Development of High Pressure (110 MPa) Gas Calibration Service at NIST

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Introduction

High gas pressures are routinely measured in military and industrial applications. Some of these applications require high accuracies which dictate state-of-the-art measurement systems for their support, such as precision gas piston gauges. While high-pressure oil calibration capability has existed at the National Institute of Standards and Technology for pressures as high as 280 MPa for a number of years, the capability for calibrating high-pressure gas piston gauges above 16 MPa was not available before the initiation of this project. The goal of this project, which was sponsored by the Department of Defense Calibration Coordination Group, was to develop a high-pressure gas calibration service (9 MPa to 110 MPa) at NIST which would provide direct traceability for new high-pressure gas piston gauges to National Standards.

Apparatus and Test Method

To develop this service, NIST proposed to characterize (calibrate) two commercially available high-pressure gas piston gauges using oil piston gauges that are traceable to National Standards. The two high-pressure gas piston gauges (PG 87 and PG 79), which differ considerably in design, would then be used to calibrate customer gas piston gauges. Given the maximum pressure of the gas gauges (110 MPa), it was determined that an existing 139 MPa oil piston gauge, PG-41, would be used to calibrate the high-pressure gas piston gauges. To effect a calibration of a gas piston gauge with an oil piston gauge requires some type of suitable interface which would minimize errors associated with the comparison. Initially a direct gas-oil interface (VLI) was chosen, but this approach was later abandoned due to operational problems; for example, an average of an hour was required to reach equilibrium at each data point as the pressure was increased, and one and a half to two hours were required after each data point as the pressure was lowered. These long equilibration times were caused by the gas going into and coming out of solution in the oil. As the gas came out of solution, care was required to minimize the formation in the VLI of gas bubbles which could be observed as frothing of the oil in the sight glass. Ultimately, a calibrated differential-pressure (DP) cell was selected as the fluid separator. The DP cell uses a thin metal diaphragm to physically separate the gas and the oil. Implementation of the DP cell eliminated gas invasion into the oil and the associated measurement problems.

PG 41 was used to characterize the two gas piston gauges using a cross-float calibration technique which is thoroughly described elsewhere[1] and is schematically shown in Fig. 1. In this technique, equal pressures of oil and gas are generated and measured with the respective piston gauges. The DP cell is used as a null meter to match the gas and oil pressures. When a calibrated DP cell is used in a two-fluid cross-float, several effects need to be considered. The operation and calibration of the

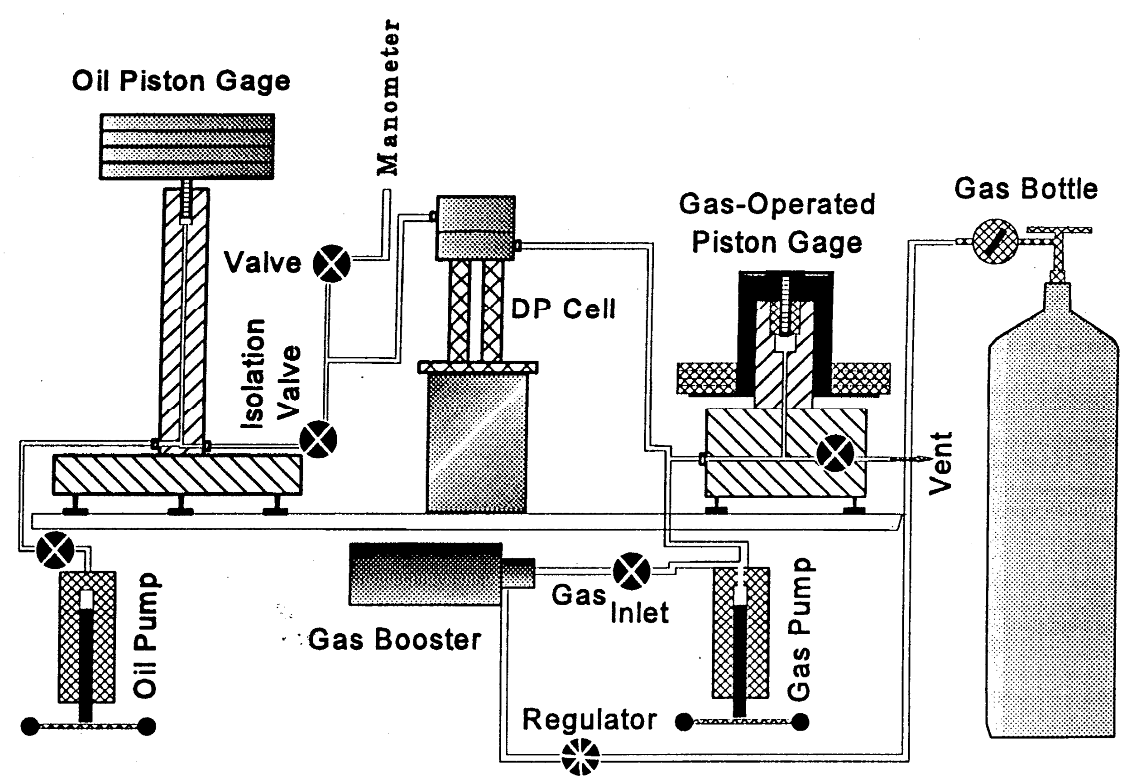

Fig. 1. Schematic of high-pressure gas calibration system

DP cell are discussed in Ruska's* user's manual for Model 2413/2416 Differential Pressure Null Transducer and Indicator. Using the DP cell as a fluid separator requires great attention to detail. In some manner, the liquid system must be opened to atmosphere at a point level with the diaphragm in order to "zero" the diaphragm. An open-tube manometer consisting of a valve with an attached glass tube serves the purpose well. With the manometer valve and the gas system both open to atmosphere, the liquid is adjusted to stand in the tube at the height of the diaphragm. Under these conditions, the pressure across the diaphragm (ΔP) is zero. The electrical circuit, with the sensitivity set at maximum or whatever value has been chosen, is then adjusted so that the meter indicates zero ΔP. As the manometer valve is closed, the pumping action of the stem causes the liquid to rise slightly in the tube and the meter point to deflect. The deflection is a normal one which results from the disturbance of the liquid in the tube and can be ignored. Before the measurement is begun, the sensitivity is reduced by placing the shunt switch in the "on" position. The shunt switch reduces the gain of the circuit by a factor of approximately 1000. First the liquid pressure and then the gas pressure are raised in the manner described above. As the gas pressure becomes approximately equal to that of the liquid, it will be observed that the two pressures will rise simultaneously as the increase in gas pressure is continued. At this time, the diaphragm is being forced away from the lower cavity surface by the gas. The displacement of the diaphragm increases the pressure in the liquid system. Although the two pressures are approximately equal, a signal will not appear on the meter until the gas pressure is within 2 psi of the liquid, since this figure is the limiting value of the indicated differential pressure. Some liquid must be withdrawn from the differential pressure cell by adjusting the oil pump (see Fig. 1.), allowing the diaphragm to move toward the center of the cavity whereupon the meter signal will approach a zero indication. With an oil piston gauge in the system, the pressure in the oil may build up high enough to float the weights. With a slight excess of gas pressure, the diaphragm will then move freely across the cavity; the weights will then be seen to rise rapidly. After the sensitivity is increased by placing the shunt switch in the off position, the two pressures may be brought to a satisfactory balance.

In reducing the pressure, the procedure is reversed. The gas pressure is first reduced and then followed by the liquid pressure. At the conclusion of the measurement, some time must be allowed for the transducer to recover before the zero-pressure conditions are verified. Particularly, if the last reduction in pressure is 100 MPa or greater, the recovery period may be as long as five to ten

*Use of a particular manufacturer's instrument does not constitute an endorsement of the instrument.

minutes.

Results

Three cross-floats were performed using PG 41 to calibrate PG 87, a Ruska** high-pressure gas piston gauge that uses helium as the pressure transmitting fluid. A calibrated DP cell was used as the fluid separator in all three cross-floats. The first two cross-floats were performed sequentially, and then the apparatus was disassembled. The apparatus was reassembled and the reference level heights were re-measured and the cross-float was repeated. This disassembly was done to determine the reproducibility of the measurement. The data from the cross-float after disassembly was consistent with the previously determined values. The measured effective area of PG 87 is shown in Fig. 2 as a function of generated pressure. The measured effective area was well represented with a quadratic fit to the data, as shown in Fig. 2. The gas gauge operated extremely well and maintained good sensitivity through the DP cell over the entire range of 9.7 MPa to 103.5 MPa The uncertainty due to random effects was 5 ppm, representing two standard deviations of the predicted values.

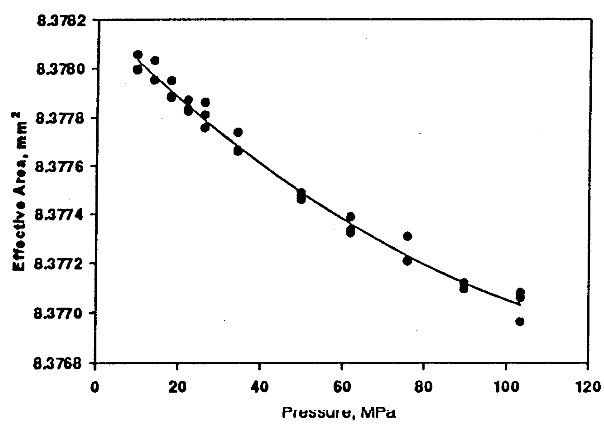

Fig. 2. The effective area of PG 87 as a function of pressure as measured using PG 41. The solid line represents a quadratic fit to the data.

Three cross-floats were performed using PG 41 to calibrate PG 79, a DHI** high-pressure gas gauge which operates with gas, but is oil lubricated. Again the DP cell was used as the fluid separator with helium as the pressure medium, and the previously described tests on PG 87 were repeated for PG 79. In particular, the process of disassembling and re-assembling the setup was performed and similar results were obtained. As in the earlier tests, the gauge operated extremely

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well and had good sensitivity through the DP cell. The measured effective area of PG 79 is shown in Fig. 3 as a function of generated pressure. The measured effective area was well represented by a linear fit to the data, as shown in Fig 3. The uncertainty due to random effects in the cross-float was 3 ppm, representing two standard deviations of the predicted values.

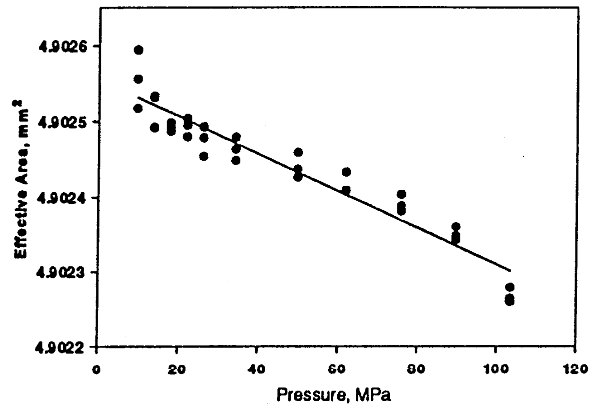

Fig. 3. The effective area of PG 79 as a function of pressure as measured by PG 41. The solid line represents a linear fit to the data.

For completeness, a cross-float between the two high-pressure gas gauges (PG 87 and PG 79) was performed over the range of 9.7 MPa to 103.5 MPa. Both piston gauges were operated with helium. For this sequence of measurements, a conventional cross-float technique[1] was used. The two gauges operated very well and the uncertainty due to random effects of the cross-float process was 5 ppm. The measured effective area of PG 79 as determined with PG 41 and with PG 87 are compared in Fig. 4. Although some systematic effects are seen in the structure of the data, the magnitude of the structure, 6 ppm maximum, is less than the estimated uncertainty of the comparison of 9 ppm. The estimated uncertainty of the comparison between PG 79 and PG 87 is small because the uncertainties due to systematic effects introduced by the common calibrating gauge PG 41 is eliminated.

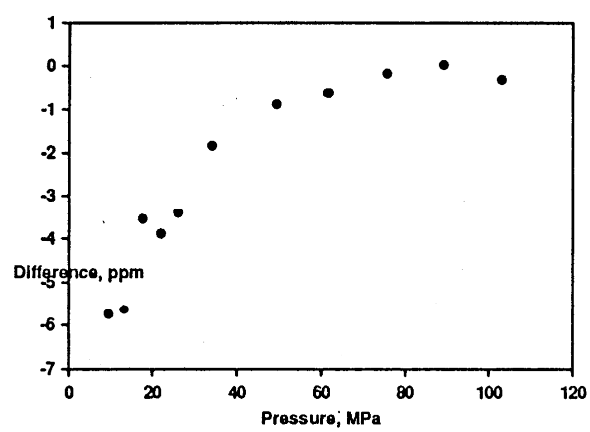

Fig. 4. Difference in the measured effective area of PG 79 as measured by PG 41 from that measured by PG 87.

Uncertainties

To determine the overall uncertainty*** of the high-pressure gas piston gauges, we examined three contributing factors: the uncertainty of the oil standard, the uncertainty of the DP cell, and the uncertainty of the cross-float process. The uncertainty in our oil standard, PG 41, is 37 ppm, which is the largest contributing factor. The uncertainty associated with the DP cell as claimed by the manufacturer is 5 ppm and can be found in the above mentioned user's manual. Last we have the uncertainty associated with the cross-float process itself, which as indicated above ranged from as low as 3 ppm to as high as 5 ppm. The total uncertainty of the high-pressure gas piston gauges is determined using the ISO preferred technique[2] of summing the individual uncertainties in quadrature. The estimated uncertainties are shown in Table 1.

Table 1. Two standard deviation uncertainties for PG 79 and PG 87 high-pressure gas piston gauges.

Uncertainties	PG 79	PG 87
Standard, PG 41	37	37
Differential Pressure Cell	5	5
Random effects	3	5
Total (RSS)	38*	38*

*Uncertainties are rounded up

Summary

The purpose of this project was to develop high-pressure gas calibration capability at NIST with the lowest level of uncertanty over the range 9 MPa to 110 MPa. Two commercially available high-pressure gas piston gauges were chosen to be used as transfer standards for this purpose. These piston gauges, PG 79 and PG 87, were characterized (calibrated) using a NIST oil piston gauge in conjunction with a differential pressure cell. Both high-pressure gas gauges operated well and were determined to be stable transfer standards for high-pressure gas measurements. The two standard deviation uncertainty for the two high-pressure gas piston gauges was determined to be 38 ppm over the stated pressure range.

***All uncertainties in this report represent two standard deviations unless stated otherwise.

Initiation of Calibration Service

The initiation of a calibration service for high-pressure gas is expected in fiscal year 1996. This service will cover a range of 9 MPa to 103 MPa, with expected two standard deviation uncertainties of approximately 40 ppm. This calibration service will be limited to helium gas only due to operational problems using nitrogen at high pressures. It is possible that the calibration service could be extended to include other inert gases in the future.

Acknowledgements

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References

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[2]Taylor, B.N., and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Technical Note 1297 (1994).