A NEW PRIMARY STANDARD FOR THE REALIZATION OF PRESSURE FROM 10 TO 500 kPa

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1. EXTENDED ABSTRACT

Fluid pressure is a derived measurand (N/m^2) that is realized primarily through manometers and piston gauges at the NMI level. Piston gauges are becoming more useful as primary standards due to the ability to characterize the effective area, the most difficult variable to measure in a piston gauge, with enough confidence and low enough uncertainty to support the needs of an NMI or private laboratories with the need to resolve pressure at the highest level [1].

Fluke Calibration has introduced a piston gauge to be used as a primary standard to realize pressure in both gauge and absolute modes from 10 to 500 kPa called a PG9607. All variables in the pressure equation for a piston gauge were addressed in the design of the piston gauge to ensure the lowest uncertainty in pressure that could be attained.

This paper discusses the design of the piston gauge and the projected uncertainties than can be resolved with a fundamental characterization.

2. PISTON GAUGE DESIGN

The piston gauge is a pressure reference that uses a close fit piston-cylinder, aligned with the direction of gravitational acceleration. The piston or the cylinder floats when the pressure to be measured applied to the area of the piston-cylinder equals the downward force of mass being accelerated by gravity. The PG9607 is of a less frequent design where the cylinder floats and the piston is stationary. When the cylinder floats it is rotated at a rate from approximately 5 to 40 min $^{-1}$ (rpm) so the gyroscopic effect can equalize very small horizontal forces and to center the cylinder on and around the piston. No rotation would cause the cylinder to “tip over” and touch the outer diameter of the piston and sensitivity would be lost. When floating, rotating and stable, the measured pressure is calculated using the equation:

$$\frac{Mg_l\left(1-\rho_{(air)}/\rho_{(mass)}\right)+\pi DT}{A_{(20,0)}\left[1+(\alpha_p+\alpha_c)(\theta-20)\right](1+\lambda P)}+Vac-\left(\rho_{(fluid)}-\rho_{(amb)}\right)g_l h \quad [1]$$

Where:

M	=	Total true mass load	[kg]
g_l	=	Local acceleration due to gravity	[m/s^2]
$\rho_{(air)}$	=	Density of air surrounding masses	[kg/m^3]
$\rho_{(amb)}$	=	Density of ambient air for gauge mode	[kg/m^3]
$\rho_{(mass)}$	=	Average density of mass load	[kg/m^3]
T	=	Surface tension (considered 0 with gas)	[N/m]
D	=	$2\sqrt{\frac{A_{(20,0)}}{\pi}}$ Diameter of the piston	[m]
$\rho_{(fluid)}$	=	Density of the test medium (gas or oil)	[kg/m^3]
h	=	Difference in height between PG7000 reference level and test reference level	[m]
Vac	=	Back pressure in bell jar (absolute with vacuum)	[Pa]
$A_{(20,0)}$	=	Piston-cylinder effective area at 20 °C and 0 gauge pressure	[m^2]
α_p	=	Linear thermal expansion coefficient of piston	[°C^{-1}]
α_c	=	Linear thermal expansion coefficient of cylinder	[°C^{-1}]
θ	=	Temperature of the piston-cylinder	[°C]
λ	=	Elastic deformation coefficient of the piston-cylinder	[Pa^{-1}]
P	=	Pressure applied to the piston-cylinder	[Pa]

In the case of the PG9607 the fluid medium is always gas. The gas can be air, nitrogen or helium. The correction for surface tension can be ignored since the surface tension for gas is negligible. This equation applies to both absolute and gauge modes as long as the term for the density of ambient air is zero in absolute mode and the VAC term is ignored in gauge mode.

Figure 1 shows the main components of the PG9607 platform. The sections that follow discuss these components and the role that improvements in design play in lower uncertainties in pressure.

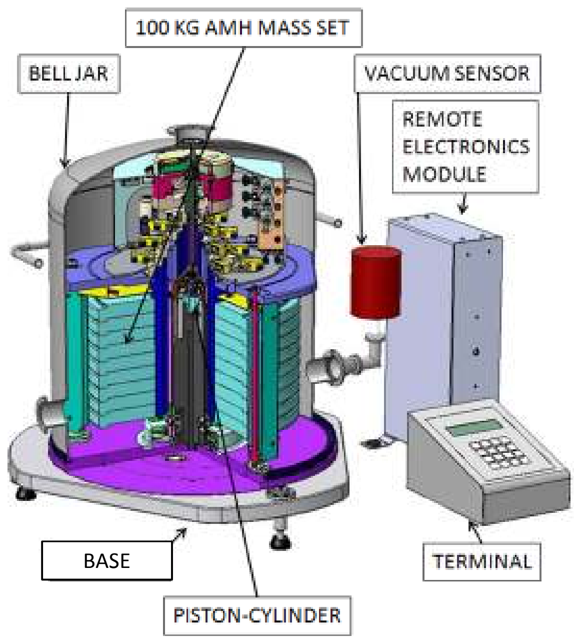

Figure 1 *PG9607 Platform Main Components*

2.1 Piston-cylinder

The piston-cylinder designed to be used with the PG9607 is a nominal 20 cm^2 area, made of tungsten carbide and is a floating cylinder on a stationary piston. A picture of the piston cylinder is shown in Figure 2.

Figure 2 *Picture of the 20 cm^2 area piston-cylinder*

The design is the same used in the PTB project described in [2]. The cylinder mass is 1.3 kg which is larger than the 20 cm^2 cylinder previously available which is 0.7 kg. The larger cylinder wall size allows for better cylinder geometry than the lighter cylinder since there is less deformation from the cap installed on the top of the cylinder. The tolerance on dimensional roundness for both the piston and the cylinder is 100 nm peak to peak. This is an improvement of 150 nm from the previous cylinder design and minimizes uncertainties due to imperfect geometry in a dimensional characterization.

In addition to the improved geometry the thicker walled cylinder also provides for lower elastic deformation. The theoretical deformation coefficient is 4.57 x 10^{-6} MPa^{-1}. This is equivalent to 2.3 parts in 10^6 change in effective are over its pressure range of 500 kPa. If the 2.3 parts in 10^6 were represented completely as a change in gap size, the change in gap from 0 to 500 kPa would be only 28 nm. This is advantageous because the predictions of effective area based on dimensional data must include the pressure distribution at a specific pressure. The dimensional data can only be taken at zero pressure so the assumption that the dimensions are the same at higher pressures should not introduce significant uncertainty to the prediction. A change in gap of 28 nm would not significantly change the integration of the pressure distribution discussed in section 3.1.

The mounting post is a controlled clearance mounting post where a separate pressure of up to 3 MPa can be applied to the inside surface of the piston to control the gap of the piston-cylinder. This can be used for studies of the elastic distortion coefficient. Figure 3 shows a drawing of the piston-cylinder installed in the mounting post.

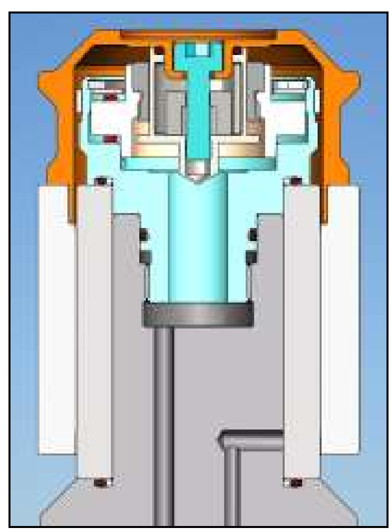

Figure 3 *Drawing of piston-cylinder assembly and mounting post*

2.2 Mass Load

The mass set defined with the PG9607 is a 100 kg binary mass set. Mass loading is automated using the AMH automated mass handler that allows for the exchange of mass while under a vacuum. This is significant since a very low vacuum can be applied while making multiple pressure measurements at different mass loads. The predecessor to the PG9607 would only allow 38 kg of mass to work inside a vacuum. Table 1 shows the breakdown of the masses in the set.

Table 1 *AMH-100 Mass Set*

AMH Mass Set Breakdown
(9 each) main mass of 10.1 kg
6.4 kg binary
3.2 kg binary
1.6 kg binary
0.8 kg binary
0.4 kg binary
0.2 kg binary
0.1 kg binary
0.8 kg mass carrying bell assembly

The minimum mass is 2.1 kg defined by a cylinder mass of 1.3 kg and a mass carrying bell assembly of 0.8 kg. The actual full scale mass load is approximately 105 kg which provides a nominal range of 10.5 to 525 kPa in gauge or absolute mode, or 110.5 to 625 kPa in absolute by addition of atmospheric pressure mode. The resolution of the automated masses is 100 g providing a pressure resolution of 0.5 kPa. There is a mass tray that allows some trim masses to be loaded manually if desired to improve the resolution.

The only time the AMH mass set is handled is when the piston-cylinder needs to be exchanged or cleaned. Since the PG9607 only allows one piston-cylinder size the frequency of handling the mass set is greatly reduced. This significantly improves the stability of the mass set.

2.3 Piston Gauge Base, Terminal and Remote Electronics Module

With the exception of the motor drive assembly used to automatically rotate the cylinder and mass load, all heat producing electronics are kept outside of the base . This was designed this way to ensure there was as little heating of the piston-cylinder and masses as possible.

The Remote Electronics Module, which in the PG9607 predecessor was inside the base, is external to the base and has the following functions:

- Supplies power to the motor drive and AMH
- Reads dual piston-cylinder temperature
- Reads ambient humidity, temperature and pressure
- Connects and reads the residual vacuum sensor
- Connects to the user interface terminal
- Performs all calculations
- Provides remote interfaces for automated control

Because of the inherent precision and expected lower uncertainties of the PG9607, the system was designed with dual platinum resistance thermometers that are installed in the mounting post on opposite sides of the mounting post. There are two reasons for the dual PRT arrangement. One is for redundancy of the same temperature measurement to help reduce type A uncertainties. The other is to better predict the piston-cylinder temperature by measuring each side of the mounting post and account for the possibility of small gradients.

2.4 Residual Vacuum Sensor

When in absolute mode the residual vacuum sensor is measured by a 13.3 Pa (100 mTorr) capacitance diaphragm gauge (CDG). The default vacuum sensor in the predecessor to the PG9607 was a thermal conductivity gauge (TCG). The CDG has a significantly lower uncertainty than the TCG. The REM discussed in 2.3 allows for the configuration of other types of vacuum transducers as well.

The residual vacuum sensor and its display can be seen in figure 4 which is a photograph of the complete system and the complete system with the vacuum bell jar removed. Note that the bell jar does not have to be removed to measure gauge pressure.

2.4 Reference Level

The reference level of the PG9607 is close to the top of the cylinder. This reference level is not easily accessible to compare head height measurements with the AMH loaded and especially with the bell jar on. To help with this a precise height difference between the reference level and the base plate, which is accessible with all components installed, is documented.

Figure 4. *Picture of PG9607 complete (above), and with vacuum bell jar removed (below).*

3. UNCERTAINTIES

As stated earlier in this document the PG9607 can be fundamentally characterized to realize pressure. To accomplish this all the parts of the pressure equation must be fundamentally characterized. This primarily includes mass, gravity and effective area and change of effective area with temperature and pressure.

A PG9607 delivered from Fluke Calibration will not be fundamentally characterized and will have a product uncertainty that is the lowest available from Fluke. But this uncertainty specification is still significantly conservative compared to what is possible if a laboratory seeks the lowest uncertainty in pressure through fundamental characterization. This section concentrates more on what is possible, yet very reasonable for a laboratory to achieve.

3.1 Effective Area

The dimensional fundamental characterization of effective area for a large diameter piston-cylinder is no longer considered to be reserved for just high end national metrology institutes. Many NMIs and non-NMI laboratories are able to perform the proper steps to dimensionally characterize a piston-cylinder and perform a proper uncertainty analysis.

What does seem to be less standardized is a specific procedure to perform the dimensional characterization and also to perform the uncertainty analysis. PTB (Germany) in their efforts to realize the Boltzmann constant have produced what seems to be the most comprehensive method for dimensional characterization [2] and [4].

The method described by PTB [2] involves producing a three dimensional model of the piston-cylinder by combining diameter, straightness and roundness measurements for each piece. This is done by ensuring the measurements connect at strategic points in the z axis and orthogonal position. The amount of measurements to take is not specifically defined, but should ensure that all significant deviations in cylindrical geometry are covered. For the PG9607, 50 mm diameter piston-cylinder there has been enough experience that 10 diameter measurements, 5 on each orthogonal plane, 5 roundness measurements and 4 straightness measurements provide sufficient data to characterize the effective area. Roundness measurements are made at the same z axis coordinates and straightness measurements are made at the four orthogonal positions the diameters were taken. Figure 5 is a graphical representation of this dimensional measurement scheme. This figure shows measurements at 7 z axis locations and is just to demonstrate the concept.

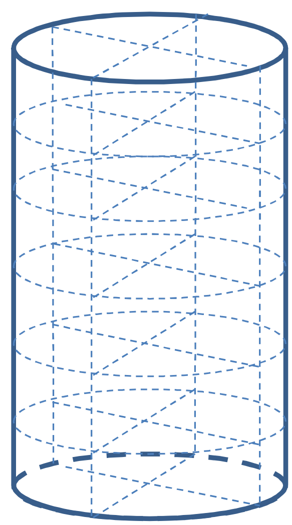

Figure 5. *Example of three dimensional characterization using diameters, straightness and roundness measurements*.

The measurements can then be related by least squares to both the radii needed to calculate the effective area at pressure and also to determine the uncertainty of the effective area based on dimensional measurements. The equations to calculate effective area at pressure are given by Dadson [3] and are:

$$A_P = \pi r_0^{\,2}\left\{1+\frac{h_0}{r_0}+\frac{1}{r_0(P_1-P_2)}\int_0^l (P-P_2)\frac{d(u+U)}{dx}dx\right\} \quad [2]$$

And

$$P=\left[P_1^2-\left(P_1^2-P_2^2\right)\frac{\int_0^x \frac{1}{h^3}dx}{\int_0^l \frac{1}{h^3}dx}\right]^{\frac{1}{2}} \quad [3]$$

Where;

r_0	=	Piston radius at gap entrance	[m]
h_0	=	Gap at entrance	[m]
U	=	Cylinder radius deviation	[m]
u	=	Piston radius deviation	[m]
P_1	=	Pressure at gap exit	[Pa]
P_2	=	Pressure at gap entrance	[Pa]
l	=	P-C engagement length	[m]
x	=	Axial coordinate in engagement length	[m]
P	=	Pressure at axial coordinate	[Pa]

What is advantageous about PTB's dimensional method is that the diameter measurements do not have to be made at z coordinate positions based on the integration method used. Instead, evenly spaced diameter measurements can be interpolated from the three dimensional model developed.

It should be noted that equations 2 and 3 are intended for viscous flow and are not necessarily for molecular or transitional flow that can occur at low pressures in absolute. However the deviations from using this method are considered to be minimal.

A method for calculating uncertainty in effective area based on this method is also proposed by PTB[2] and [4]. The method uses the uncertainty in diameter measurements and the deviations found between the diameter and roundness measurements and the diameter and straightness measurements.

$$u(r_S)=\left\{\left[\frac{u(D)}{2}\right]^2+\delta^2(r_{D-S})+\delta^2(r_{R-S})\right\}^{0.5} \quad [4]$$

$$u(r_R)=\left\{\left[\frac{u(D)}{2}\right]^2+\delta^2(r_{D-R})+\delta^2(r_{R-S})\right\}^{0.5} \quad [5]$$

In these equations u(D) are the uncertainty in diameter measurements, $\delta(r_{D\text{-}R})$ is the difference between the diameter and roundness measurements, $\delta(r_{R\text{-}S})$ the difference of the roundness and straightness and $\delta(r_{D\text{-}S})$ the difference between diameter and straightness measurements. A more thorough discussion of the least squares method and uncertainty analysis is given in [4].

Equations 4 and 5 are unique in that they capture the uncertainty of the full three dimensional model. With the dimensional measurement capabilities of most NMI dimensional laboratories, and the manufacturers tolerance on geometry of the 50 mm piston-cylinder it is not unreasonable to assume an uncertainty of ±2 parts in 10^6 with a 95% confidence can be attained.

3.1.1 Change In Effective Area With Pressure

The PG9607 piston-cylinder uses a theoretical deformation coefficient developed by Fluke Calibration. The value is 4.57 x 10^{-6} MPa^{-1} and is considered to have a conservative uncertainty of 10%. Even if this is considered a rectangular distribution one standard uncertainty is equal to 0.13 parts in 10^6 for 500 kPa and introduces very little uncertainty to the final uncertainty in pressure.

Though this value is theoretical, as was previously stated the PG9607 is a controlled clearance piston gauge and allows for further studies of the validity of the theoretical deformation coefficient.

3.1.2 Change In Effective Area With Temperature

The thermal expansion coefficient for tungsten carbide piston cylinders has been well documented to be
9 x 10^{-6} $^{\circ}C^{-1}$. A conservative estimate in uncertainty of 5% provides a 1 standard uncertainty of 0.22 parts in 10^6 for each degree of correction.

The temperature of the piston-cylinder for a PG9607 is not measured directly. However, the design of the mounting post is such that two platinum resistance thermometers are placed in close proximity to the inner piston wall. During the development of the PG9607 studies were performed by comparing the piston-cylinder mounting post PRTs with a PRT placed in the internal bore of the mounting post where the cylinder would normally be positioned. The agreement was within ±0.03 ˚C.

What helps significantly in the assumption that the piston-cylinder is the same as the mounting post temperature is the stability of temperature. This is one of the primary reasons for designing as much heat producing electronics outside of the platform. Figure 6 is a chart showing the difference between ambient and piston-cylinder temperature in a lengthy test of a PG9607 and another commercial piston gauge offered by Fluke Calibration (PG7601). The tests were performed automatically in absolute mode with an automated mass handler and were performing the exact same test on the same device.

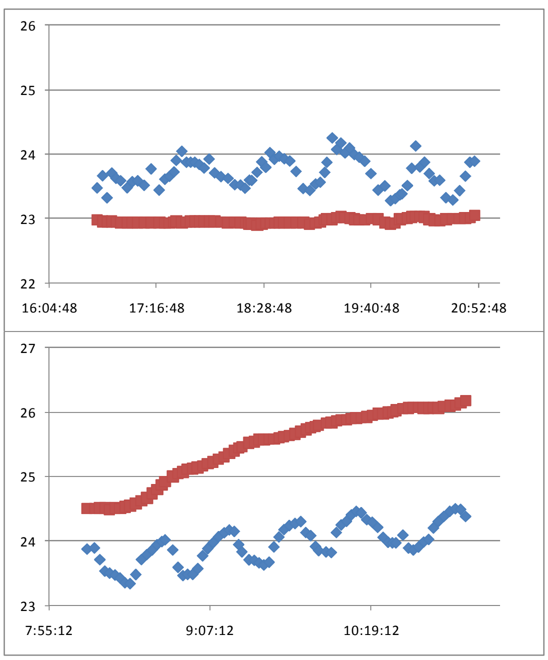

Figure 6. *Piston-cylinder temperature (red) and ambient temperature (blue) of a PG9607 (top) and a PG7601 (bottom) performing the same pressure test.*

The difference between the ambient temperature and the piston-cylinder temperature is significantly higher for the PG7601. Note that this effect is usually only noticed in long automated absolute tests using automated mass handlers and does not jeopardize the uncertainty in pressure for a PG7601. Whereas for the PG9607 there was no detectable ramp in piston-cylinder temperature over the course of the test. This offers confidence that the temperature is stable and has equalized well between the mounting post and piston-cylinder temperatures.

The uncertainty of the temperature of the piston-cylinder depends on the uncertainty of the device making the measurement and the assumption that the piston-cylinder is the same as the temperature of the mounting post. With the capabilities of high end temperature laboratory to calibrate over a very short range of 20 to 25 ˚C it is reasonable to assume an uncertainty of ±0.05 ˚C in piston-cylinder temperature measurement or one standard uncertainty of 0.22 parts in 10^6.

3.1.3 Other Uncertainties Associated With The Piston-Cylinder

Other uncertainties that are normally included and are associated with the piston-cylinder are linearity and stability.

Linearity is an uncertainty associated with the assumption that the effective area is linear over its range. Though the uncertainty of the elastic deformation encompasses much of this, there is still a possibility that the effective area is not linear due to gap modelization effects not mechanical linearity errors in the elastic deformation. One standard uncertainty for this influence is estimated to be 0.23 parts in 10^6.

It has always been difficult to predict what the stability of a 50 mm tungsten carbide piston-cylinder is. Fluke Calibration uses one of these piston-cylinders as a primary reference for approximately 10 years. The three determinations of effective area for this reference have not changed more than 1 part in 10^6. So one standard uncertainty of 0.5 parts in 10^6 is used as an estimate.

3.2 Mass

Uncertainty in mass for pressure metrologists using piston gauges has traditionally been relatively easy to maintain. This was primarily because the uncertainty in effective area was so high that it was not worth putting in the effort to reduce mass uncertainties. But with the expected low uncertainties of a PG9607, mass uncertainty becomes a significant focus. The main contributors to mass uncertainty are the method and reference used to calibrate the mass, the uncertainty in density, and the uncertainty due to changes over time.

One of the most significant attributes of an automated mass handler (AMH) is the fact that there is virtually no wear. The amount of movement required to load a mass is only a few mm. This is significant especially when compared to a manual mass set where it is almost impossible not to scrape masses when loading and unloading along the entire length of the bell assembly. For an AMH used with a PG9607 this would only occur when the piston-cylinder needs to be removed. Because of this an estimated uncertainty due to stability of the masses is ±2 parts in 10^6 and a standard uncertainty of 1 part in 10^6.

The Fluke Calibration mass measurement uncertainty is accredited to an uncertainty of approximately 3 parts in 10^6. The product uncertainty analysis assumes 5 parts in 10^6 and is valid for 2 years for an AMH mass set. However it is reasonable to assume the AMH masses could be calibrated to a level of ±2 parts in 10^6 at 95% confidence.

The density of the AMH mass set is stated to be 7 920 kg/m^3 ± 40 kg/m^3 , k=2, for all the main and binary masses. The cylinder mass has a density of 11 988 kg/m^3 ± 100 kg/m^3 and the mass carrying bell assembly has a density of 4 537 kg/m^3 ± 100 kg/m^3. The higher uncertainties for the bell and the cylinder are due to the fact they are a combination of different materials, primarily tungsten for the cylinder and primarily titanium for the mass carrying bell. For the main and binary masses one standard uncertainty is listed as a relative uncertainty of 0.38 parts in 10^6. One standard uncertainty for the mass carrying bell assembly and the cylinder density is listed in pressure and is 0.023 Pa. Note that these uncertainties are listed in absolute mode and not in gauge mode. This is because the masses are calibrated in ambient air, so the error due to the mass density is greater as conditions change from the calibration. Since the worse case condition are when the ambient air is removed, as is the case in absolute mode, then the uncertainty is the greatest there and insignificant when ambient air is reasonably close to calibration conditions as it is in gauge mode.

An additional uncertainty is added to account for krytox grease lubrication on the threads that connect the mass carrying bell assembly. If the proper procedure is followed to apply the lubrication, approximately 15 to 20 mg of the grease will be on the threads when calibrated. Measurements have shown that the grease will reduce its mass by 5 to 10 mg over the course of one year. Using ±10 mg as an uncertainty in pressure and one standard uncertainty is approximately 0.025 Pa.

One slight concern of the AMH mass set is the fact that the masses loaded are very close to the masses that are unloaded. If there were undefined forces from magnetism this could affect the uncertainty in mass. There has been little study in the area of magnetism or magnetic susceptibility with regards to the AMH masses. Only one AMH mass set has had susceptibility tests performed which passed, however it would be a recommendation to perform the susceptibility tests at the time of mass determination.

3.3 Residual Vacuum

The residual pressure measured inside the bell jar is performed by an MKS 100 mTorr (13.3 Pa) unheated capacitive diaphragm gauge. The uncertainty is expanded to ±0.5% of reading + 0.05 Pa, whichever is greater. History has shown that this specification is easily met without regular zeroing for one year. This is with the condition that the sensor is isolated with a vacuum valve and continually kept under a vacuum.

Since the system operates in absolute without having to break vacuum, and assuming a sufficient vacuum pump is used, the residual pressure is more frequently below
0.5 Pa. In this case one standard uncertainty is 0.025 Pa.

3.4 Type A

Type A uncertainty is estimated from crossfloats maintained to support the Fluke Calibration piston cylinder pressure calibration chain. This was also verified in crossfloats performed with the piston-cylinders manufactured for the PTB Boltzmann constant determination project. All crossfloats at various mass loads were well within ±2 parts in 10^6 and one standard uncertainty of 1 part in 10^6.

3.5 Sensitivity

The uncertainty due to the sensitivity of a piston-cylinder used in a piston gauge is similar to resolution. What is detected as sensitivity is considered a full width uncertainty at k=2 and is therefore reduced by a factor of the square root of 12 to represent one standard uncertainty. The sensitivity has been measured to be 0.005 Pa + 0.5 parts in 10^6. Since it is given as the sum of a pressure and a relative value it is entered twice in the uncertainty budget after being reduced by the square root of 12.

3.6 Combined and Expanded Uncertainty

Table 2 provides an uncertainty budget listing all uncertainties. Many of the influences are not discussed here and are easy to maintain. These include gravity, fluid head correction, ambient air density, resolution and verticality.

Table 2 is structured so that all relative uncertainties are listed above the combined and expanded values and all the uncertainties listed in pressure are below. The uncertainty analysis does not combine the relative and pressure uncertainties. However being careful to avoid any correlation, these uncertainties could be root-sum-squared at specific pressures.

Table 2 *Proposed Uncertainty Budget of a Fundamentally Characterized PG9607 Piston Gauge*

Variable or Parameter	Absolute	Gauge
Full Mass Load	100 kg	100 kg
(relative unc's)	[parts in 10^6]	[parts in 10^6]
Mass (M)	1	1
Local G	0.5	0.5
Air Density	n/a	0.38
Mass Density	0.38	n/a
Head (height)	0.35	0.35
Head (density)	0.23	0.23
Resolution	0.29	0.29
PC Temp	0.22	0.22
Verticality	0.1	0.1
Effective Area	1	1
Linearity	0.23	0.23
Elastic Deformation	0.23	0.23
Thermal Expansion	0.22	0.22
Stability Mass	0.5	0.5
Stability Ae	0.5	0.5
Sensitivity	0.14	0.14
Type A	1	1
COMBINED	2.1 ppm + 0.042 Pa	2.1 ppm + 0.025 Pa
EXPANDED	4.2 ppm + 0.08 Pa	4.2 ppm + 0.05 Pa
(absolute Unc's)	[Pa]	[Pa]
Vacuum	0.025	n/a
Sensitivity	0.002	0.002
Mass Bell Grease	0.025	0.025
Bell Assembly Density	0.023	n/a

The uncertainty budget in table 2 is not a Fluke Calibration product uncertainty budget. The product uncertainty that Fluke Calibration can support for a PG9607 is ±10 parts in 10^6 + 0.1 Pa. The uncertainty budget shown in table 2 is a very reasonable estimate of what an NMI could maintain with this system as discussed throughout this paper.

4. CONCLUSION

Following a strict and well founded procedure and uncertainty analysis for the dimensional characterization of the piston-cylinder should allow the PG9607 to be used as a primary standard for pressure for either an NMI or any metrology laboratory wanting pressure realized at the highest level. The uncertainties should be lower than what is currently used for mercury manometers and extends to a range that is higher than the mercury manometer in absolute and gauge modes.

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