DEVELOPMENT OF A
LOW DIFFERENTIAL-PRESSURE STANDARD

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Abstract

The National Institute of Standards and Technology has developed a new differential-pressure calibration system covering a range of 13 kPa (50 inches of water) for operation with line pressures up to 200 kPa. The system includes a primary-standard mercury manometer, pressure control system, and test-instrument manifold. The primary standard is characterized by an expanded uncertainty (k=2) due to systematic effects of 10 mPa (40 microinches of water) + 1.1×10^{-5} of the pressure. Performance of the entire system is limited in part by pressure instabilities, which vary from standard deviations of 3 mPa at the lowest differential pressures, to 60 mPa at full-range. The primary standard and the pressure control system are described, along with the results of performance testing.

Introduction

Differential-pressure measurements are often a limiting factor in flow metrology. Large numbers of pressure transducers are used for this purpose, and there is a correspondingly large demand for calibrations. As the performance of pressure transducers has improved there has been an increased demand for lower-differential-pressure and higher-accuracy calibrations. There are now a significant number of commercial transducers available with full-scale ranges of 1 kPa or less, and the best of these will repeat to within 0.01%. Most of them are used with nominal line or reference pressures of 100 kPa. Many different types of calibration standards are in use, but generally the types that have well-understood metrological characteristics lack the sensitivity for the lowest-range measurements. At the same time, there is an increased uncertainty about the absolute accuracy of some of the more-sensitive standards.

The National Institute of Standards and Technology (NIST) has long used deadweight piston gages for differential-pressure calibrations. These are in many ways ideal for this application, but their operation becomes increasing difficult below about 5 kPa. NIST has also used a group of mercury Ultrasonic Interferometer Manometers (UIMs) as primary pressure standards. These standards have full-scale ranges between 13 kPa and 360 kPa (1 Pa = 0.0075 torr, or 0.00401 inches of water at 4 °C), they can be used for either absolute or differential pressure calibrations with respect to line or reference pressures between 0 and 200 kPa, and they achieve both high accuracy and high sensitivity. However, the associated calibration equipment- pressure manifolds and control systems- were designed primarily for absolute-mode measurements and have proven difficult to use for low-range differential measurements. The recent development of a new UIM that uses a low-vapor-pressure oil as the manometric fluid, and its associated calibration system, has not only extended the lower limit of our absolute pressure manometry by a decade, it has also allowed us to dedicate the 13 kPa mercury UIM to differential-pressure calibrations. The major challenge in making best use of the 1 mPa resolution of this UIM has been developing a system to adequately control the differential pressures. This paper briefly describes the UIM, but describes in more detail the development of the pressure-control system and the overall system performance.

Ultrasonic Interferometer Manometers

Different elements of the UIM's design and performance have been previously described[1-3]. The unique feature of the UIMs is that the change in height of the manometric-fluid surfaces (column heights) is determined by an ultrasonic technique. A transducer at the bottom of each liquid column generates a pulse of ultrasound (typically near 10 MHz) that propagates up the column, is reflected from the liquid-gas interface, and returns to be detected by the transducer. The change in phase of the returned signal is proportional to the length of the column (allowing for temperature and pressure corrections), and, with careful phase measurement, length changes of 10^{-5} mm can be detected. The manometers are otherwise conventional, although care has been taken to minimize errors[1]; they employ a three-tube design to correct for possible tilt, large-diameter (75 mm) liquid surfaces to minimize capillary effects, thermal shields to stabilize the temperature and minimize its gradients, and high-vacuum techniques to minimize leaks and pressure gradients.

There are relatively minor differences in the performance and uncertainties of the different NIST mercury UIMs. At low pressures, standard deviations of the measured pressures vary from about 1 mPa to 3 mPa, depending on the range of the UIM. However, for pressures above about 1 Pa the low-pressure uncertainty is limited by residual systematic nonlinearities in the phase-measurement circuitry. These nonlinearities are difficult to evaluate, but for the 13 kPa UIM used for differential measurements a conservative estimate is that they contribute no more than 10 mPa to the expanded uncertainty (coverage factor, k = 2). The limiting factor at higher pressures, the relative uncertainty due to systematic effects, is principally determined by the uncertainty of the speed of sound (and hence the ultrasonic wavelength), nonlinear propagation characteristics of the ultrasound, and temperature corrections. The 13 kPa UIM uses only two platinum resistance thermometers, compared to the 6 to 10 used in the higher-range UIMs, and their resistance is determined with a digital multimeter rather than an ac resistance bridge. The density of the mercury used to fill this UIM has not been directly measured, but it is estimated from that of other samples purified at NIST. Taking these differences into account, the total expanded uncertainty (k=2) due to systematic effects of a measured pressure P is 10 mPa + Px1.1x10^{-5}.
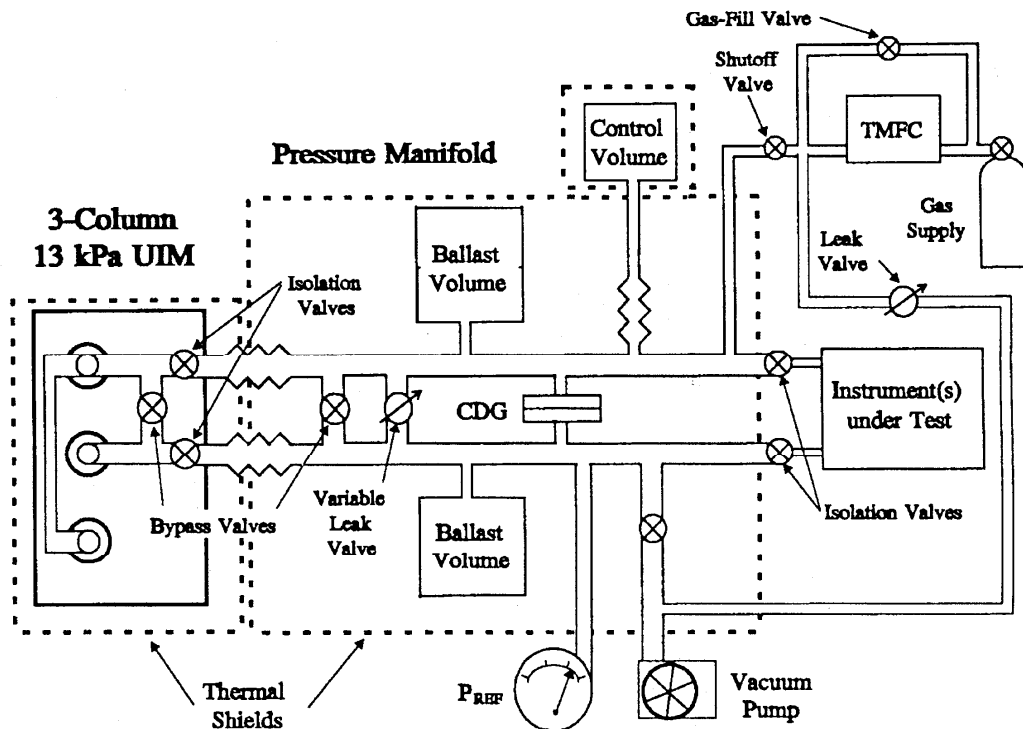
Figure 1; Schematic of the prototype calibration system.

Calibration System

The prototype calibration system, illustrated in Fig. 1, evolved with our different experiments on pressure control. It consists of three main parts; the UIM, a pressure manifold, and the pressure control system, which incorporates a variety of valves and auxiliary volumes.

The UIM is enclosed in a separate thermal enclosure- an aluminum shell with a minimum thickness of 5 cm, surrounded by 5 cm of foam insulation. It is connected to the pressure manifold by bellows for thermal and mechanical isolation, and includes a set of valves to isolate it from the rest of the system when not in use, and a bypass valve that can be opened to generate a zero-pressure reference condition.

For purposes of convenience, the pressure manifold is assembled from standard high vacuum components with internal diameters of 15 mm, which is larger than required for impedance considerations. It includes ports and valves to connect test instruments, a bypass valve that can be opened when setting the zero of test instruments, a connection to a vacuum pump so that room air can be pumped out and the system filled with dry gas, and a digital pressure gage used to monitor the line or reference pressure, P_{REF}.

Pressures are generated or set by a gas-fill valve connected to a compressed-gas bottle and a variable leak valve connected to the vacuum pump. A 2 sccm (10^{-6} mol/s) Thermal Mass Flow Controller (TMFC) can be used alone, or in opposition to the leak valve, to generate small changes in the pressure. As the pressure-control system evolved, different ballast or control volumes were connected to the system, along with one (as shown in Fig. 1), and later two, differential Capacitance Diaphragm Gages (CDGs). Their use is described in the next section.

Temperature-generated pressure instabilities are a major problem for low-range differential pressure calibrations. For differential measurements made with respect to a reference or line pressure near 100 kPa, a change in the temperature difference of 30 μK between the reference and "high" pressure sides of the system will cause a differential pressure change of 10 mPa. The effects of such changes will depend on the pneumatic impedance of the pressure plumbing and the response time of measuring instruments.

The effects of plumbing impedances can be minimized by minimizing system volumes, maximizing plumbing diameters, and maintaining symmetry of the impedances and volumes of the two sides of the system. However, the response times of instruments are generally fixed and can represent a significant limitation. In particular, small displacements of the mercury in the UIM generate damped oscillatory height changes with periods somewhat less than 15 s, so the response time of the UIM to small pressure changes is on the order of 1 minute. The response times of the instruments being calibrated are typically much less, so unstable differential pressures generate random errors in the calibration data that can significantly exceed the inherent capabilities of the standard and possibly those of the test instrument. Therefore, to minimize these errors it is necessary to control the pressures.

Pressure Control

We have used both passive and active pressure control. The passive control consists in part of thermally isolating the pressure manifold by enclosing it with foam insulating board, the thermal enclosures indicated in Fig. 1. There are limits to this approach. Not all of the system can be well insulated. In particular, parts of the pressure adjustment system require regular access, and the instruments being calibrated often include significant volumes (as much as a liter) that cannot be insulated, or they may incorporate on-off temperature control that causes periodic pressure perturbations. In addition, the thermal time constants of a practical temperature isolation system are short compared to the 10 to 20 minute time constants of our laboratory temperature, so they achieve only a limited attenuation of the laboratory ambient temperature changes, which are of the order of ±1 K.

The pressure changes caused by the temperature variations of the pressure manifold and test instruments can be attenuated by other system volumes with more stable temperatures. The temperature of the UIM is both uniform and stable- temperature changes are typically a few mK/hour. However, the volume of gas enclosed within the UIM is only 2 L, so it provides rather limited attenuation for the pressure perturbations generated in the 0.5 L pressure manifold and the additional volume of attached test instruments. Additional attenuation is achieved by adding, to each side of the system, ballast volumes that are matched in size, maintained in close thermal

contact, and connected to the pressure system with matched impedances. We have used two sets of volumes, one set of 10 L each, the other of 45 L. The temperature of the smaller set is lagged by enclosing it in foam insulation, while the temperature of the larger set is controlled by an ice bath.

The effectiveness of the ballast volumes is discussed in detail in the next section, but for now it suffices to say that with these volumes alone the pressure instabilities of the system remain significantly larger than the resolution of the UIM. Further improvement requires active pressure control. There are several possibilities for active pressure control. The most attractive would be a differential deadweight piston gage. Unfortunately, this won't work for the same reason deadweight piston gages are difficult to use as low-range standards- their operation deteriorates below about 5 kPa. Pressures can also be controlled by controlling the flow of gas through a conductance. Other research in our laboratory encouraged us to try this using a TMFC to control the flow, the variable-leak valve between the two sides of the pressure manifold, and a controlled conductance to the vacuum pump. We quickly discovered that the flow instabilities are much too large for this application.

The eventual, and very satisfactory solution, is to use a temperature-controlled volume (the Control Volume of Fig. 1) attached to one side of the pressure manifold. The control volume is 50 cm^3, consisting of an array of 1-cm diameter holes drilled into a copper block. The volume is pneumatically connected to, but thermally isolated from, the pressure manifold by a thin-wall stainless steel bellows. The temperature of the volume is controlled using a thermoelectric module, a bi-directional power supply, and an electronic controller employing both proportional and integral (reset) control. Depending on the range of the differential pressure to be controlled, the controlling signal is generated by the difference between a stable adjustable voltage source and the output of either a 1 torr or a 100 torr differential CDG connected between the two sides of the pressure manifold. For low differential pressures, using an integral time constant of 200 s and proportional gains varying from 0.1 V/Pa to 100 V/Pa, we have found it possible to achieve both short- and long-term pressure stability within a few minutes of generating a new differential pressure. Including the perturbations of test instruments with on-off temperature control, the instabilities are comparable to the limiting performance of the UIM. At full-scale the stability deteriorates by about a factor of 20.

System Performance

If pneumatic time constants are short, the overall system performance will be primarily limited by pressure instabilities over times comparable to the response times of the standard and the test instruments. Typically, this will be the 1 minute response time of the UIM. Figure 2, with data taken at 1 minute intervals, illustrates the time dependence of UIM measurements for different configurations of the system. All data were obtained with a nominal 100 kPa line pressure and zero differential pressure. Section A of the figure illustrates the random variations of the UIM readings when the UIM is isolated from the rest of the system and with an open bypass valve maintaining zero differential pressure between the two sides of the system. The standard deviation of these data, 1.1 mPa, is indicative of the smallest pressure change than can be detected and sets a limit on the performance of the UIM standard and the entire system.

Following the data of section A, the bypass valve was closed so that the "zero" differential pressure was maintained only by the various control techniques used throughout this experiment. Section B illustrates the best performance of the system using only ballast volumes for pressure control. In this case the system includes both the 10 L and 45 L volumes, and four test or control instruments with on-off temperature control and a total volume on the order of 1 L. The standard deviation of these data, 98 mPa, is significantly larger than that obtained with the UIM alone. These data exhibit a periodicity typical of the laboratory's ambient temperature control. When only the

Figure 2; Deviations of the differential-pressure as measured by the UIM at nominal zero differential and 100 kPa line pressure, using different pressure control techniques as explained in the text. The data of section B are scaled to the right.

10 L volumes are used the pressure variations are strongly cyclical with an amplitude about 2.5 larger than when both sets of volumes are used, and, depending on the stability of the laboratory temperature, can exhibit longer-term changes that approach 100 mPa/minute. These variations in pressure cause random differences between the test instruments and the UIM that can be as large as 200 mPa.
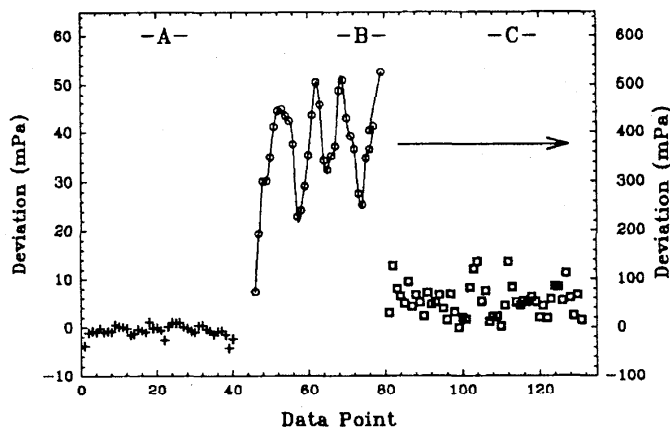
Data obtained with both ballast volumes in the system and active pressure control, using the 1 torr CDG, are shown in section C. The standard deviation of these data, 3.3 mPa, is larger than that due to the UIM alone, but less than the limiting nonlinearity of the UIM. If the active pressure control is used with only the 10 L ballast volume the standard deviation is a factor of 5 larger, but there is no detectable long-term trend. The data of section C were taken 9 hours after the data of Section A. In the intervening period a series of experiments were performed on the system, but the bypass valves remained closed and none of the instruments were reset or rezeroed. Thus, the 5.5 mPa offset between the means of the data of sections C and A is indicative of the long-term instability of the system, and is about a factor of 2 larger than the long-term (days) zero instability of the UIM. The difference is most likely due to zero drift of the CDG used to control the pressure.

The data of Fig. 2 were taken after an over-night equilibration and with deliberate perturbations of the system during the experiment that did not exceed a few Pa. Under these conditions, within a few minutes of a perturbation the control system returns the system to the original set point, and to the same magnitude of random variations seen in section C. However, during a typical calibration cycle, as the generated differential pressure increases the mechanical and thermal perturbations of the system also increase because of the larger amounts of gas that must be introduced to generate the pressure. Further, there are practical limits to the equilibration time following the establishment of a new pressure. Thus the pressure instabilities increase with

increasing pressure. Up to 150 Pa, where the 1 torr CDG can still be used for control, the instabilities are no greater than a factor of 2 larger than exhibited in section C, with an equilibration time following the pressure change of less than 5 minutes. At higher pressures, where the less-stable 100 torr CDG must be used for control, standard deviations vary from 15 mPa at 200 Pa to 60 mPa at 10 kPa. The time to establish this level of control increases to 10 to 15 minutes at the highest pressures. To put this in perspective, this largest instability is less than 10^{-5} of the full-scale pressure.

While pressure control is a necessary condition, the ultimate test of the system is whether or not it can satisfactorily calibrate a test instrument. Since test-instrument calibration data are limited in part by the performance of the test instrument, it is desirable to perform this test with an instrument with high resolution and proven stability. Figure 3 presents the residuals from a quadratic fit to the differential-pressure calibration data of a 7.5 kPa quartz Bourdon-tube gage. This gage has a 10 V full-scale output that is measured with a 7-digit multimeter with a calibration uncertainty that is periodically verified to be within 10^{-5} of the measured voltage. This gage was earlier adapted for absolute-mode measurements by providing a reference vacuum at the low-pressure port, and has been calibrated several times over the past year with our absolute-pressure calibration systems. These results, discussed in another paper in these proceedings[4], have established that the gage has very good short- and long-term stability. Since absolute-pressures are relatively easy to control, these results are not corrupted by pressure instabilities and they represent the ultimate capabilities of the gage.

The data in Fig. 3 are effectively the differences between the pressure indicated by the calibrated gage and the UIM measurements. The standard deviations of these differences range from about 15 mPa at the lower pressures to 40 mPa at the higher pressures, which is about 5×10^{-6} of the measured pressure. The standard deviations of the UIM readings alone can be separately evaluated. They are about 60 mPa at the highest pressures and are indicative of the pressure instability, as previously noted. The reduction in the standard deviation of the differences, compared to the standard deviation of the measured pressure, is indicative of the degree to which both instruments can track the changing pressure. The residuals of Fig. 3 are also about 50% larger than the residuals of absolute-mode calibration data, further indicating that the effect of pressure instabilities on the differential-mode calibration are relatively minor.
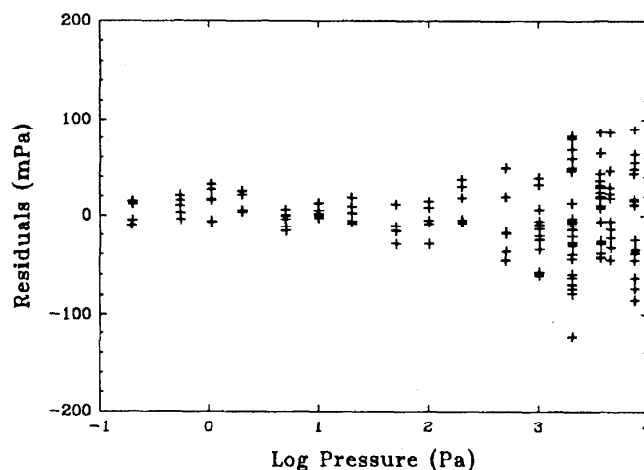

Figure 3; Residuals to the fit of calibration data for a 7.5 kPa fused-quartz Bourdon-tube gage. The scatter of the data are indicative of the combined performance of the calibration system and the gage.

It should be noted that Fig. 3 does not include data taken with deliberately altered control system parameters. In particular, increasing the gain of the pressure-control system beyond a certain point results in more stable UIM readings, but increased scatter in residuals of the calibration data. This indicates that the control system has improved the stability of the UIM readings by "fast" pressure adjustments which the UIM averages out, but the test instrument does not. Fortunately this condition is indicated by visibly increased "hunting" of the control system.

In general, we consider these calibration results to be satisfactory. If necessary, the effects of random errors, including those caused by pressure instabilities, can be reduced by averaging or fitting repeat data. For instance, the fit to the calibration data illustrated in Fig. 3 will have an uncertainty close to the limiting uncertainty of the UIM. However, this is meaningless if it is less than the instability of the instrument. For this gage, the zero drift is about 10 mPa/hour, which is a small fraction (10^{-6}) of its full-range, but will be a limiting factor for low-pressure measurements. Similarly, the higher-pressure performance of this instrument will be limited by its calibration instability, which at 0.01%[4] over the upper 90% of full-scale is small for a mechanical transducer, but much larger than either the uncertainty of the fit or the uncertainty of the UIM (it is of interest to note that the results of this differential-pressure calibration agree with the absolute-pressure calibrations to within this same tolerance). In short, the overall performance of the calibration system does not limit the uncertainty of a gage of this type.

Planned Improvements

We believe that the performance of the prototype calibration system is adequate for most or all of the calibration needs of which we are presently aware (if there are more demanding requirements we would like to hear of them). However, the system can definitely be simplified, and the ease-of-use, and possibly the performance, can be improved. The effects of room-temperature instabilities are evident in all of the data. Therefore, we plan to actively control the temperature of all parts of the system. With active control of the reference pressure, as well as the differential pressure, we hope to minimize the ballast volumes. We expect that a more thorough evaluation of the limiting nonlinearity of the UIM will probably result in a reduction of its low-pressure uncertainty. If the need exists, and the pressure control is adequate, the resolution and low-pressure uncertainty can be further improved by an order of magnitude with a UIM that uses a low-density manometric fluid (oil or water).

Acknowledgements

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