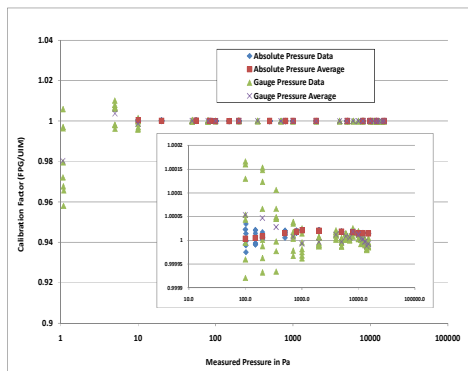


Fig. 2. Data collected in both absolute and gauge mode.

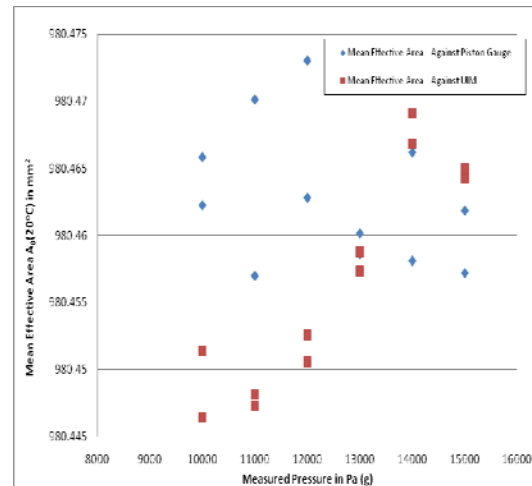


Fig. 3. Effective area of FPG estimated against UIM and APG.

3.1.1. Estimation of expanded uncertainty in Effective Area

Equation 4 is the model equation for the budget preparation. According to P Delajoud and M Girard [3, 4], the expanded uncertainty of effective area was worked out as ± 26.0 ppm at $k=2$, where the uncertainty components due to force correction terms are ignored based on insignificant contribution. As the budget proposed by them is for the cross float method, the major uncertainty component is that of the reference standard, namely the Air Piston Gauge. But in our case, the reference standard is UIM and its measurement uncertainty is notified as $Q(0.0092 \text{ Pa}, 7.2 \text{ ppm})$ at $k=2$ in Appendix – C of KCDB of BIPM [5]. According to our budget prepared including the UIM uncertainty and repeatability, the expanded uncertainty of effective area is worked out to be ± 19.98 ppm at $k=2.025$ (effective degrees of freedom=74).

Quantity (X_i)	Estimate (x_i)	Uncertainty u_{xi}	Probability distribution	Type (A or B)	Degrees of Freedom	Uncertainty Contribution $u_i^2(y)$
Calibration Mass - m_{cal} (kg)	7.79E-01	3.90E-06	Normal	B	∞	2.40E-17
Accln. Due to Gravity g (m/s^2)	9.79E+00	1.96E-05	Normal	B	∞	3.85E-18
Lub air Density ρ_{lair} (kg/m^3)	1.61E+00	2.80E-03	Normal	B	∞	1.21E-19
Cal Mass Density ρ_{mcal} (kg/m^3)	7.90E+03	9.12E+01	Normal	B	∞	5.31E-18
Pressure Medium Density ρ_{mair} (kg/m^3)	4.83E-01	1.06E-07	Normal	B	∞	4.65E-20
Thermal coeff of Piston - α_p ($^{\circ}C^{-1}$)	4.50E-06	2.59E-07	Normal	B	∞	5.79E-19
Thermal coeff of Cylinder - α_c ($^{\circ}C^{-1}$)	4.50E-06	2.59E-07	Normal	B	∞	5.79E-19
Temp.diff from the ref. temp. ($t-t_{ref}$) ($^{\circ}C$)	3.00E+00	8.10E-05	Normal	B	∞	5.11E-25
Measured Pressure- p (Pa)	1.50E+04	5.42E-02	Normal	B	∞	1.25E-17
Fluid Head Correction (Pa)	4.73E+00	1.65E-06	Normal	B	∞	1.17E-26
Verticality (Pa)		1.20E-03	Normal	B	∞	6.15E-21
System Stability (Pa)	0.15	4.33E-02	Rectangular	B	∞	8.01E-18
Load Cell Precision (Pa)		1.52E-02	Normal	B	∞	9.88E-19
Resolution (Pa)	1.00E-03	2.89E-04	Rectangular	B	∞	3.56E-22
Std Dev of Mean Eff Area (m^2)	7.80E-09	2.25E-09	Normal	A	11	5.07E-18
Repeatability (m^2)	1.80E-08	5.69E-09	Normal	A	9	3.24E-17
Total Variance						9.35E-17
Overall standard uncertainty (m^2)						9.67E-09
Effective Degrees of Freedom						74
Relative expanded uncertainty (ppm) at $k=2.025$						19.98

3.2. Estimation of expanded uncertainty of FPG

From the raw data collected, the repeatability for each nominal pressure point is worked out through normalization process and the uncertainty component of repeatability arrived at for absolute and gauge mode measurements are depicted in figures 4. The expanded uncertainty of FPG estimated including the repeatability component for absolute and gauge mode measurements are depicted in figure 5.

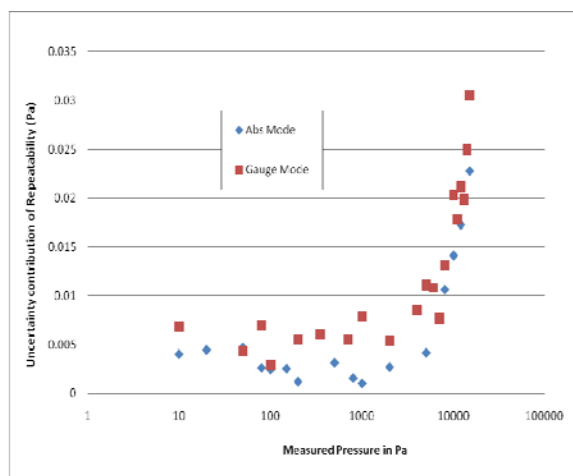


Fig. 4. Uncertainty contribution of repeatability - absolute and gauge mode

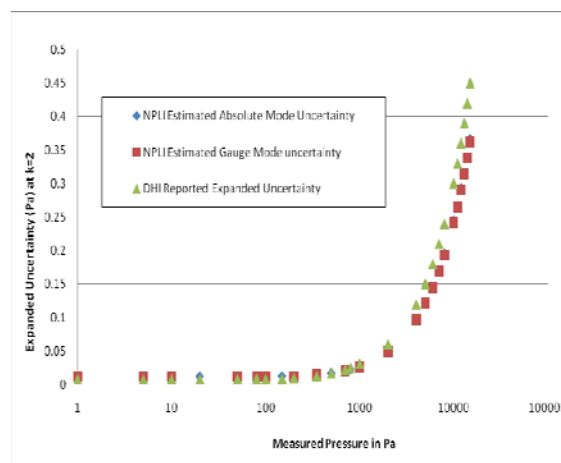


Fig. 5. Expanded uncertainty of FPG - NPLI estimated and DHI reported

Uncertainty budgets were prepared for two different methods namely, theoretical method and the comparison method proposed by D Arun Vijayakumar [6]. The budget prepared using theoretical and comparison methods for absolute and gauge mode are reproduced in tables 2 and 3 respectively.

Table 2: Theoretical method - expanded Uncertainty Budget of p_{FPG} at 15.0 kPa

Quantity (X_i)	Estimate (x_i)	Uncertainty u_{x_i}	Type (A or B)	Degrees of Freedom	Uncertainty Contribution $u_i^2(y)$	
					Independent	Dependent
Calibration Mass - m_{cal} (kg)	7.79E-01	3.90E-06	B	∞		5.63E-03
Accln. Due to Gravity g (m/s ²)	9.79E+00	1.96E-05	B	∞		9.00E-04
Lub air Density ρ_{lair} (kg/m ³)	1.61E+00	2.80E-03	B	∞		2.83E-05
Cal Mass Density ρ_{mcal} (kg/m ³)	7.90E+03	9.12E+01	B	∞		1.24E-03
Pressure Medium Density ρ_{mair} (kg/m ³)	4.83E-01	1.06E-07	B	∞		1.09E-05
Thermal coeff. of Piston - α_p (°C ⁻¹)	4.50E-06	2.59E-07	B	∞		1.36E-04
Thermal coeff. of Cylinder - α_c (°C ⁻¹)	4.50E-06	2.59E-07	B	∞		1.36E-04
Temp.diff from the ref. temp. ($t-t_{ref}$) (°C)	3.00E+00	1.35E-06	B	∞		3.32E-14
Effective Area- A_0 (m ²)	9.80E-04	9.80E-09	B	∞		2.25E-02
Fluid Head Correction (Pa)	4.73E+00	1.23E-06	B	∞		1.51E-12
Ref Pressure (Pa)		3.35E-03	B	∞	1.12E-05 (a)	
		0			0 (g)	
Verticality (Pa)		1.20E-03	B	∞		1.44E-06
System Stability (Pa)	0.15	4.33E-02	B	∞		1.88E-03
Load Cell Precision (Pa)		2.50E-03	B	∞	6.25E-06	1.00E-12
Resolution (Pa)	1.00E-03	2.89E-04	B	∞	8.33E-08	8.33E-08
Repeatability (Pa)	7.21E-02	2.28E-02	A	9		5.20E-04 (a)
	9.64E-02	3.05E-02				9.30E-04 (g)
Total Variance					1.76E-05	3.30E-02
Overall standard uncertainty (Pa)					4.19E-03	1.82E-01
Effective Degrees of Freedom					3.62E+04 (a)	
					1.16E+04 (g)	
					Absolute Value (Pa)	Relative Value (ppm)
Expanded uncertainty at k=2					8.38E-03	24.2 (a)
					7.64E-03	24.4 (g)

Table 3: Comparison Method – Uncertainty budget of p_{FPGc} at 15.0 kPa

Quantity (X_i)	Estimate (x_i)	Uncertainty u_{x_i}	Type (A or B)	Degrees of Freedom	$u_i(y)^2$	
					Independent	Dependent
Repeatability (Pa)	0.0721	2.28E-02	A	9		5.20E-04 (a)
	0.0964	3.05E-02				9.29E-04 (g)
Resolution (Pa)	0.009	2.50E-03	B	∞	6.25E-06	
Mean Calibration Factor	1.000015	1.09E-05	A	14		2.66E-02 (a)
	1.000009	1.13E-05		19		2.88E-02 (g)
Normalized Ref. Std. Reading (Pa)	14999.775	5.42E-02	B	∞	2.12E-05	2.92E-03
	14999.865					
Total Variance					2.74E-05	3.00E-02
					$u_i(y)$	
					5.24E-03	1.73E-01
Absolute Mode Standard Measurement Uncertainty = Q(0.0052 Pa , 0.00116 % of reading)						
Gauge Mode Standard Measurement Uncertainty = Q(0.0061 Pa , 0.0012 % of reading)						
Estimated Effective Degrees of Freedom						18
						24
Estimated Coverage Factor (k) at Degrees of Freedom =18 and 95.45 % Confidence level (a)						2.15
Estimated Coverage Factor (k) at Degrees of Freedom =24 and 95.45 % Confidence level (g)						2.11
					$U_i(y)$	
					1.13E-02	3.73E-01
					1.29E-02	3.81E-01
Expanded Measurement Uncertainty = Q(0.0113 Pa , 0.00248 % of Reading) for k=2.15 (a)						
Expanded Measurement Uncertainty = Q(0.013 Pa , 0.00254 % of Reading) for k=2.11 (g)						

The mean differences of measured absolute pressure between FPG and UIM with error bars especially in the low pressure regions are plotted in between the expanded uncertainty bands of NPL estimated and manufacturer reported expanded uncertainty and the same are depicted in figure 6 and 7 for absolute and gauge mode measurements respectively.

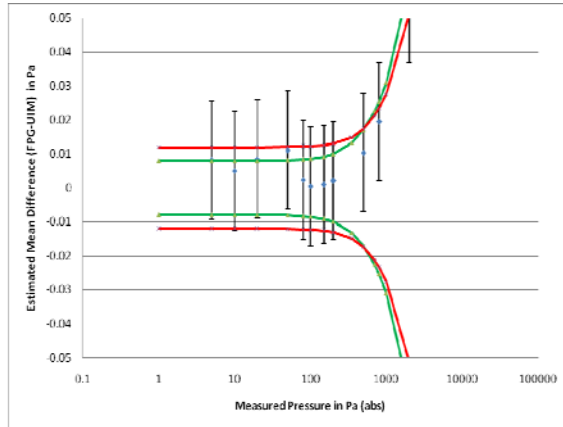


Fig. 6. Estimated mean difference of FPG and UIM with error bars – Absolute Mode

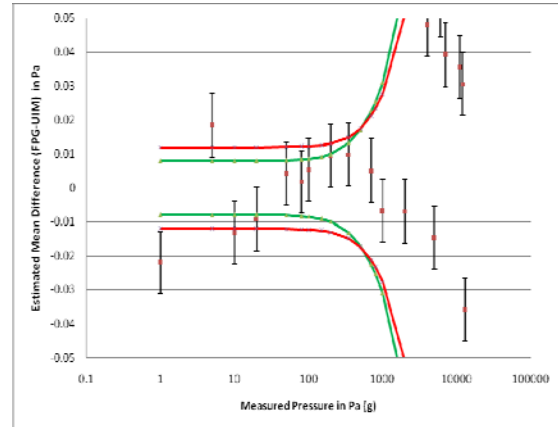


Fig. 7. Estimated mean difference of FPG and UIM with error bars – Gauge Mode

4. Conclusion

The expanded uncertainty of piston cylinder assembly of FPG is evaluated as ± 19.98 ppm at $k=2.025$ (calculated effective degrees of freedom is 74), which is less compared to the manufacturer's reported value of ± 26.0 ppm at $k=2$. This may be attributed to the choice of reference standard, namely the UIM, instead of a simple air piston gauge.

The expanded uncertainty of FPG is evaluated using theoretical method is $Q(0.0084$ Pa, 24.2 ppm of reading) at $k=2$ for absolute mode and $Q(0.0076$ Pa, 24.4 ppm of reading) at $k=2$ for gauge mode measurements.

The expanded uncertainty of FPG is evaluated using comparison method is $Q(0.0113$ Pa, 24.8 ppm of reading) at $k=2.15$ (calculated effective degrees of freedom is 18) for absolute mode and $Q(0.013$ Pa, 25.4 ppm of reading) at $k=2.11$ (calculated effective degrees of freedom is 24) for gauge mode measurements.

The expanded uncertainty evaluated through the budgets prepared using theoretical and comparison methods are found to be very much comparable for both absolute and gauge mode measurements, even when there is some difference observed in the estimated coverage factors. This proves the validity of the comparison method proposed [6].

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