NEW DEVELOPMENTS IN BAROMETRIC RANGE PRESSURE STANDARDS

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ABSTRACT

The largest number of pressure measurements and the most exacting accuracy requirements exist in the atmospheric or barometric pressure range, which extends from a few pascal to a few tenths of a megapascal. Standards in this range are maintained using mercury manometer primary standards or gas-operated piston gage transfer standards. In an effort to improve NBS pressure standards, and allow industrial laboratories to go beyond the current 25-30 ppm state-of-the-art, NBS has developed a new type of mercury manometer and is exploring the performance limitations of gas-operated piston gages. The ultrasonic interferometer manometer is a highly automated mercury manometer that uses an ultrasonic phase shift measurement technique to achieve a reproducibility of 10^{-2} Pa at low pressures and an uncertainty of 15 ppm at higher pressures. The design and performance of this instrument will be described, along with the results of experiments in which the manometer has been used to explore the dependence of piston gages on various operating parameters, including gas composition, and gage or differential vs. absolute mode operation.

INTRODUCTION

The beginning of pressure measurements can be properly credited to Evangelisti Torricellis's invention of the mercury barometer three and a half centuries ago. Since that time the field has progressed significantly; the National Bureau of Standards now maintains pressure standards and calibration services over most of a fifteen-decade range from 10^{-6} to 10^{+9} pascal (10^{-8} Torr to 200,000 psi) and a constant effort is underway to develop primary standards and services within this range and beyond to meet the increasing accuracy needs of U.S. industry and science. This program has included, within recent years, efforts to upgrade our standards and provide needed information to users in the Barometric Range where Torricelli worked. Within this range, somewhat broadly defined as 1 to 300,000 Pa, are found a very diverse group of users, some with very difficult pressure measurement problems and/or stringent accuracy requirements. In the lower part of this range the operating efficiency, product yield, and product quality obtained from semiconductor fabrication processes, freeze drying of pharmacueticals and food products, and vacuum processing of super alloys can depend critically on the control of process pressures. In the middle and upper part of this range both civilian and military aircraft rely on the pressure altimeter as a primary flight instrument and maintenance of required accuracies requires laboratory pressure standards with accuracies as high as 10 parts per million (ppm). Throughout this range thermodynamic and cross section data are required for numerous materials and the quality of these data are directly linked to the accuracy of the pressure or density measurements. Flow measurements for process control or commodity transfer require accurate differential pressure measurements, often at very low values.

The Barometric Range Standards program has included the development of improved primary standards, characterization of transfer standards, and provision of new or improved calibration or special test services. The initial emphasis was on primary standards, where two choices exist; mercury manometers or gas-operated deadweight piston gages. As one part of this program we chose to develop a new type of mercury manometer, the ultrasonic interferometer manometer (UIM), described below. This decision was made for several reasons; mercury manometers are capable of better accuracies than piston gages, they can be used at lower pressures where there was a notable lack of standards, and their operation is more flexible, readily allowing absolute or differential measurements for both static or slowly changing pressures. However, piston gages have a number of complementary advantages, particularly as transfer standards. They are simpler (and less expensive), less susceptible to environmental perturbations and more forgiving of operator errors. They are widely used in industry and as reference or working standards in a parallel NBS program that is also developing improved standards for atmospheric and higher pressures. Since there is only very limited information on piston gage performance at the 10 ppm or better level, we have used the UIM's to determine piston gage characteristics for different operating conditions, as described below. This should permit a better understanding of the fundamentals of piston gage operation and improve their performance as transfer standards. The UIM's have also been used to obtain performance data on low-range capacitance diaphragm gages (Ref. 1) and quartz spiral gages.

The initial impetus to develop the UIM's came from requests for calibrations in the low pressure part of the Barometric Range and the lack of a serviceable NBS standard for those pressures. Thus, the UIM's were initially developed as low range (10 kPa, 100 Torr) standards, where they achieved an imprecision (one standard deviation) of 2-3 mPa ($3x10^{-5}$ Torr). This allowed the initiation of a new calibration service over the range of the first UIM's. Subsequent developments have allowed an extension of the UIM range to 160 kPa (1200 Torr) with a three-sigma systematic uncertainty of 15 ppm. The calibration service for electromechanical transducers and ball gages has been correspondingly extended and improved and a special test service for piston gages is offered over this range and with those uncertainties. A model currently under development further extends the UIM range to 360 kPa (110" Hg).

This paper will briefly describe the design and performance of the UIM's and the results obtained on the performance of gas-operated piston gages. A more detailed analysis of the UIM and its use in the NBS calibration service will be presented in Ref. 2.

ULTRASONIC INTERFEROMETER MANOMETER

Liquid column manometers determine a differential pressure, P, by measuring the vertical displacement, h, of a liquid column of known density, ρ, in a gravitational field with acceleration g:

$$P = \rho g h \qquad (1)$$

If the reference pressure is reduced to "zero" by evacuating the space above the liquid in the reference column, an absolute pressure is determined. For high accuracy manometry, mercury is the preferred liquid. Its reference density is well known, its thermal expansion and vapor pressure are relatively small, and its density is high enough to allow measurements in the atmospheric pressure range with reasonable column lengths. The accuracies of high performance manometers are generally limited by the measurement of the heights of the liquid column and the temperature of the liquid. Accurate low pressure measurements require high resolution length measurements and stable temperature gradients. Measurements at higher pressures with a low percentage uncertainty require a corresponding accuracy in the height measurements and an accurate determination of the average temperature of the mercury. Although the major difference between manometer designs is, often as not, the technique used to measure the heights, the thermal design is equally important. Therefore, in initially designing a low range, high resolution manometer suitable for efficient everyday use, we selected an ultrasonic technique for the height measurement because it achieves a high length resolution ($\sim 10^{-5}$ mm), is relatively immune to disturbances on the mercury surface, and minimizes temperature perturbations. No optical, mechanical, or human access to the manometer is required, the measurement process generates microwatts of power, and the manometer can be thermally isolated to ensure stable and uniform temperatures. These advantages are somewhat offset by an effective tripling of the temperature coefficient of an ordinary mercury manometer (to 484 ppm K^{-1}), but the results discussed below demonstrate a net improvement both in performance and ease of use.

Ultrasonic Measurement Technique

The technique employed in the UIM involves the measurement of the phase change of a pseudo-continuous ultrasound signal, avoiding the rise-time problems associated with the measurement of the time of flight of ultrasound pulses. A 15 μs-long packet of nominal 10 MHz ultrasound is generated by a transducer at the bottom of the mercury as shown in Fig. 1. The ultrasound travels up through the mercury, is reflected from the mercury-gas interface and returns to the transducer, where it generates an electrical signal. The phase of this return signal or echo is measured, relative to the continuous wave used to generate the original ultrasound signal, near the center of the wave packet where it is unperturbed by risetime effects. Length changes of the mercury column cause corresponding changes in the phase of the ultrasound echo. Lengths of columns under 100 mm are typically measured with a standard deviation of 10^{-5} mm.

Since the ultrasound wavelength is about 150 μm, large phase changes must be measured for manometry applications. This was originally done by up-down "fringe" counting as the phase changed through multiplies of $\pi/2$, but this is susceptible to error if the data are interrupted. In the newer systems a relatively crude length measurement is first made using a

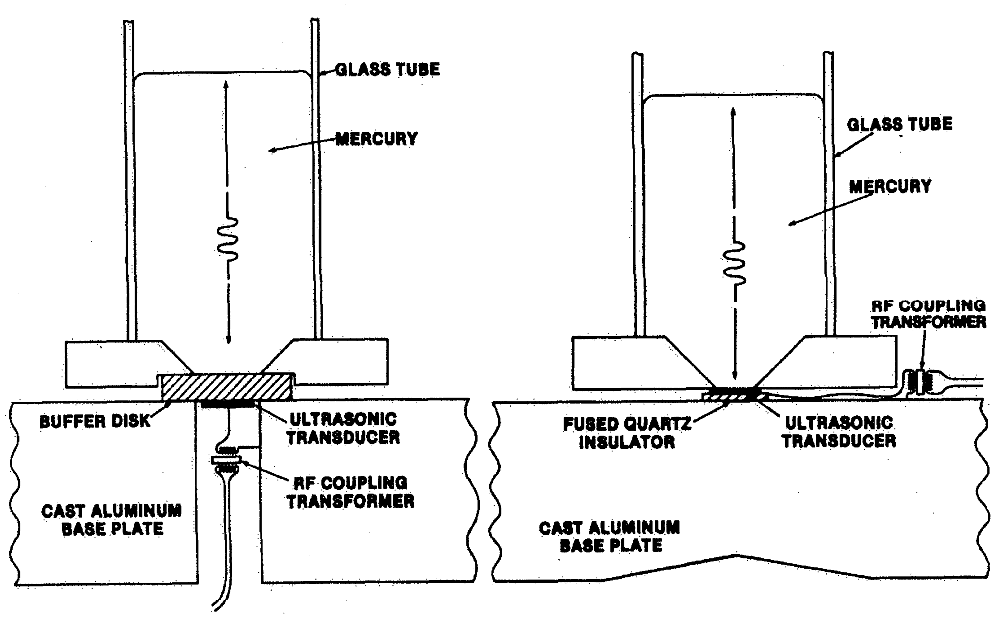

Figure 1. In different versions of the UIM ultrasound is transmitted to and from the mercury in two different ways. On the left an optically polished beryllium disk is used as a buffer material between the transducer and the mercury, on the right a single crystal quartz transducer is placed in direct contact with the mercury. In this case the ultrasound also transmits into the aluminum base, where it must be dispersed if spurious return echoes are to be avoided.

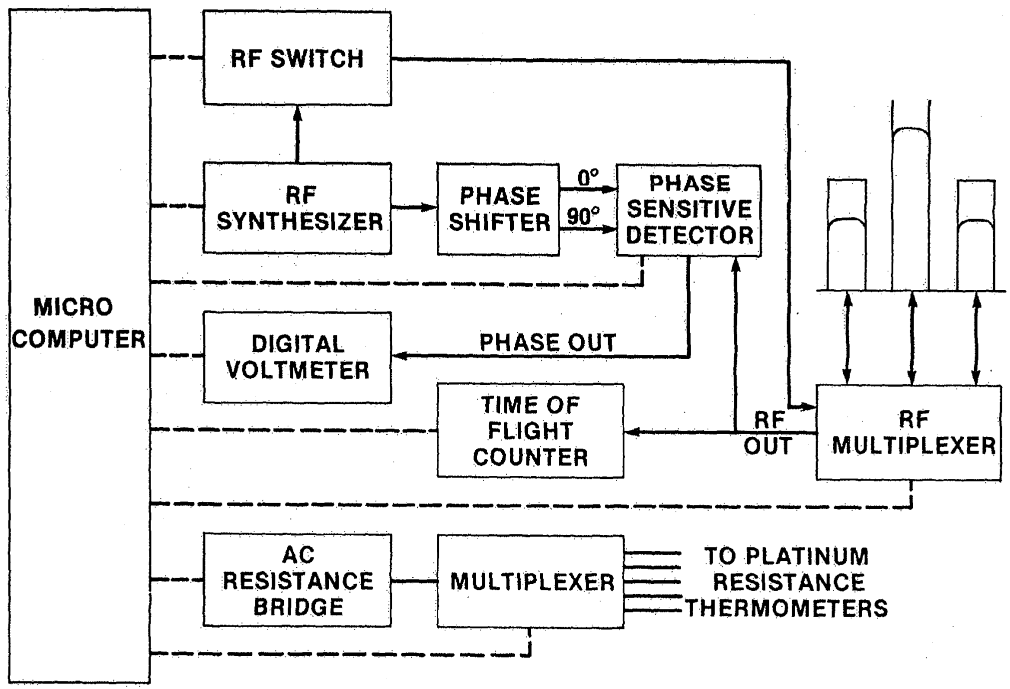

Figure 2. Schematic of the UIM rf electronics, data acquisition, and control system. Solid lines indicate analog signals, dashed lines are IEEE 488 buses.

time-of-flight method. Phase measurements are then made at four unevenly spaced frequencies between 9.5 and 10.5 MHz. The phase measurements are combined with the time of flight measurement using an exact fractions algorithm (Ref. 3). This permits the measurement of lengths, with the full precision of the phase technique, but without fringe counting, over the full 2.8 m range of the highest pressure manometer. The different frequencies are generated by a frequency synthesizer. The synthesizer and phase measurement system are controlled by a computer. In normal operation the computer is programmed to make fourteen measurements of the height of three different columns at four different frequencies over a 2-4 second time interval. This system is shown in schematic form in Fig. 2 and its operation is more fully described in Ref. 4.

Measurements with an imprecision of 10^{-5} mm require an imprecision in the phase measurement of better than 1 mrad (10^{-4} of a circle). Imperfections in the double balanced mixers used to measure the phase, or "interfere" the return echo with the carrier, cause systematic errors an order of magnitude larger than this. These can be corrected by statistically analyzing phase data taken over a range of lengths (Ref. 5). These corrections to the phase data are made by the computer before the lengths are calculated with the exact fractions algorithm.

Mechanical Design

The basic mechanical design of the 13 kPa manometer and the operation of an early version of the electronics are described in Ref. 6. For all versions the manometer is of the "W" or three-tube type. The pressure is applied to two tubes equally spaced about a center reference column. Measurements in the two outer tubes are used to detect and correct for any tilt of the manometer structure. The tubes are 75 mm-diameter borosilicate glass, coated on the inside with an evaporated nickel-chromium conducting layer that eliminates static charges. In the 13 kPa-range UIM, described in Ref. 6, all three tubes are 120 mm long. The later, longer range instruments, illustrated in Fig. 3, differ from that described in Ref. 6 in that the length of the center or reference tube is increased to correspond to the range of the manometer. Mercury is supplied to this center column from stainless steel reservoirs as the pressure is increased. In this way the heights of all three columns are less than 90 mm at zero pressure, even for the 360 kPa UIM. This permits the maintenance of the 10^{-5} mm height imprecision, and corresponding pressure imprecision, for low pressure measurements, while allowing optimum accuracy over a large range of pressures. The 13 kPa UIM is no longer maintained in service, since low range calibrations can be performed quite satisfactorily with one of the longer-range instruments.

The manometer structure is mounted on a heavy aluminum base and totally surrounded by

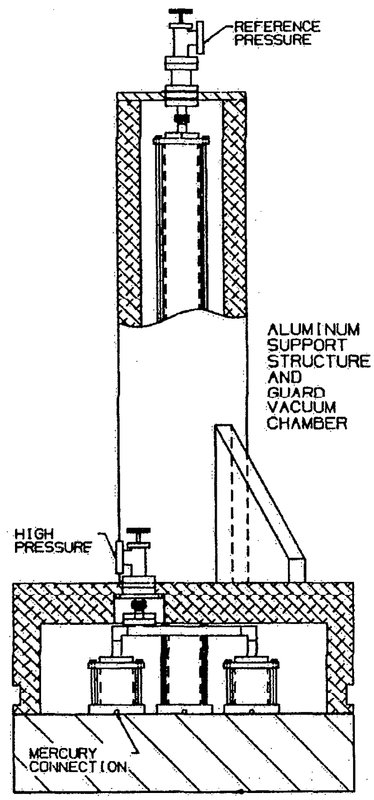

Figure 3. Schematic of the longer range UIM's. The three columns are connected at the bottom by stainless steel plumbing, not shown, attached to the stainless flanges at the bottom of the glass tubes. Mercury is supplied to the center column from stainless steel reservoirs, also not shown. Except at the ends of the glass tubes, all gas-pressure plumbing is welded or uses metal-gasketed ultra-high vacuum fittings. A guard vacuum below 10 Pa is maintained inside the surrounding aluminum structure.

a heavy-wall aluminum vacuum chamber. A guard vacuum is maintained around the manometer to prevent leakage of air through teflon seals at the ends of the glass tubes and in parts of the stainless steel mercury plumbing. The parts in contact with the mercury are glass, stainless steel, teflon, and beryllium or quartz. The aluminum structure is also used to stiffen the center glass tube and promote uniformity and stability of temperature. The entire manometer is surrounded by 5 cm of expanded thermal foam. Depending on the range, three to nine platinum resistance thermometers (PRT's) are used to sense the temperature. A weighted mean of the PRT temperatures, depending on the height of the mercury in the center column, is used to calculate the density of the mercury.

Operation of the ultrasonic measuring circuitry, measurement and calculation of the temperature, and calculation of pressures, including gas hydrostatic corrections, are all computer-controlled. For each measurement this requires 30 to 50 seconds, including logging of data from instruments under test and recording of the results on paper and on floppy disks. The responses of the manometers are generally limited by their 30-60 second pneumatic time constant. About 5-30 minutes are required to achieve temperature equilibrium after a significant pressure change.

UIM Uncertainty

The performance of the UIM's have been evaluated over several years from the repeatability of zero pressure measurements, the random errors of low pressure transducer calibrations, the random errors of the speed of sound measurements reported in Ref. 4, the comparison of different UIM's over their full ranges, and the random errors and systematic trends with pressure in the determination of piston gage effective areas. These include measurements in both the absolute mode ("zero" reference pressure) and differential mode. The latter are generally made with a reference pressure of 93 kPa to simulate gage mode operation with an atmospheric reference pressure. No significant difference is seen in the performance of the manometers in these two modes or with different gases. Many of these details will be reported in Ref. 2.

Random errors in the measured pressure will include those due to the measurement of the heights of the three columns and temperature perturbations. The standard deviation of measurements with zero applied differential pressure over a 24 hour period is 2-3 mPa. At higher pressures, in both the absolute and gage modes, the comparisons of two UIM's over periods of days have standard deviations that are typically 0.7 - 0.8 ppm, implying a standard deviation for each manometer of 0.5 - 0.6 ppm. Long-term stability has been monitored by the repeated calibration of a gas-operated piston gage over a four year period. The calibrations have changed by less than 1 ppm. Analysis of these measurements gives an estimate of the random error at the three sigma level of the UIM's, for all pressures within their ranges, of 10 mPa + 1.7 ppm.

The largest contribution to the systematic uncertainty is from the determination of the ultrasonic wavelength and propagation characteristics. The speed of sound in a UIM structure for column lengths up to 400 mm was determined by comparing ultrasonically measured lengths with those measured using a frequency-controlled infrared laser (Ref. 4). The three-sigma uncertainty of these measurements was 4.3 ppm, and an additional 0.75 ppm is allowed for variations of the speed with the square root of the reference mercury density, which we have estimated might vary by 1.5 ppm due to isotopic variations. However, the generation, propagation, and detection of the ultrasound in the manometer structures may cause phase shifts in the received signal and attendant errors in the height measurement. These could be due to imperfections in individual ultrasonic transducers, strains in the transducer mounting, and diffraction effects due to the finite size of the manometer tubes and transducer mounting. We estimate that errors due to these effects do not exceed 6 ppm.

A linear sum of the component systematic uncertainties gives a three-sigma systematic uncertainty of 15 ppm.

PISTON GAGES

The essential parts of a piston gage or pressure balance are a piston and cylinder fabricated with a small clearance (micrometers or less) between the piston and cylinder and as nearly perfect cylindrical shapes as possible. A pressure applied to the bottom of the vertical piston and cylinder is balanced by a force on the piston generated by the mass of the piston and attached weights. Thus, when the piston and weights are "floating" or balanced, with no friction between the piston and cylinder, the pressure, P, is given by

$$P = mg/A_{eff} \tag{2}$$

where m is the effective mass of the piston and weights (true mass corrected for gas buoyancy), g the acceleration of gravity, and A_{eff} is the effective area of the piston and cylinder combination. The effective area is the critical property of the gage and is defined by Eq. 2 as the ratio of the gravitational force to the applied pressure. It is usually assumed that at low pressures, where distortion of the piston and cylinder are negligible, that the effective area is an invariant property of the gage, controlled by the dimensional properties of the piston and cylinder. However, other factors will also affect the effective area since the pressurizing gas flowing through the annular gap between the piston and cylinder will exert a force on the piston. Furthermore, since the piston and weight stack are rotated to center the piston within the cylinder and relieve friction there may be non-gravitational forces due to the rotation of the piston.

Piston gages have been reliably used for many years as both primary and transfer standards with uncertainties as low as 20-30 ppm. However, if significantly better accuracies are to be achieved, it is reasonable to re-examine to what extent the effective area is dependent on geometric imperfections of the piston and cylinder, on interactions between the pressure fluid and the piston, and on non-gravitational forces. Some idea of the possible magnitude of these effects can be obtained by determining piston gage effective areas as a function of the height of the piston relative to the cylinder, gas species, and pressure, using an independent standard, e.g., a mercury manometer.

We have obtained such data for two different designs of gas-operated piston gages over a period of five years using a UIM. We have obtained these data for operation in both the absolute and "gage" mode (differential operation with a reference pressure of 93 kPa). One of the piston gage designs has a nominal piston diameter of 1 cm, a full-scale pressure range of 50 psi (340 kPa), and the piston and cylinder are fabricated of 440C, a hardenable stainless steel. The other design has a nominal piston diameter of 2 cm, a full range of 44 psi, and the cylinder is tungsten carbide while the pistons are either tungsten carbide or tool steel. The gages were operated using an NBS-designed piston gage base which includes sensors to measure the temperature, height of the piston relative to the cylinder, and rotation rate of the weights and piston. The measured temperatures were used to correct all effective areas back to a common reference temperature (23 °C). For absolute mode measurements the reference pressure in the piston gage bell jar, typically 0.2 to 0.4 Pa, was maintained by a trapped mercury diffusion pump. For the differential mode measurements the reference pressures were generally maintained at 93 kPa ± 20 Pa, to simulate gage mode measurements where the reference pressure is atmospheric. In all cases the same gas was used for both the reference and high pressure, e.g., if helium was used to pressurize the gage, it was also used for the reference atmosphere.

Piston Height

The pistons and cylinders are not perfect geometric shapes. As the piston moves up and down relative to the cylinder during the operation of the gage, changes in the relative position of irregularities in both pieces can cause significant changes in the effective area. Since fabrication of high quality pistons and cylinders is an art it is to be expected that the results will vary from one gage to another. In order to operate a gage with a reproducible effective area, its variation with piston height or "engagement length" must be determined. This will allow the definition of a reasonable operating range for the gage or the determination of a correction factor that can be used to correct data to a common reference height. Figure 4 shows examples of the change in effective area as a function of the distance between the bottom of the piston and the bottom of the cylinder for two different gages.

The data plotted with a "+" are for a gage operated with helium at 113 kPa in the absolute mode. On the basis of this type of data we defined the operating range for this gage to be in the region of minimum change between piston heights of 1 and 5 mm. The average of data within this range was used in calculating effective areas. This first gage is, in our experience, a particularly, but not uniquely, bad example of the manufacturer's art.

Data for the second gage, plotted with a "0", are for an absolute nitrogen pressure of 116 kPa. For this gage we used a linear fit to the data between 2 and 10 mm to obtain a correction factor used to reduce the effective area to a reference value at 6 mm. As can be imagined, the effective area for this gage can be determined more precisely than it can be for the first gage.

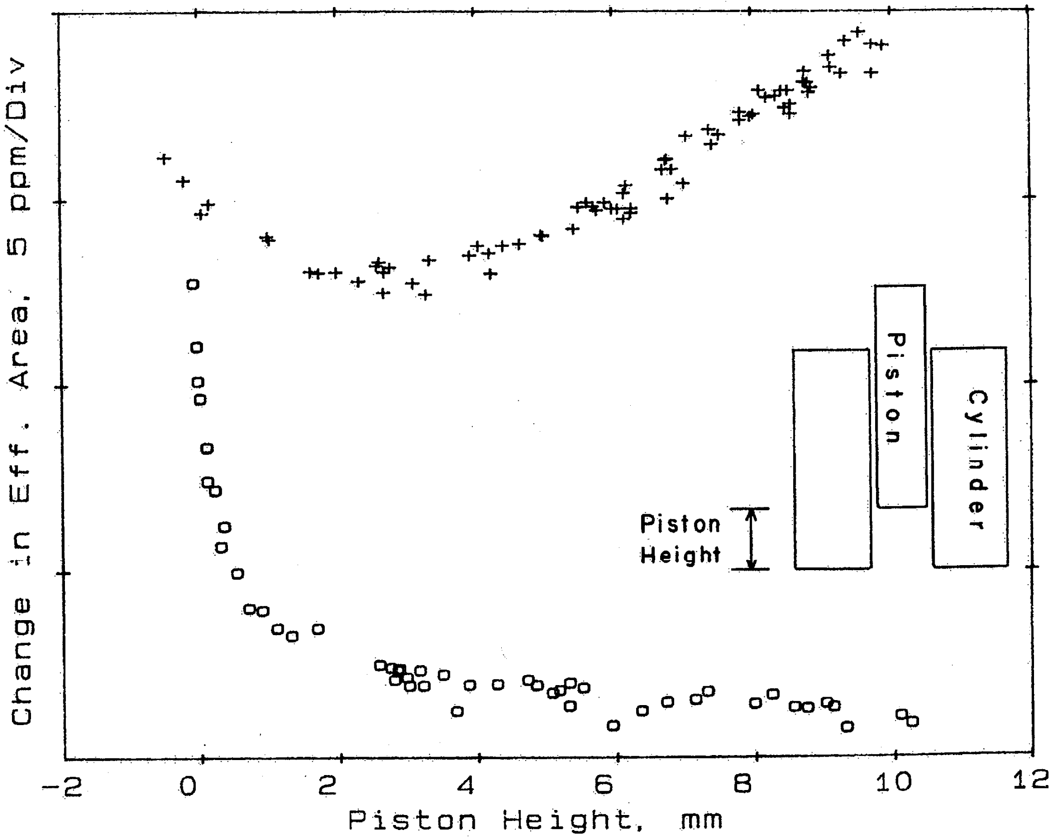

Figure 4. Variation of the effective areas with piston height of two different piston gages. At a given pressure these data are typically obtained over a 30-100 minute period, with 50 seconds required to obtain each data point.

It is clear that if gages are to be used as transfer standards at the ppm level the variations of effective area with height must be known and taken into account. Although these procedures will minimize errors, they will not eliminate them. The best results will be obtained with those gages with the minimum variation of effective area with piston height.

Change of Effective Areas with Pressure

Figure 5 illustrates the pressure dependence of the effective area for nitrogen in the absolute mode for one gage. Similar changes of the effective area at low absolute pressures have been seen for other gages.

An obvious explanation for the data in Fig. 5 is that there is a mass error. The masses were carefully checked and the densities of all masses, including the piston and weight carrier, experimentally determined. We believe the errors in the masses are significantly below the level of the effects seen in Fig. 5. Another possible explanation are magnetic forces

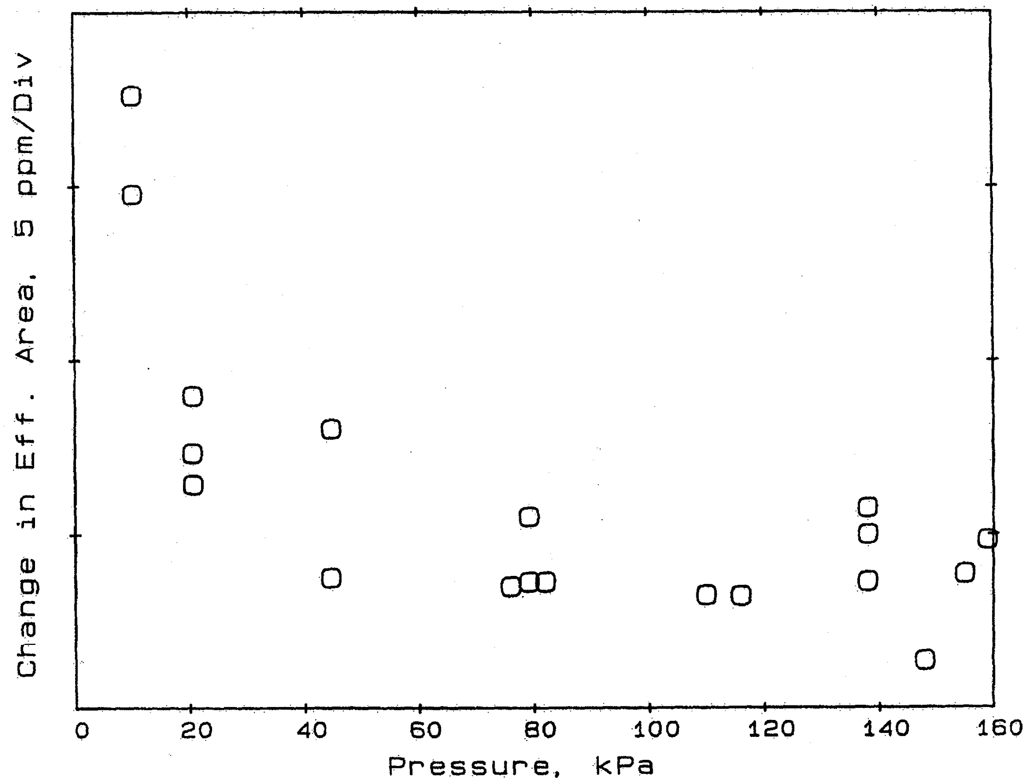

Figure 5. The effective areas for one gage operated with nitrogen in the absolute mode. Each point is the average of 30 to 100 points taken over a corresponding number of minutes. Data at different pressures were taken over a six month's period.

between the piston and cylinder. The piston and cylinder were demagnetized and the magnetic fields checked with a Hall probe. Residual fields of a few Gauss were detected at the corners between the cylindrical surfaces and the ends of the pistons and cylinders. Since the fields are so localized and difficult to map it is hard to calculate the resultant force, but it seems unlikely that the force would be significant except near zero height when the ends of the piston and cylinder are in close proximity. It is possible that the changes in area at low pressure seen in Fig. 5 are caused by changes in the behavior of the gas in the annulus.

Piston and Weight Rotational Rate

For some commercial piston gage bases the piston and weight rotation rate may be as high as 16 Hz. After spinning the piston and weights they are allowed to freely coast, with the rotational rate asymptotically decreasing until at some point the rotation abruptly decreases, presumably because the centering has deteriorated and metal to metal contact occurs. The lower end of the operating range or cutoff rate for different gages varied from below 0.1 Hz to 2 Hz. This is probably determined by the uniformity of the clearance between the piston and the cylinder, the verticality of the piston rotation axis, and the concentricity of the weights with the rotation axis (Ref. 7).

There is clear evidence for a significant aerodynamic force on the weights, dependent on the rate of rotation, when operated in the gage mode (Ref. 8, 9). The magnitude of the effects observed by Prowse and Sutton was such that this effect must be understood and taken into account if reliable differential or gage mode effective areas are to be obtained.

We have investigated this rotation rate-dependent effect for several gases and different types of weights. Preliminary results (Ref. 10) were in qualitative agreement with Prowse and Sutton's observations, but further indicated that the magnitude and sign of the effect, for a given rotation rate, depends on the outside diameter of the weights. The magnitude, at a given rotation rate, also depends on the gas, being smallest for helium, significantly larger for nitrogen, and larger still for argon, and varies linearly with the reference or line pressure.

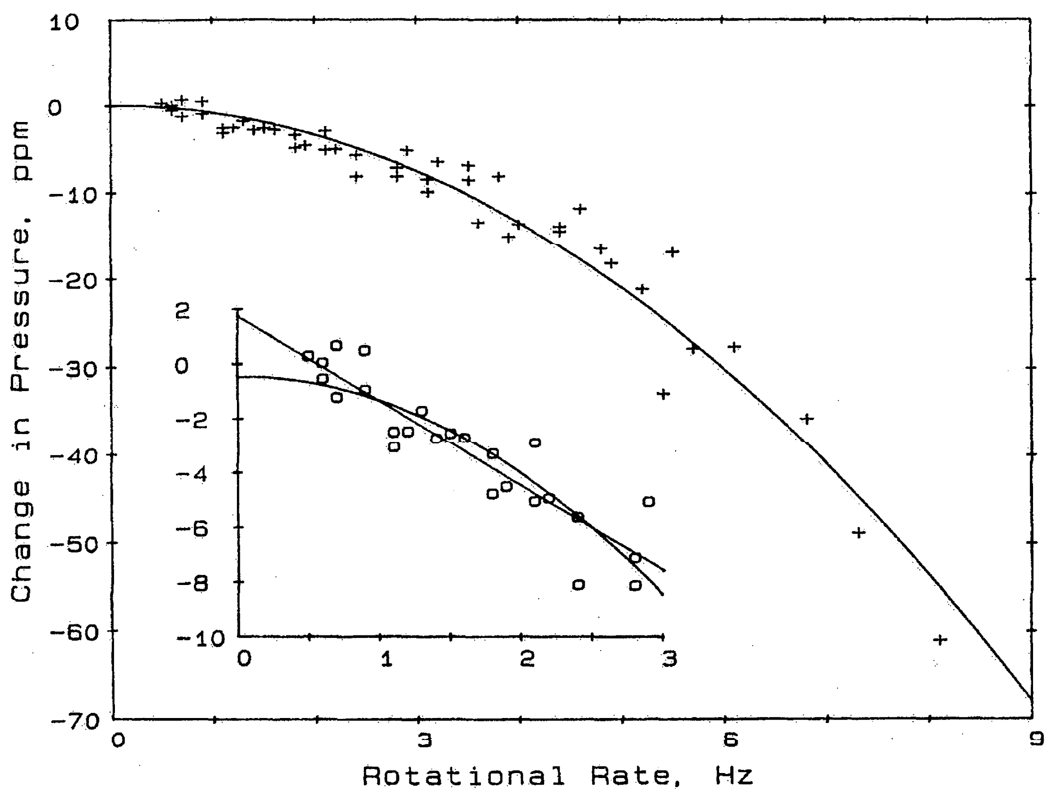

Figure 6. Variation of the nitrogen differential pressure generated by a piston gage in a nitrogen reference atmosphere. The linear and quadratic curves in the inset are fits to the data below 3 Hz, but are referenced to the intercept of the curve fit to the full range of data. The shifts of the intercepts of these two curves are indicative of the possible errors in extrapolating measurements to zero frequency.

Subsequent work confirmed Sutton's observation that the magnitude of this effect varies quadratically with the rotation rate, at least for higher rotation rates. This is illustrated in Fig. 6 where the change in the pressure generated by the gage is plotted as a function of the rotation rate, along with a quadratic curve derived from a least squares fit to the data. The inset shows the lower frequency portion of this same data where the effect becomes almost linear, but still significant. These data were obtained with weights with a diameter of 116 mm operating in a nitrogen reference gas at 93 kPa. The differential pressure was 121 kPa. In determining effective areas in the gage or differential mode we use the zero-frequency intercept of the fitted quadratic equations. For our bell jar, if the outside diameter of the weights is reduced to about 80 mm, the magnitude of the weight rotation effect decreases to zero. With a further reduction in weight diameter the sign of the effect reverses. The magnitude of the effect also scales with the molecular weight of the reference gas, being quite small for helium.

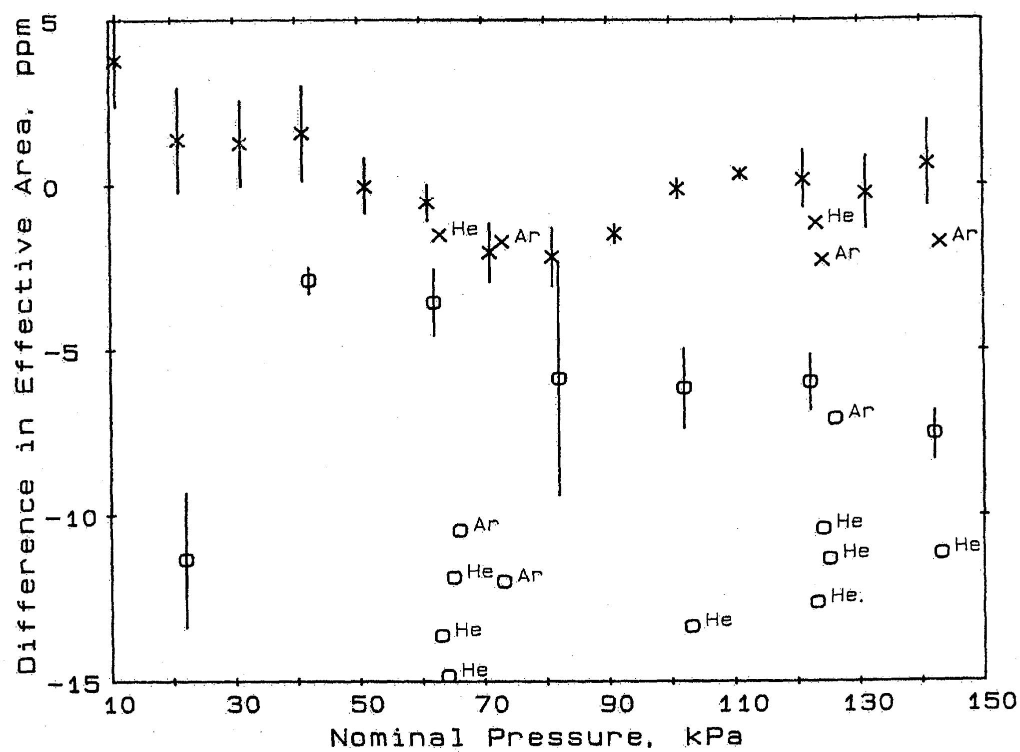

Figure 7. The effective area of a 1 cm diameter piston gage as a function of pressure, gas, and mode of operation. "X" indicates absolute mode values, "0" indicates differential mode with a reference pressure of 93 kPa. The points with error bars are average values obtained for nitrogen over several week's time. The error bars are ± 1 standard deviation. The helium and argon points were single determinations. The apparent change in area with pressure between 40 and 100 kPa is due to a systematic nonlinearity in the UIM. This nonlinearity is the same for both modes of operation and all gases.

Dependence on Gas Species and Mode of Operation

For all gages we have determined the effective areas in the absolute mode for both nitrogen and helium. For many of the gages we have made measurements in the differential mode as well, and in some cases both the absolute and the differential mode measurements have also been made with argon. For some of the gages no difference could be detected in the effective areas for different gases or different modes of operation to within the 2-4 ppm errors that are typical of these experiments. However, much more commonly, we observed differences in the effective areas for different gases, and the area changed for absolute versus differential mode operation. These changes were as large as 25 ppm in an extreme case. An illustration of these effects for one gage is shown in Fig. 7, where effective areas for helium, argon, and nitrogen in the differential mode can be seen to differ from one another and from the absolute mode effective areas for all three gases, the latter being virtually the same to within the errors of the experiment. For other gages we have found, for helium and nitrogen, the absolute mode effective areas to depend on the gas species while the differential mode areas did not.

Summary of Piston Gage Results

The results presented here illustrate that the dependence of the effective area on piston height and the effect of weight rotation rate must be taken into account if piston gages are to be used as transfer standards at the ppm level. They also show that the effective area depends on the operating gas, the mode of operation (absolute or differential), and the pressure. The magnitude of these effects varies from gage to gage, up to 20-25 ppm in the extreme case. In many cases the areas also vary with pressure at low pressures. Unfortunately, no consistent pattern has emerged for these effects from our measurements. As an example, to within experimental errors, it is clear that for some gages we did not detect significant differences among the absolute mode effective areas for nitrogen, helium and argon; however, for all gases there were significant differences between the absolute and differential mode effective areas, with the largest differences for helium and argon. Conversely, for one gage we could not detect differences between the differential mode helium and nitrogen areas, nor the absolute mode helium areas, but the absolute mode nitrogen area did have a large difference from the others.

Primary standard piston gage effective areas determined without taking these effects into account will clearly have significant uncertainties. Similarly, if piston gages are to be used as transfer standards at the ppm level, or even the 20-30 ppm level, the magnitude of these effects must be known for individual gages, or we must develop a theory to explain these effects and allow us to predict the performance of individual gages.

ACKNOWLEDGMENTS

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