

APPLICATION OF A NEW METHOD FOR THE AUTOMATED CALIBRATION OF VERY LOW GAUGE AND ABSOLUTE PRESSURES IN A COMMERCIAL CALIBRATION LABORATORY

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Abstract - Maintaining traceability for transfer standards in the range of 10 Pa to 5 kPa is critical to many calibration laboratories. However, standards with suitable uncertainty that can also be practically implemented in the typical laboratory have not been available.

A new pressure standard based on piston-cylinder technology has been developed that provides the measurement uncertainty needed to support today's rapidly improving transfer standards, requires no special facilities and runs fully automated calibration sequences. The new standard has been implemented in the DH Instruments, Inc. (DHI) accredited metrology laboratory. Various laboratory transfer standards have been calibrated, in particular capacitance diaphragm gauges, giving new insight into the behavior of these devices.

INTRODUCTION

In our industrial environment, traceability in the measurand of pressure can be achieved from very low pressures on the order of 1×10^{-7} Pa to very high pressures up to 1 000 MPa and beyond. Many types of pressure measuring instruments are supported throughout this range. As technology advances and processes change new requirements emerge in parts of the global pressure range that require better standards.

To support a variety of industries, including in particular the aerospace, semiconductor and pharmaceutical sectors, process measuring instruments and transfer standards in the range of 10 Pa to 5 kPa in both absolute and gauge have reached new levels of performance. Manufacturers of instruments such as capacitance diaphragm gauges, piezo-resistive transducers, quartz resonating transducers and others claim measurement

uncertainty as low as ± 0.05 % of reading. Though standards exist to produce measurements with low enough uncertainty to support these instruments, they are usually at the national measurement institute (NMI) level and are too expensive and difficult to implement for the typical calibration laboratory. This leads to the situation in which the only calibration source for some transfer and even process instruments in this range is at the NMI level.

Table 1 lists standards available at NIST as of the date of this paper for low pressure ranges [3]. These standards are clearly sufficient to maintain traceability for the measurement community but there are some problems when a commercial calibration laboratory tries to support these ranges. Piston gauges are widely used but are limited at

Table 1 – NIST References for Low Pressure Ranges

Range of Pressure	Reference	Reference Uncertainty
10^{-7} to 30 Pa absolute	Dynamic Expansion	0.3 to 2 %
0 to 135 Pa absolute and gauge	Oil UIM	100 ppm + 2 mPa
0 to 360 kPa absolute and differential	Mercury UIM	5.2 ppm + 18 mPa
2 kPa and up absolute and gauge	Piston Gauges	≥ 13 ppm

the low end by the mass of the smallest floating piece. Many of the ranges requiring traceability have full scales that are below the first pressure of a piston gauge. Manometers and other devices may cover the range under that covered by piston gauges but they are not normally available to commercial laboratories and can be very complicated to use.

To meet the need for a laboratory standard in the range under that covered by piston gauges, **DHI** has implemented a newly developed low pressure standard called a Force Balanced Piston Gauge (FPG) [1]. The FPG uses a state of the art, non-rotating piston-cylinder coupled with a force balanced load cell to enable fully automated calibrations from 5 Pa to 15 kPa with a resolution of 10 mPa and an uncertainty of $\pm (25 \text{ mPa} + 30 \text{ ppm})$. This instrument extends the range of the **DHI** metrology laboratory low enough to accommodate increasing demand for calibration in the low absolute and differential ranges.

THE FORCE BALANCED PISTON GAUGE

The FPG includes two major components [Fig. 1]: the pressure measuring portion (left) and the pressure controlling portion (right). The overall system is interfaced with and controlled by a dedicated personal computer running specialized software.



Figure 1 – Force Balanced Piston Gauge System

The FPG pressure measuring portion operates on the piston gauge principle, measuring a differential pressure on a piston by suspending it from a load cell [1]. Differential pressure is measured by connecting high test pressure to the top chamber and reference test pressure to the lower chamber. The difference in pressures acting on the effective area of the piston generates a change in force measured by the load cell. The non-rotating piston is attached to the load cell by a linkage and is centered in the cylinder by a small lubrication gas flow through a double conical cylinder [Fig. 2] [2].

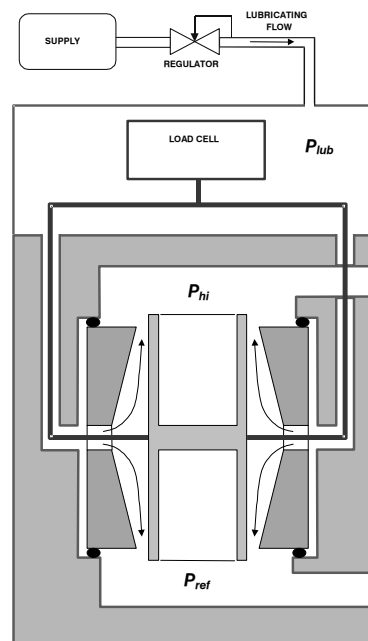


Figure 2 – FPG Pressure Measuring Portion

Zero and span of the load cell are easily verified and maintained by internal calibration. Zero is set through a tare of the system with the upper and lower chambers tied together. Internal calibration of span is used to compensate for small changes that may result from changes in operating conditions. Internal span calibration is performed with a dedicated internal reference mass and an automated loading mechanism. Warnings in the software trigger the user when conditions have changed enough to merit tare or internal calibration.

The pressure controlling portion of the FPG is in a separate enclosure. The pressure control principle is the adjustment of flow across different flow restrictions. The upstream side of the restriction is connected to the upper FPG pressure chamber. The downstream side is connected to the lower FPG pressure chamber. The lower chamber is also connected to either atmosphere for gauge mode operation or an independent vacuum source for absolute mode operation. Switching ranges or modes is accomplished with pneumatically operated valves and is fully automated by the system.

Pressure control precision is a function of the pressure control range and varies from $\pm 0.01 \text{ Pa}$ at the lowest gauge range to around $\pm 0.40 \text{ Pa}$ for 15 kPa ranges in gauge and absolute.

All metrological characteristics and control logic are located in component embedded software. The user interface and system controls, however, are provided by the system PC based software that is integral to the use of the unit [Fig. 3]. The user interface allows for monitoring of pressure indicated by the standard, selection of mode, selection of pressure control range, control of pressures and fully automated calibration of up to five DUTs through serial or GPIB data acquisition.

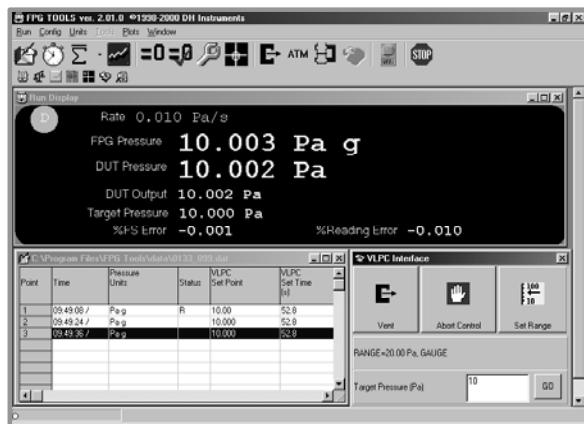


Figure 3 – System Software

Automated Testing

Automated testing is an integral part of the FPG platform. In order to perform an automated test the device under test (DUT) should be capable of communications through either serial RS-232 or GPIB (IEEE-488) interfaces. In many instances devices either have one of these communications ports or an analog output which may be measured by a digital multimeter (DMM) with appropriate interface. With the device connected to the system as shown in Figure 4 the pressure indicated by the DUT may be displayed and compared on the FPG user interface.

Automated test sequences are created, saved and executed within the system software. Significant test sequence functions include:

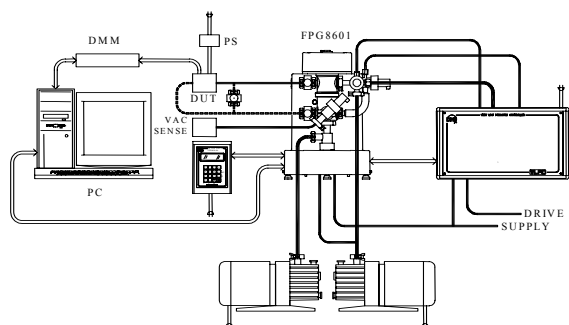


Figure 4 – FPG system and DUT connections

- Internal Calibration – The FPG may execute a tare or internal load cell span calibration at any time during a test sequence.
- Hold Limits – Hold limits specify the upper and lower limits within which pressure must remain for the system to be considered “ready.”
- Dwell Time – The delay after a “ready” condition is achieved before data taking begins is user controlled, allowing the DUT and the system to come to equilibrium.
- Averaging – Average, maximum, minimum and standard deviation of pressure indicated by the FPG and the DUT can be taken over a user specified time interval.
- Test Files – Files may be created and saved for each DUT profile, including up to 10 cycles of a test sequence with up to 100 points per sequence.
- Semi-Automatic Testing – If a digital interface is not available it is still possible to use the FPG automated test sequence functions. In this case the operator is prompted for each DUT pressure.

Uncertainty

Measurement uncertainty of the FPG consists of absolute and relative uncertainties [6]. Principle among the relative uncertainties is the uncertainty in the effective area, followed by uncertainty in local gravity and the internal calibration mass [Table 2]. The most significant absolute uncertainty is the linearity of the load cell, followed by the measurement of vacuum in the lower chamber (absolute mode only). The measurement uncertainty depends on the operating mode due to measurement of pressure in the lower chamber in absolute mode. In gauge and absolute differential modes the uncertainty is $\pm (20 \text{ mPa} + 30 \text{ ppm})$. In absolute mode the absolute component of the uncertainty is increased to give a value of $\pm (25 \text{ mPa} + 30 \text{ ppm})$.

Table 2 – FPG8601 Uncertainty Budget

Variable or Parameter	Gauge Differential Mode	Absolute Differential Mode	Absolute Mode
Full Mass Load	1.5 kg	1.5 kg	1.5 kg
Relative Uncertainties	ppm	ppm	ppm
Cal Mass	2.5	2.5	2.5
Local Gravity	2.5	2.5	2.5
Air Density(lube)	0.15	0.105	0.105
Cal Mass Density	2.09	0.58	0.58
Head (height)	0.35	0.35	0.35
Head (density)	0.23	0.23	0.23
PC Temp	0.5	0.5	0.5
Verticality	0.1	0.1	0.1
Effective Area	13	13	13
Linearity	1	1	1
Elastic Deformation	0	0	0
Thermal Expansion	0.25	0.25	0.25
Stability Mass	0.1	0.1	0.1
Stability Ae	2	2	2
Sensitivity	0.114	0.114	0.114

Absolute Uncertainties	mPa	mPa	mPa
Resolution	2.89	2.89	2.89
Vacuum	0	0	5
Linearity	10	10	10

Combined	13.84 ppm + 10.41 mPa	13.69 ppm + 10.41 mPa	13.69 ppm + 11.55 mPa
Combined & Expanded (K=2)	27.67 ppm + 20.82 mPa	27.38 ppm + 20.82 mPa	27.38 ppm + 23.10 mPa

When using the FPG as a reference the uncertainty in the test includes the combined and expanded uncertainty in Table 2 and also includes a Type A uncertainty. This Type A uncertainty depends upon the type of test being performed, the environment of the standard and the capabilities of the pressure controller. FPG system software is able to quantify averages and standard deviations measured by the FPG and the DUT to support a Type A uncertainty analysis.

CALIBRATION

Calibration of the FPG, as with all piston gauges, is performed by calibrating, or determining, the fundamental parts of the pressure equation, i.e. effective area, mass/force balance and gravity. Also a calibration is performed on all ancillary measurement devices used for corrections on the FPG such as mounting post platinum resistance thermometers, internal lubrication pressure transducer and the device used to measure the reference pressure on the low side of the FPG in absolute mode.

Force Balance and Mass Calibration

Because the FPG can zero the force balance with the two pressure ports open to each other and uses an internal calibration mass for real time span corrections, it is only necessary to calibrate the force balance for its linearity and repeatability. This is accomplished by replacing the piston-cylinder assembly with a calibration bracket that is coupled to the same location as the piston-cylinder assembly. Reference masses are placed in increments to the full scale of the balance for its as received condition. Adjustments are made as appropriate and the as left condition determined and recorded for the balance.

The internal calibration mass is calibrated like any other working standard mass and is directly traceable to NIST. However it has the advantage that once it is determined, it is placed back into a controlled environment where it is not touched by human hands until its next calibration or maintenance, improving the probability that it will not change between calibrations.

Mounting Post Platinum Resistance Thermometers (PRTs)

An FPG utilizes two PRTs to measure the temperature of the piston-cylinder. This temperature measurement is also used to determine the temperature of the gas media for pressure head and thermal transpiration corrections.

The PRTs are calibrated by comparing them to two standard PRTs (SPRTs) maintained in a temperature bath. The SPRTs are traceable to ITS90 temperature measurements through an approved accredited vendor.

Lubrication Pressure Transducer

A dedicated transducer monitors lubrication pressure. Changes in the lubrication pressure cause changes in the buoyant force on the balance, piston and piston

carriage that require compensation. The pressure transducer is calibrated from 20 to 200 kPa to cover the two lubrication pressures of 40 and 140 kPa

Absolute Reference Vacuum Sensor

The sensor that is used to monitor reference pressure in absolute mode is a 13.3 Pa [100 mTorr] capacitance diaphragm gauge (CDG). The CDG is read by FPG internal electronics and used to add the residual pressure to the calculated pressure of the FPG. Since the CDG is only used below 1 Pa in measuring the residual pressure, the sensor is expected to have an uncertainty of $\pm 1\%$ of reading.

The calibration of the reference vacuum CDG is performed by first zeroing the sensor using a turbo molecular pump referenced to an ion gauge. Once this is done the slope can be determined by calibrating it with the FPG that is using an alternate vacuum sensor to determine the slope of the CDG through 13 Pa. Knowing the slope and zero of the transducer is sufficient for its intended purpose to measure the residual absolute pressure in the lower chamber of the FPG with uncertainty of $\pm 1\%$.

Effective Area

Once all other component calibrations have been performed, the effective area of the FPG is determined by crossfloat with a **DHI** 5kPa/kg (50 mm) calibration chain reference. The FPG is zeroed and the crossfloat is performed over the range of 5 to 15 kPa in gauge mode.

Though the effective area is determined over a range of pressure, it is expected to be constant over the entire range, whether it is in gauge or absolute within the uncertainty in the effective area. Normally, the effective area is determined in gauge mode and then checked in absolute. However, it is possible to determine an effective area for each mode. There is an entry in the operating software for the effective area in each mode, but at this point no significant difference in effective area between gauge and absolute modes has been observed within the uncertainty in the effective area.

Verification of Calibration

The FPG is verified with a new effective area by crossfloating again to a reference piston-cylinder from the **DHI** calibration chain. This can be performed directly in gauge mode or by comparison through a differential transducer in gauge or absolute mode. Though **DHI's** accredited scope includes the range covered by the FPG8601, the FPG has yet to

be included in a formal proficiency test or round robin. This is not surprising considering the lack of availability of standards in this range. As other laboratories implement their own FPG systems, opportunities will arise for proficiency testing and round robins with more participants in this range.

IMPLEMENTATION

In January of 2001, **DHI** implemented an FPG8601 in the metrology laboratory. With the implementation of the FPG8601, **DHI** was able to utilize a starting pressure for its accredited scope of 10 mPa, the resolution of the FPG8601.

Operating Environment

No special facilities were required to put the FPG in operation in the metrology laboratory. The laboratory ambient temperature, usually $23 \pm 0.5\text{ }^{\circ}\text{C}$, is sufficient not to introduce instability in pressure control. Air currents are less significant than with a traditional piston gauge in gauge mode because the components sensitive to air drafts are enclosed.

It is advisable to place the FPG on a surface that is not exposed to excessive vibration. Since the vibration sensitive component of the FPG is a force balance, the same vibration criteria used for mass calibration laboratories that use high precision mass comparators apply to the FPG.

When calibrating ball gauges, or other low pressure deadweight testers of similar type, it is necessary to minimize rapid changes in ambient pressure in the laboratory. Changes can be caused by laboratory doors opening or large air currents. The reason for this is that when these instruments are calibrated there is no low pressure reference port to connect to the low side of the FPG. The calibration must be done with the low port open to atmosphere where rapid changes in ambient pressure induce fluctuating pressure differences between the FPG and the device.

All connecting and manifold hardware are clean and dedicated to the FPG calibrations. Care is taken to use hardware with sufficient conductance not to create un-quantified differential pressures. Leaks are less of a problem than in higher pressure calibrations but care is taken to ensure connections are leak free.

Devices Under Test

The added range of the FPG8601 in the **DHI** accreditation scope allowed the laboratory to support the calibration of a number of new instruments. These include, but are not limited to:

- Capacitance Diaphragm Gauges – 13 330, 1 333, 133 and 13.3 Pa [100, 10, 1 and 0.1 Torr], absolute and differential modes
- Low Pressure Strain Gauges – absolute and gauge
- Thermal Conductivity Gauges
- Ball Gauges – up to 15 kPa [60 in H₂O] in gauge and differential modes

DHI has calibrated ball gauges for many years but was not able to support them down to 1 000 Pa [4 in H₂O] with a 4:1 test uncertainty ratio until the implementation of the FPG8601. Also, with the FPG, **DHI** is also able to calibrate new deadweight testers similar to the design of the ball gauge that reach pressures as low as 6.4 Pa [0.025 in H₂O].

The log-log chart in Figure 5 presents the uncertainty of the FPG8601 in percent of reading. Since the uncertainty is a combination of a pressure constant and a relative value the uncertainty expressed in percent of reading increases as pressures decrease [Fig. 6]. The uncertainty is less than $\pm 0.005\%$ of reading down to 500 Pa and is less than $\pm 0.05\%$ of reading down to 50 Pa. Many test devices with ranges below 50 Pa have uncertainty specifications of $\pm 1\%$ or greater allowing good test uncertainty ratios at lower pressures as well.

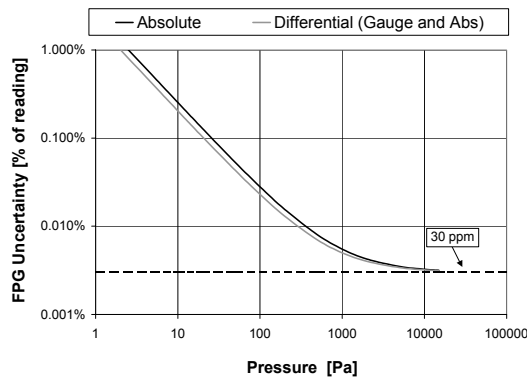


Figure 5 - FPG8601 Uncertainty Vs. Pressure

Generally capacitance diaphragm gauges can be calibrated down to the 133 Pa [1 Torr] range in either absolute or differential. 13.3 and 6.6 Pa [100 and 50 mTorr] ranges in absolute or gauge are not currently supported to manufacturers' specifications with the FPG8601, but are tested with reasonable results to support the primary function of the devices. Considering the lack of standards available in this range and possibility that manufacturers specifications may represent reproducibility more so than measurement uncertainty, many customers choose to have these low range devices calibrated by the FPG8601.

RESULTS OF CALIBRATIONS

Since the initial implementation of the FPG in the **DHI** accredited calibration laboratory, there have been many opportunities to calibrate and observe various low range pressure devices. A sample of representative calibrations is given below.

Gauge Mode Calibration

Gauge mode calibration with the FPG results from control of pressures over live atmosphere. Figure 6 shows the residual error (difference between calibrated DUT pressure and FPG pressure) for three verification sequences. For these calibration runs the FPG executed an automated sequence with a 60 second dwell time and a 30 second data averaging time. As each pressure is ready in less than 90 seconds, the total test time for each 19 point run is around 50 minutes.

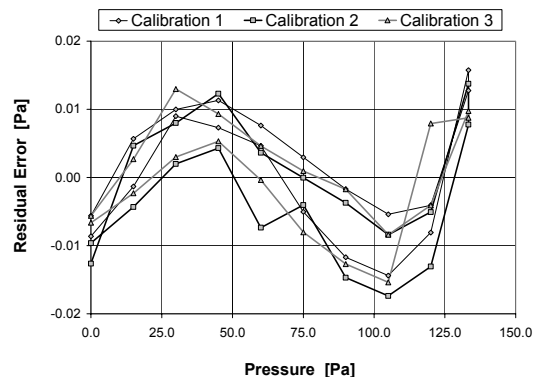


Figure 6 – Gauge Mode Calibration of a 133 Pa [1 Torr] CDG

Note that the repeatability of each point on the calibration is within 0.01 Pa. Note also that the shape of the characteristic “S curve” is seen clearly in every calibration run even though the resolution of the FPG is 0.01 Pa and the peak to peak magnitude of the shape is only two to three times that value. This demonstrates both the consistency of pressure control and the power of automated data averaging during a calibration. This type of performance is not possible with manual data recording.

Figure 7 shows another example of gauge mode calibration. A new low range differential pressure controller manufactured by **DHI** uses a ± 15 kPa differential quartz transducer [7]. By reversing the connections to the upper and lower chambers of the FPG halfway through the calibration, a span of up to 30 kPa may be calibrated by the FPG. The device is calibrated in three ranges, giving optimum performance in each range based on the higher degree of linearity

in the center of full scale span. The results shown are for the three ranges of calibration. Use of this calibration technique with the FPG may allow an improvement in the specification of this device from the current manufacturer's specifications.

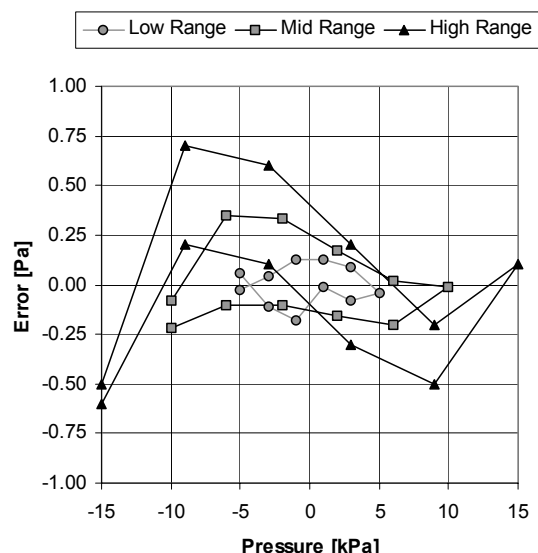


Figure 7 – Gauge Mode Calibration
of ± 15 kPa Quartz Transducer in a Pressure Controller

Absolute Mode Calibration

A 1 333 Pa [10 Torr] absolute CDG is a device commonly calibrated in the **DHI** metrology laboratory. Although many customers require only an 11 point calibration (20 % increments), some need greater density for characterization. Figure 8 shows the results from a 30 point calibration.

Note that the shape and transducer hysteresis can be seen very clearly in the data. This calibration run took 143 minutes to complete with 60 second dwell and 30 second averaging.

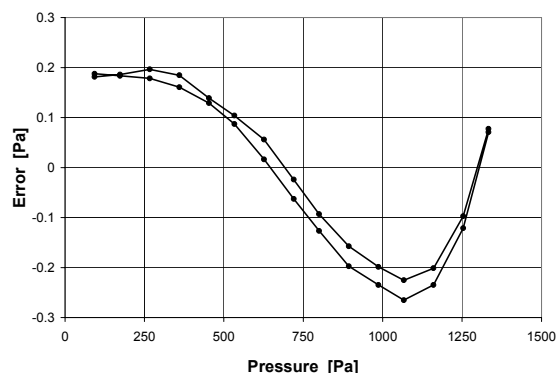


Figure 8 – Absolute Mode Calibration
of a 1 333 Pa [10 Torr] CDG

Thermal Transpiration

A discussion of calibration and results for absolute pressure devices under 1 kPa full scale would not be complete without a mention of thermal transpiration. Detailed descriptions of the phenomenon are available in literature [4, 5]. For purposes of this discussion transpiration is an effect that creates a pressure difference between a heated device and another (unheated) location in the system. This phenomenon manifests itself most significantly in pressures below 150 Pa. For reference, Figure 9 shows the difference in pressure which would result from a typical 45 °C heated CDG with a 4 mm minimum ID calibrated by a 23 °C device with nitrogen gas as the pressurized medium. Note that the peak difference (at around 10 Pa) is on the order of 0.1 Pa.

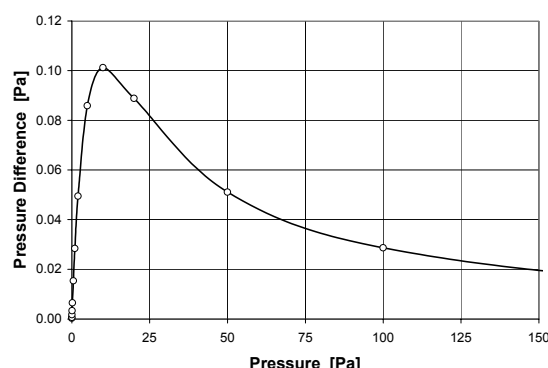


Figure 9 – Typical Transpiration Pressure Difference

It is important to be able to quantify the effect of transpiration in an absolute calibration. The FPG system is capable of correcting for transpiration real time with a user input of CDG head temperature and minimum diameter. The transpiration correction may also be disabled or the user may perform their own correction using data included in the extensive FPG output log file. The **DHI** metrology lab reports pressure with a transpiration correction on request.

The method of calculating the thermal transpiration correction in the FPG is based on the equations outlined by Poulter, et al [4], although the specific coefficients which are used for the calibration gas may be input by the user based on published values, user research or other findings [5].

Figure 10 shows a comparison graph of a 133 Pa [1 Torr] heated CDG calibrated with the FPG. A curve shows the results of the calibration without the transpiration correction. A second curve shows the much more linear behavior indicated by taking into account the transpiration effect. It appears vital, particularly with the higher end ± 0.05 % devices, to be able to quantify and include the transpiration effect during calibrations.

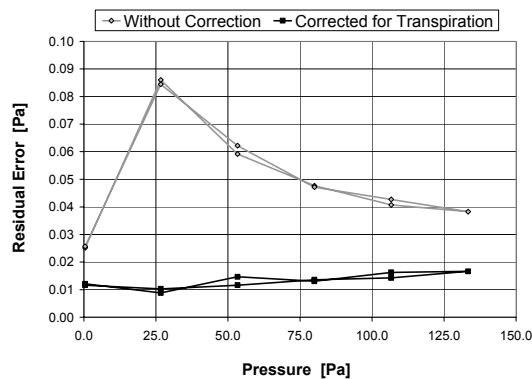


Figure 10 – CDG Calibration With and Without Transpiration Correction

CONCLUSION

Aside from the obvious benefit of enabling calibration of more instruments, one of the greatest benefits to implementing the FPG8601 at **DHI** is the ability to automate calibrations. Many test devices, particularly CDGs, have digital or analog output. With either of these the FPG software allows full automation of calibration. This has allowed the calibration laboratory to support a significant addition to its range of service without a significant increase in man power. Calibrations can be setup using multiple test points to expand the laboratory output or to perform long term evaluations that would normally be very time consuming. Automation also removes human biases that are typically present in manual calibrations, providing more consistent results.

Even when the test instruments offer no remote output the automation of the FPG offers significant benefits. Once a test instrument is connected and a calibration begins, the reference is entirely controlled by the computer. No manipulation of the FPG is required. Technicians run the calibration from menus provided on the computer display. Predefining points, dwell and stability times in test files allows semi-automatic calibrations to experience most of the same benefits as fully automated calibrations.

The other significant benefit of the FPG is its robust nature. Since implementation in the laboratory, little maintenance has been required. This has much to do with the design of the FPG. All sensitive components are protected from outside influences because they are enclosed in the FPG housing. It is almost impossible to contaminate the piston-cylinder because the lubrication gas is controlled by the FPG not the measured pressure which is exposed to a possibly contaminated test instrument. Because of this, cleaning the piston-cylinder assembly is seldom required.

Having the FPG as a standard has generated more confidence in the calibrations performed at the very low end of the range of traditional piston gauges used in the laboratory, which do not always perform as well at the lowest part of their range. The measured agreement in the overlapping range between the FPG and the lowest ranges of piston gauges increases confidence in both methods.

The FPG8601 is now an irreplaceable standard in the **DHI** calibration laboratory. It is in continuous use supporting new measurement devices and has offered new insight into the performance of low pressure transfer standards. With data obtained like that presented in the calibration results section of this paper, **DHI** continues to gain experience with these transfer standards.

There is still opportunity and motivation for further improvement in the **DHI** accredited scope to reach lower pressures. The support for CDGs in the 6.6 and 13.3 Pa [50 and 100 mTorr] ranges and very low pressure deadweight testers will demand improvement in standards. Increased resolution (below 10 mPa) and decreased uncertainty on the FPG, particularly the absolute uncertainty value of 20 or 25 mPa will improve this device to meet those requirements.

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