

The NBS Ultrasonic Interferometer Manometer
and Studies of Gas-Operated Piston Gages

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ABSTRACT

A series of mercury manometers, with full scale ranges from 13 to 350 kPa, using an ultrasonic technique to measure the column lengths, have been developed at the National Bureau of Standards. These manometers have a three-sigma systematic uncertainty of 15 parts per million (ppm) of the reading, and random errors of 10 mPa + 1.7 ppm of the reading. They are highly automated and are used for research on pressure instrumentation as well as routine calibrations. In particular, the manometers have been used to characterize a group of gas-operated piston gages. Significant pressure dependencies have been found for the effective areas of some gages at low pressures. A dependence of the effective area on operating gas and mode of operation, differential or absolute, has also been found, with the size of the effect varying between gages. For all gages the pressure generated in the differential or gage mode depends on the rotation rate of the piston and weights. The magnitude of this effect depends on the gas, the reference or line pressure, and the outer diameter of the weights.

INTRODUCTION

A large number and variety of industrial processes and research efforts require the measurement of pressure in the range near atmospheric. These measurements may be absolute, differential, or gage, the last being differential measurements referenced to atmospheric pressure. Accuracy requirements vary greatly, but probably include the most demanding of any pressure range. In the case of standards supporting aircraft instrument calibration programs, or used in the determination of thermodynamic properties, uncertainties of 10 parts per million (ppm) or less may be desired. Because of the accuracy requirements and the number of measurements, considerable effort has gone into the design and evaluation of both primary and transfer standards for this range, including the two which are capable of the highest accuracies, the mercury manometer and the gas-operated deadweight piston gage or pressure balance.

Over the past decade a series of manometers have been developed at the National Bureau of Standards that use an ultrasonic technique to measure the heights of the liquid columns. The Ultrasonic Interferometer Manometer (UIM) was originally developed as a low range (13 kPa), high resolution (10 mPa) standard for the calibration of low pressure or "low vacuum" transducers. Initial experience demonstrated that this automated instrument was very easy to use and, at higher pressures, capable of repeatability at the ppm level. A series of design improvements have extended the range to 350 kPa and further improved the ease of use. Improvements in determining the speed of sound in mercury, which limits the accuracy of the ultrasound wavelength, have reduced the systematic uncertainty to 15 ppm. These instruments are in daily use at NBS for routine calibrations of pressure transfer standards, and for research on the characteristics of high performance pressure transducers and gas-operated piston gages.

It is generally conceded that at low pressures deadweight piston gages are not capable of the ultimate accuracy that can be achieved with mercury manometers, and are usually somewhat more awkward to use, particularly in measuring absolute pressures. However, they are much simpler (and less expensive) than a high accuracy manometer, less susceptible to environmental perturbations and more forgiving of operator errors. Under controlled conditions the better designs can routinely achieve ppm short-term repeatabilities. For these reasons they are widely used as transfer standards and, at pressures above the range of mercury manometers, as primary standards. With current trends towards improved accuracy there is interest in determining what factors affect gas-operated piston gage performance at the ppm level, how operating parameters should be controlled for the best accuracy, and what factors must be taken into account in the theory of primary standards piston gages.

This paper will briefly describe the design and performance of the UIM's, and results obtained with UIM's on the characteristics of one model of a gas-operated piston gage.

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 :

$$P = \rho gh \quad (1)$$

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. Low pressure, high resolution 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 (to 484 ppm K^{-1}) of an ordinary mercury manometer, 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 $15 \mu\text{s}$ -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.

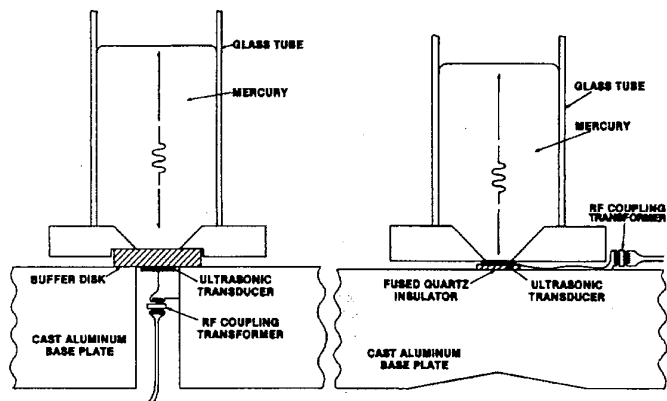


Figure 1. In different versions of the UIM ultrasound is transmitted to and from the mercury in two different ways. On the left an optically polished beryllium disk is used as a buffer material between the transducer and the mercury, on the right a single crystal quartz transducer is placed in direct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

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 multiples of $\pi/2$, but this is susceptible to error if the data are interrupted. In the newer systems measurements are made at four unevenly spaced frequencies between 9.5 and 0.5 MHz. Using an exact fractions algorithm⁽¹⁾, these are combined with a relatively crude time of flight measurement. This permits the measurement of lengths, 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. Operation of this system is more fully described in Ref. 2.

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⁽³⁾. 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 design of the structure and the operation of an early version of the manometer and its electronics are described in Ref. 4. 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

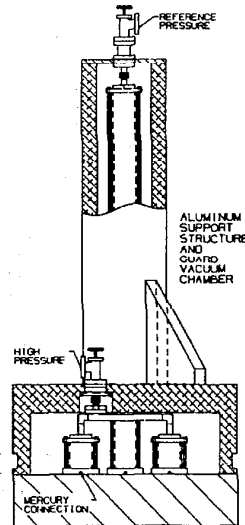


Figure 2. Schematic of the longer range UIM's. The three columns are connected at the bottom by stainless steel plumbing, not shown, attached to the stainless flanges at the bottom of the glass tubes. Mercury is supplied to the center column from stainless steel reservoirs, also not shown. Except at 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.

static changes. In the 13 kPa range UIM, described in Ref. 4, all three tubes are 120 mm long. The later, longer range instruments, illustrated in Fig. 2, differ from that described in Ref. 4 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 350 kPa UIM. This permits the maintenance of the 10^{-5} mm height imprecision, and corresponding pressure imprecision, for low pressure measurements, 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 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.

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. 2, 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 measurement in both the absolute mode ("zero" reference pressure) and differential or gage mode ("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. 5. The standard deviation of the height measurement for a single column in the 40-90 mm range is 10^{-3} mm. Random errors in the measured pressure will include those due to the measurement of the heights of the three columns and temperature perturbations. If ambient temperature changes do not exceed 0.1-0.2 C over any eight-hour period the standard deviation of measurements with zero applied differential pressure over a 24 hour period is 2-3 mPa. If proper corrections are made for errors in the phase measurements system, systematic nonlinearities at low pressures are less than the random errors. The random errors of high precision low pressure transducer calibrations are consistent with a 2-3 mPa standard deviation. 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. This is consistent with the standard deviations of absolute mode piston gage effective area determinations. Long-term stability has been monitored by the repeated calibration of a gas-operated piston gage over a four year period. Average values of the effective area have changed by less than 1 ppm. Analysis of these measurements gives an estimate of the random error of the UIM's at the three sigma level, of 10 mPa : 1.7 ppm.

The known systematic uncertainties are listed in Table I. Cook's mean value for the density of mercury at 20 C and 100 kPa is used with its attendant uncertainty of 0.6 ppm⁽⁶⁾. Correction of the density over the temperature and pressure range of UIM operation adds small additional uncertainties because of uncertainties in the compressibility and thermal expansion of mercury. Significant variations of the density of individual samples from Cook's mean value are theoretically possible because of isotopic composition variations. Based on Cook's measurements of six different samples and a measurement of a sample purified in our laboratory⁽⁷⁾, we have added an additional uncertainty of 1.5 ppm for this contingency. The systematic calibration and measurement uncertainty of the platinum resistance thermometers contribute a systematic uncertainty of less than 2 mK in the mercury temperature, corresponding to 1 ppm in the pressure. The acceleration of gravity has been measured at the UIM location with an uncertainty of 0.02 ppm. However, corrections are not made in our calculations for diurnal variations, which introduce an uncertainty of 0.2 ppm. The reference oscillator of the frequency synthesizer has been found to be within 0.1 ppm of the assumed value, and comparisons of different synthesizers indicate instabilities with time of less than 0.1 ppm. The ultrasonic signal propagates along an axis that is adjusted to within 100-200 μ rad of the vertical, so the combined uncertainty contributed by the non-verticality of the three columns is 0.1 ppm.

The largest uncertainty is in 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⁽²⁾. 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.

Since the component systematic error contributions in Table I are dominated by two sources, which could be correlated, we have chosen to linearly sum those errors to obtain a total systematic uncertainty of 15 ppm.

TABLE I

Uncertainties of the Ultrasonic Interferometer Manometers, absolute and differential pressures. Some of the components of the systematic or type B uncertainties are correlated, so all systematic components have been linearly summed to obtain the total.

Systematic or Type B (three sigma)	
	ppm
Mercury Density	
Cook's mean value	0.6
Variations Due to:	
Isotopic Composition	1.5
Compressibility	0.2
Thermal Expansion	0.05
Acceleration of Gravity	0.2
Temperature	1.0
Speed of Sound	
Measured Value	4.3
Isotopic Variation	0.75
Ultrasonic Frequency	0.1
Verticality	0.1
Ultrasonic Phase	<u>6.0</u>
Total Systematic Uncertainty	15 ppm

Random or Type A

Evaluated at three times the standard deviation.

1.7 ppm + 10 mPa

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

$$P = mg/A_{eff} \quad (2)$$

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 often 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. Elementary flow theories predict that the effective area will be very nearly the mean of the geometric cross-sectional areas of the piston and cylinder. If the cross sections can be determined from dimensional measurements, the effective area can be calculated from such theories, and the piston gage considered a primary pressure standard. The uncertainty is generally considered to be determined largely by the uncertainty of the diameter measurements and the departures of the piston and cylinder from perfect cylindrical shapes. The effective area may also be determined directly from Eq. 2 by calibrating the gage against a pressure standard, for instance, a mercury manometer, thus establishing the piston gage as a transfer standard.

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 determined

by interactions between the pressure fluid and the piston. Attempts to calculate the force on the piston from first principles have been frustrated by a lack of knowledge of the microscopic geometry of the piston and the molecular interactions between gas molecules and the piston and cylinder surfaces. These will clearly vary from one set of piston and cylinders to another and with the gas. What is not so clear is what magnitude of error will be caused by these variations when the effective area of a primary standard is calculated from dimensional measurements and elementary flow theory, or what error will occur when a transfer standard is calibrated under one set of conditions and used under another, e.g., a different gas.

Some idea of the possible magnitude of these effects can be obtained by determining piston gage effective areas as a function of gas species and pressure using an independent standard, e.g., a mercury manometer. We have obtained such data for six different gas-operated piston gages over a period of five years using a UIM, which is a primary reference independent of gas species or mode of operation. We have not obtained data over the full range of parameters of interest for all of the gages, and the quality of the data vary, depending on the quality and performance of the individual gages, ambient conditions, and our varying (generally increasing) skill in operating the piston gage-manometer combination. In all cases the differential mode data were of poorer quality than the absolute mode data. This was due in part to the added difficulty of having to maintain the two stable pressures required for a differential pressure measurement, and errors in correcting for weight rotation rate effects, discussed below, but we also believe that the piston gages inherently operate better in the absolute mode. We have also examined the effect of piston and weight rotation rate when gages are operated in the differential or gage mode. Rotation of the weights in the differential mode can cause large forces and errors that must be minimized or corrected if reliable results are to be obtained.

The six gages are all of the same design. They have nominal piston diameters of 1 cm and full-scale pressure ranges of 50 psi (340 kPa). Five were of standard commercial construction, employing 440C, a hardenable stainless steel, for the piston and cylinder. The sixth was independently fabricated to the same dimensions. The gages were operated using an NBS- designed piston gage base. This base 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). Precautions were taken to stabilize the piston gage temperature and to minimize temperature-induced pressure perturbations and pressure differences between the piston gage and the UIM. In particular, 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, and the same reference pressure was applied directly to the UIM by a separate line between the bell jar and the UIM. The pressure applied to the bottom of the piston gage was also applied directly to the UIM without an intervening differential transducer. For the differential mode measurements the reference pressures were generally maintained at 93 kPa \pm 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 3 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 in Figs. 3-6 were obtained using the UIM.)

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 were 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.

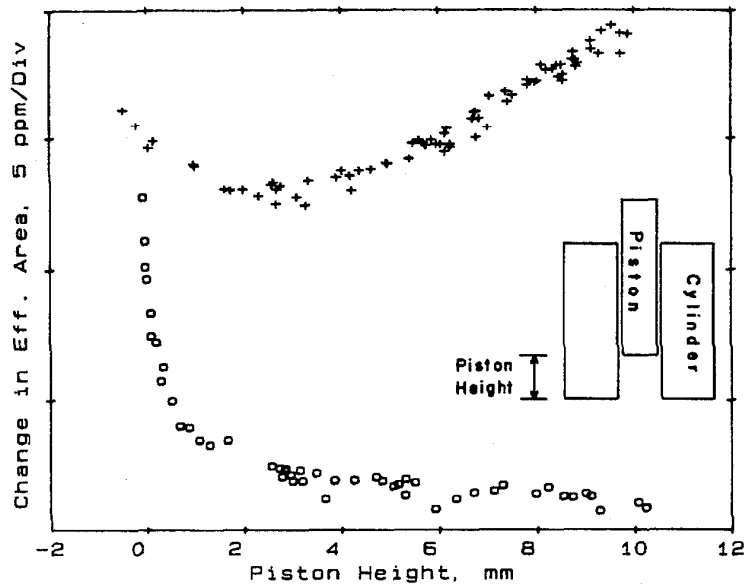


Figure 3. Variation of the effective areas with piston height of two different piston gages. At a given pressure these data are typically obtained over a 30-100 minute period, with 50 seconds required to obtain each data point.

The characteristics of the four other gages varied; in some cases the effective area varied by no more than 1 or 2 ppm over a 6-10 mm range, but in another case it had a large linear variation of 1.4 ppm/mm for almost the entire 15 mm range over which the gage would operate. Effective areas were determined as a function of piston height for four of the gages over most or all of a 10 to 160 kPa range in the absolute mode with helium and nitrogen. For three of the gages data show the variation of effective area was unchanged with pressure or gas. However, for a fourth gage there is a definite change in the dependence of the effective area on piston height as the pressure is reduced below about 30-40 kPa.

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. Beyond this, the apparent changes in the dependence of the effective area on height at low pressures for one gage suggests the influence of non-geometric factors and the possibility of errors when calculating an effective area from dimensional measurements for a primary standard.

Change of Effective Areas with Pressure

Figure 4 illustrates the pressure dependence of effective area for nitrogen in the absolute mode as a function of pressure for one of the four gages for which we have data below 50 kPa. Similar changes of the effective area at low pressures have been seen for two other gages, although in one case the effective area decreased with decreasing pressure. A fourth gage showed no significant change down to 10 kPa.

An obvious explanation for the data in Fig. 4 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. 4. Another possible explanation are magnetic forces 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 (in this gage design the top of the piston always protrudes through the top of the cylinder). It is possible that the changes in area at low pressure, as seen in Fig. 4, are caused by changes in the behavior of the gas in the annulus.

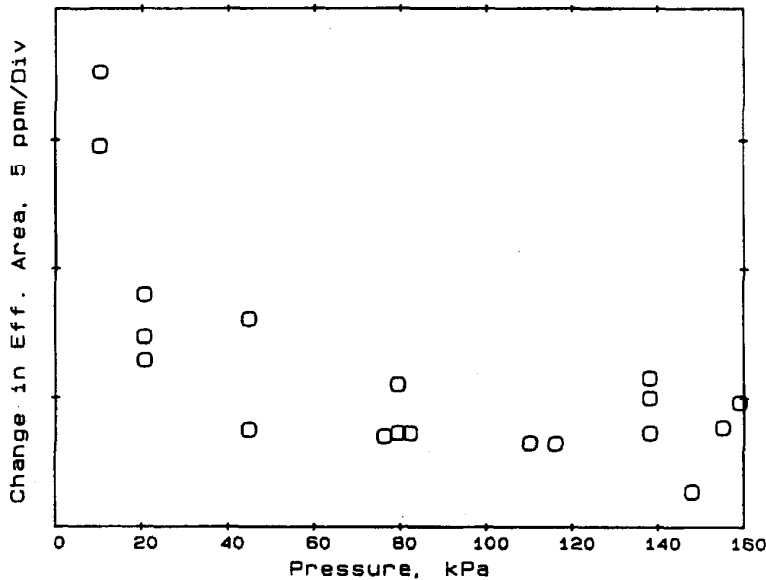


Figure 4. The effective areas for one gage operated with nitrogen in the absolute mode. Each point is the average of 30 to 100 points taken over a corresponding number of minutes. Data at different pressures were taken over a six month's period.

Piston and Weight Rotational Rate

The piston and weights are rotated with respect to the cylinder so that hydrodynamic forces will center the piston within the cylinder, avoiding metal-to-metal contact and relieving frictional forces between the piston and cylinder. This rotation rate may be as high as 16 Hz in some commercial piston gage bases. 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 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⁽⁸⁾.

There is a concern that during normal operation irregularities in the piston or cylinder surface will generate a force on the piston dependent on the rate and direction of rotation ("corkscrew" effect). For the poorest performing of the six gages, we did see such a change with rotational direction, amounting to a pressure difference of 0.3 Pa at an absolute helium pressure of 114 kPa. However, this difference was independent of rate of rotation. For the other gages we saw no evidence for such an effect at the 1 ppm level.

There is clear evidence for a significant aerodynamic force on the weights, dependent on the rate of rotation, when operated in the gage or differential mode^(9,10). 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 rotational rate-dependent effect for several gases and different types of weights. Preliminary results⁽¹¹⁾ 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. 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. 5 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 slightly larger diameter (116 mm) than those supplied by the piston gage manufacturer (114 mm), operating in a nitrogen reference gas at 93 kPa. The differential pressure was 121 kPa.

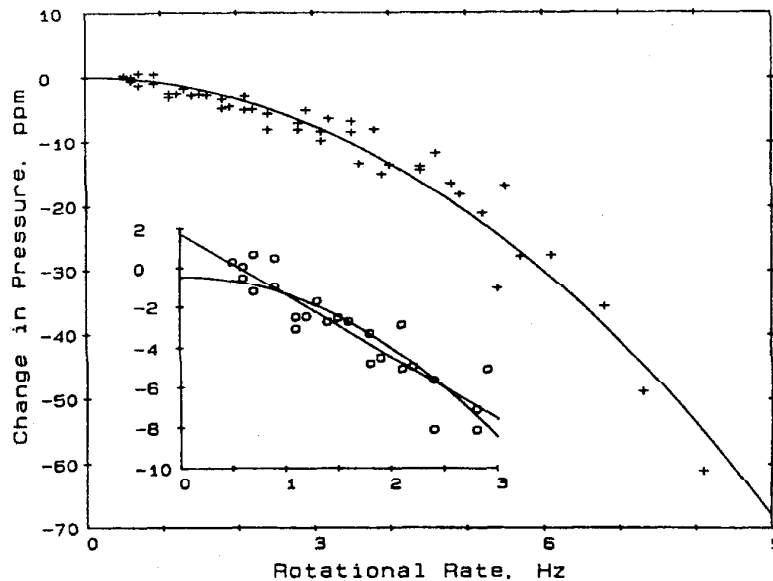


Figure 5. Variation of the nitrogen differential pressure generated by a piston gage with the 116 mm diameter weights rotating in a reference atmosphere at 93 kPa. The curve is a quadratic function of the rotation rate determined by a least squares fit to the data. The linear and quadratic curves in 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.

In determining effective areas in the gage or differential mode we use the zero-frequency intercept of the fitted quadratic equations. These fitted equations can also be used to illustrate the rotation rate effects for different gases and weight diameter. Figure 6 shows the change in pressure with rotation rate for different gases. All values are relative to the pressure generated by this gage with nitrogen in the absolute mode, with gas buoyancy effects taken into account. Two different curves are shown for argon, the one with the large change obtained with the 116 mm diameter weights, the one with the small change, labelled "Ar-small wts", obtained with 82 mm diameter weights designed to minimize the rotation rate effects. If the outside diameter of the weights is further reduced the generated pressure will increase with rotation rate. The difference between the zero frequency intercepts of the two argon curves is typical of the errors of this experiment.

The helium and nitrogen curves were obtained using the 116 mm weights. The dependence of the rotation weight effect on gas species is evident from the three curves obtained with the large weights. As discussed below, to within the errors indicated by the two argon curves, the offsets from zero of the zero frequency intercepts are a measure of the changes in effective area, for this gage, that depend on the mode of operation and gas species.

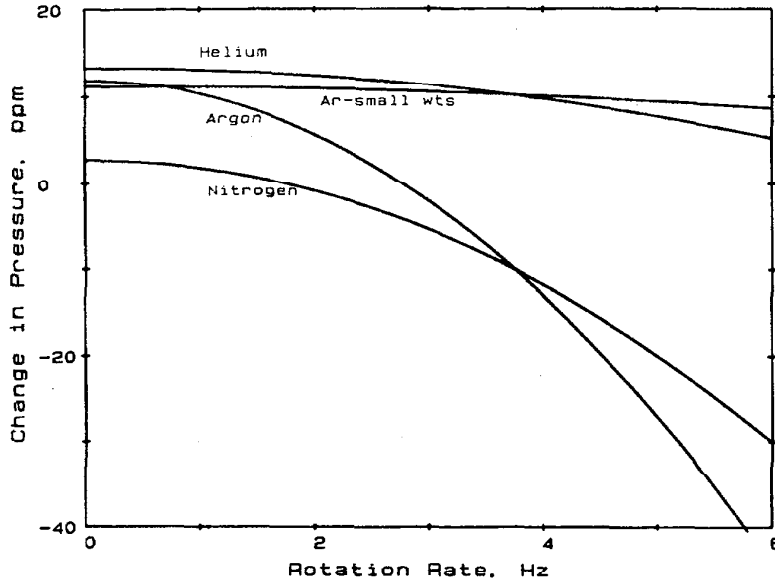


Figure 6. Quadratic curves least squares fit to the differential pressure generated for different gases as a function of the rotation rate of the weights. The reference pressures were 93 kPa. The "Ar-small wts" data were obtained with 82 mm diameter weights designed to minimize the dependence on rotation rate. All values are with respect to the pressure generated with nitrogen in the absolute mode, corrected for gas buoyancy forces on the weights. The offsets at zero frequency indicate changes in this gage's effective area with different gases and modes of operation.

Dependence on Gas Species and Mode of Operation

For all six gages effective areas were determined for nitrogen in both the absolute and differential modes. Helium effective areas were also determined in both modes for five of the gages, and argon effective areas in both modes for two of the gages. All differential measurements were made with a reference pressure of 93 kPa. The amount of data and number of pressures at which measurements were made varied considerably from gage to gage. In all cases the precautions previously described were taken to minimize errors due to piston height and weight rotational rate effects. In addition, care was taken to accurately correct the pressure measured at the manometer mercury surface to the bottom of the piston, located 70 cm higher. The accuracy of this correction was checked by comparing the readings of two UIM's operating at different heights using different gases. Within an imprecision of about 1 ppm there were no differences in the two UIM corrected pressures for helium, nitrogen, and argon.

The results of these measurements are summarized in Table II, which lists the differences in the effective areas for a given gage relative to the absolute mode nitrogen area for that gage. Note that Table II gives the amount by which the absolute mode nitrogen area exceeds the other areas, i.e., in all but one case (gage C, helium, absolute mode) the absolute mode nitrogen areas were the largest. The differences tended to vary with pressure, and the range of values listed in Table II indicates the range of observed differences, going from the lowest to the highest pressures. As noted before, the quality

of the data varies from gage to gage. The best absolute mode data have typical standard deviations of 0.3 to 1 ppm, while typical standard deviations of the best differential mode data are 0.5 to 2 ppm. In the worst cases random errors were about three times larger. Within these limits significant differences in effective area for different gases and modes of operation were observed for several of the gages.

TABLE II

Differences in parts-per-million between the effective areas determined for nitrogen in the absolute mode and those determined for helium and argon in the absolute mode and all three gases in the differential mode (reference pressure = 93 kPa). Where a range of values is indicated the value observed at the lowest pressure is given first followed by the value observed at the highest pressure. As explained in the text, some observed differences are less than experimental uncertainties and may not be significant.

$$[A_{eff}(N_2, Abs) - A_{eff}(gas, mode)] / A_{eff}(N_2, Abs), \text{ ppm}$$

Gage	He Abs	Ar Abs	N ₂ Diff	He Diff	Ar Diff
A	1	0 → 2	1 → 8	11 → 12	9 → 7
B	14 → 18		13 → 21	15 → 23	
C	-2 → 0		0 → 5	3 → 2	
D	3 → 4	0	5	4 → 5	6
E	3		4	3 → 8	
F	0		5		

For gage A a large amount of data was obtained for both modes of operation between 10 and 140 kPa. These data show no differences greater than 2 ppm in the absolute mode areas for all three gases. The differential mode effective areas did show definite differences, with the largest differences being for helium and argon. An example of these differences can be seen in Fig. 6. This gage operated very well, had a reasonable dependence of the effective area on piston height, and could be categorized as a "good" transfer gage.

For gage B data were obtained for helium and nitrogen at only two pressures, 59 and 119 kPa, in both modes. Since the data in Table II are referenced to the absolute mode nitrogen values it may not be readily apparent, but to within the errors of this experiment the differential mode helium and nitrogen and absolute mode helium effective areas were the same. However, a large and significant difference from those values was observed for the absolute mode nitrogen effective areas, which also increased by 11 ppm in going from 59 to 119 kPa. This gage has been extensively studied in a separate set of experiments by B. E. Welch of this laboratory⁽¹²⁾. Those results are in qualitative agreement with those obtained by Welch. This gage was the one of the six that was not of commercial manufacture and it had a large linear change of effective area with piston height, but operated well.

For gage C, data exist from 10 to 160 kPa, primarily in the absolute mode. The 2 ppm difference listed in Table II between the nitrogen and helium absolute mode effective areas is greater than the experimental errors for this gage. The indicated differences in the differential mode are probably within the larger errors which are observed for that mode of operation. This gage has been used for 20 years as an NBS calibration reference standard and shows signs of that use, requiring frequent cleaning. At higher pressures the effective area has a relatively small dependence on piston height, but below 40 kPa there is a significant change in this characteristic. Changes with pressure in the absolute mode nitrogen effective areas are shown in Fig. 4. The nitrogen effective area above 50 kPa, as measured by the UIM, has changed by less than 1 ppm over four years' time.

Gage D was not a high quality gage. Its effective area as a function of piston height is illustrated in Fig. 3 ("+"), and it showed evidence of mass change, possibly due to water adsorbed on anodized aluminum surfaces and/or trapped cleaning fluid. Nevertheless, enough data were taken between 40 and 130 kPa to indicate significant systematic changes in the effective area of the order indicated in Table II.

Limited but high quality data were obtained for gage E at 59 and 119 kPa. These indicate significant differences between the helium absolute and differential mode and nitrogen absolute mode data. The difference indicated for the differential mode nitrogen marginally exceeds the experimental errors. This gage operated very well and its effective area had little dependence on piston height as shown in Fig. 3, ("0").

Absolute mode nitrogen data are available for gage F from 10 to 160 kPa. The much more limited absolute mode helium and gage mode nitrogen data indicate no differences in the effective areas outside the probable experimental errors. This gage operated moderately well.

Summary of Piston Gage Results

The results presented here illustrate that the effective area as a function of 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 and the pressure (the dependence on mode of operation is presumably a pressure or density dependence). The magnitude of these effects varies from gage to gage, up to 20-25 ppm in the extreme case. They also clearly vary with pressure. Unfortunately, no consistent pattern has emerged for these effects from our measurements. As an example, to within experimental errors, it is clear that for gage A 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 gage B 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 added uncertainties. Development of a theory to properly take these effects into account may be aided by systematic investigations of "bad" gages, i.e., those that show large gas or pressure dependant effects.

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