Reliability of high vacuum measurements

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(Received 19 August 1982; accepted 3 December 1982)

In order to allow the users of vacuum measurements to assess the reliability of their measurements, the current state of high-vacuum standards and gauging is reviewed. The discussion includes several types of high-vacuum primary standards currently in use, an assessment of probable errors for several types of hot filament ion gauges, user-controlled factors that may serve to increase the errors, and the information available to date on the performance of a commercially available molecular drag gauge. Examples are given of the introduction of additional errors by the improper application of vacuum gauges.

PACS numbers: 07.30.Hd, 07.30.Dz

I. INTRODUCTION

The development of vacuum science and technology has depended in no small way on vacuum measurements. Reliable vacuum pressure measurements were needed to verify the performance of new pumps and vacuum system construction techniques, and the pressure gauge was at one time one of the few quantitative instruments available to the surface scientist. The development of the Bayard–Alpert (BA) gauge[1] was a major advance that initiated two decades of intensive work on vacuum standards and gauging. Not altogether coincidentally, this period was also one of rapid maturation of vacuum technology, making possible major research and development projects, such as the space program and the fusion energy effort, as well as large scale industrial processes. With this maturation has come changes in both the requirements and capabilities for vacuum measurements. Critical vacuum measurement requirements are now more likely to be related to determining engineering performance and maintaining efficiency or product quality for a process in the high-vacuum (HV) range rather than to verifying a new breakthrough in the ultra-high-vacuum (UHV) range. Many of the surface effects that limit UHV measurements will not be of major concern, but uncertainties as low as a few percent may be required. A wide range of off-the-shelf commercial instrumentation is available, but in some cases the stress is on convenience features rather than on the control of metrological parameters that may limit accuracy. The user is less likely to know how to operate a primary vacuum pressure standard, and more likely to rely on a calibration laboratory or the accuracy of manufacturers' specified performance. These changes all have an effect on the reliability of vacuum measurements.

If reliability is a measure of the confidence that the user's requirements for accuracy or reproducibility have been met, then both the requirements and capabilities of the user must be considered in assessing the reliability. It is much more difficult to make a measurement at a given pressure and under given conditions with an uncertainty of 10% than with an uncertainty of 100% (a factor of 2). However, it can be fairly said that a user with a 10% requirement who has properly conditioned the vacuum system and gauge, understands the parameters affecting gauge performance, and is using a gauge with known characteristics, is obtaining a far more reliable measurement than a user with a 100% requirement that makes the measurement as the system is being pumped down from air, using an ion gauge tube without a verified sensitivity, and a control unit with digital readout but no control of the emission current.

In order to help the user compare his requirements with current capabilities, the status of HV standards and what is known about the stability and operating characteristics of HV gauges will be reviewed in this article. No attempt has been made to achieve historical completeness; the emphasis is on equipment and techniques that are available and used today. The discussion has been further limited to the HV range, 10^{-4} to 10^{-1} Pa (10^{-6} to 10^{-3} Torr), because of the large number of vacuum measurements made in this range, some with quite stringent accuracy requirements (needs for uncertainties as low as 1% having been identified in the fusion program[2]). New data on HV measurement accuracy are becoming available from vacuum standards programs established at national standards laboratories in several countries. One of the major problems, often the limiting one, in vacuum measurements is establishing the vacuum environment so that the quantity of interest is what is actually measured. The problems here are too varied and diverse to receive justice in this discussion, but examples will be given of errors due to the location of a gauge or the manner in which it is attached to the vacuum system.

II. VACUUM STANDARDS

Commonly used vacuum gauges rely on the measurement of variables that cannot be reliably theoretically related to the quantities generally desired—pressure or gas density. As an example, the operation of an ion gauge is so complicated that it is virtually impossible to theoretically relate the measured variable, the ion current, to a pressure or density with any meaningful accuracy. If it is desired to relate the indicated gauge reading to accepted physical units, the vacuum gauge must be calibrated against a standard that generates or measures a pressure in a manner that can be related to accepted units of measure with a reliably estimated uncertainty. The error of this standard represents an irreducible minimum error for all measurements related to the standard.

Several factors constrain the types of devices that can be used as standards in the HV range. The pressures over the

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entire range are low enough that mechanical-force-per-unit-area standards widely used at higher pressures lack adequate sensitivity. At the high end of the range the gas dynamics go through a transition from being dominated by molecule–vessel collisions (molecular flow) to molecule–molecule collisions (viscous flow). This greatly complicates the theory of some standards and the operation of most gauges. At the low end of the range the effects of outgassing and adsorption become increasingly important. The major approaches to high vacuum standards in use today have been reviewed in detail by Poulter.[3] We will briefly describe the main types of high vacuum standards and note the work appearing since Poulter's review.

A. Mechanical and McLeod standards

The McLeod gauge or compression manometer traps a known volume of gas at the pressure being measured, compresses the gas into a known smaller volume, measures the resultant pressure, and uses the ideal gas law to derive the initial pressure. Generally mercury is used to trap and compress the gas and measure the final pressure. The McLeod gauge has a long and distinguished career in vacuum metrology. It is inexpensive to construct and the theory of operation is straightforward. In its refined forms and with a knowledgable and skillful operator it can achieve uncertainties of a few percent throughout the HV range and has been successfully used in a number of outstanding vacuum experiments. It is also fair to say that some of the poorest vacuum measurement results have been obtained with McLeod gauges. The simple theory of operation may serve to conceal from the unwary user serious problems arising from outgassing, adsorption, capillarity, measurements of small volumes and heights, and pressure gradients between the gauge and cold traps. The operation of the McLeod gauge is relatively slow as well. Although McLeod gauges are still used as primary standards in the high-vacuum range[4,5] the trend has been for their use to decline as alternatives have become available. The casual user of McLeod gauges is advised not to.

A mechanical-force-per-unit-area vacuum standard designed to be used over most of the HV range with an uncertainty of about 1% has recently been described by Warshawsky.[6] It is an extrapolation of a common higher pressure standard, the piston gauge. It measures the force generated by the pressure on a thin disk placed in a close fitting orifice between the pressure chamber and a region of lower pressure.

B. Static expanders

Static or Knudsen expansion devices expand a volume of gas at a readily measured pressure into a larger volume. If the temperature of the gas is known or constant, and the ratio of the volumes are known, the pressure after expansion can be related to the original pressure using the equation of state of the gas. In most cases the ideal gas law suffices. This process can be repeated to obtain lower pressures, giving rise to an alternate name, series expansion. In addition to Poulter's review, Berman has recently treated these types of standards.[7]

The theory of operation of this type of standard is simple and well understood. The practical application can be much more complicated. The necessity to start from a readily measured initial pressure and achieve a low final pressure means that large volume ratios must be accurately measured. In addition, the initial volume is usually small enough that the volumes of valves must be accounted for and maintained constant. Bellows, designed to flex and often used for seals, are an obvious source of concern. For very small volumes it may even be necessary to periodically redetermine the volume ratios to account for the progressive deformation of copper valve sealing disks. Since the system is static and relies on maintaining a fixed quantity of gas, careful checks must be made to ensure that gauge pumping or outgassing are not causing significant pressure changes. As the pressure is lowered, leaks, outgassing, and adsorption effects become increasingly important and any system for use over the HV range must be bakeable and elastomer seals cannot be used. Clearly, active gases must be avoided as well.

Although static expansion systems require great care in construction, may involve substantial expense, and are slow to operate, they have been successfully operated to well below the HV range and have been convincingly demonstrated to achieve uncertainties of a few percent or less over the HV range. Their ability to accurately generate pressures through the transition range between molecular and viscous flow is a significant advantage.

C. Dynamic expanders

Dynamic expanders or orifice flow devices pass a known flow of gas through a known conductance, generally an orifice, to generate a known pressure difference across the conductance, quite analogous to generating a voltage difference by passing a current through a resistor. Variants of this technique using multiple orifices are also known as flow dividers. The limitations and advantages of dynamic expanders are quite different from those of static expanders.

The calculation of conductance at any pressure requires a random distribution of molecular velocities on both sides of the orifice, and pressure inhomogeneities due to the flowing gas must be avoided at all gauge locations. This requires that attention be paid to the baffling of the incoming gas and the maintenance of symmetry and proper geometric ratios throughout the design. Relatively large pumping speeds, typically up to 1 m^3/s for a 10-mm orifice system, are required to maintain satisfactorily low pressures downstream from the conductance. The calculation of orifice conductances further assumes molecular flow conditions, which is an adequate assumption for a typical 10-mm orifice up to about 10^{-2} Pa. Corrections can be made for nonmolecular flow at pressures not too distant from the molecular flow regime and Poulter[8] has described an orifice flow device operating up to 10 Pa. Care must be taken that localized heat sources, such as hot filament gauges, do not significantly perturb the random distribution of molecular velocities.

Since the dynamic system maintains a continual flow of gas, the effects of leaks, outgassing, adsorption, and gauge operation are much less severe than for the static expansion technique. This makes it particularly well suited for low pressures, and allows for its use with active gases. In addition, these devices can change the generated pressure relatively quickly or maintain a stable pressure for long periods of time.

The dynamic expansion technique has been widely used in a number of variants. A system combining a McLeod gauge, a static expansion, and a dynamic expansion has been recently described by Close, Vaughan-Watkins, and Yarwood.[9] Hojo, Ono, and Nakayama have described a dynamic expansion system that is replacing a McLeod gauge as the Japanese national standard.[10] Many different dynamic systems are distinguished by the technique used to measure the gas flow. Peggs has reviewed a number of these techniques.[11] McCulloh[12] describes another variant that passes a known quantity of gas rather than a known flow through the conductance. McCulloh's version is specifically designed for calibration of a molecular drag gauge, described later in this article.

D. Comparison of standards

Evaluating the uncertainty or estimated error of a primary standard is undoubtedly one of the most difficult tasks in metrology. Random uncertainties can be evaluated from repeated measurements, but estimating systematic uncertainties implies a complete theory or model of the operation of the standard. Even if the operation of the instrument is carefully analyzed, component errors evaluated for the significant error sources, and a total estimated error derived using reasonable statistical techniques, the possibility remains that some significant source of error may have gone unrecognized. This possibility can be checked by comparing the results for one primary standard with those of another type, preferable one operated by another laboratory with different experimental techniques and operator biases. Agreement of two different standards within their combined estimated errors helps to establish the reliability of those estimates.

Results have been published for a few intralaboratory comparisons of high-vacuum standards. Comparison of static and dynamic expansion standards with Ar between 10^{-3} and 10^{-1} Pa at the Leybold Company, Cologne, showed agreement to within 4%.[13] Comparison of a McLeod gauge and series expansions apparatus with Ar and N_2 between 3×10^{-4} and 10^{-1} Pa at the National Physical Laboratory (NPL), Teddington, demonstrated agreement to within a few percent.[14] Static and dynamic expanders at the Physikalisch-Technische Bundesanstalt (PTB), Berlin, have been found to have differences of less than 2.5%.[15] A dynamic expander and McLeod gauge at the Electrotechnical Laboratory, Tokyo, agreed to within the 3% uncertainty of the McLeod gauge.[10]

Comparisons between different laboratories are harder to organize and depend much more on the stability of transfer gauges. Three such comparisons between high-vacuum standards have recently been reported in the literature. The first of these[16] concluded that standards at the national standards laboratories of Great Britain, Italy, France, and West Germany were within 2% of a mean value for Ar between 8×10^{-5} and 8×10^{-2} Pa. The data lend definite credence to the claim that well-executed primary vacuum standards can achieve uncertainties of 1% or less. A separate comparison between French and German national standards found a mean difference of 0.35% for Ar at 3×10^{-2} Pa.[17] A comparison between the standards at four United States industrial and military standards laboratories for N_2 between 7×10^{-5} and 1.3×10^{-2} Pa produced quite different results. Differences as large as 25% were reported[18] and there were apparent large nonlinearities in one standard, an unbaked static expansion device with elastomer seals.

It is worth noting that the reported results of two of the comparisons were "sanitized" to eliminate larger difficulties due to obvious problems with the standard or apparent problems in operation of the transfer gauges. These discrepancies, which obviously affect the delivered accuracy, might have gone undetected were it not for the comparison.

III. TRANSFER GAUGES

The best standard is of little use if stable gauges are not available to transfer the measurement capability to the user. There are also numerous cases, indeed the great majority of cases, where the user does not have access to a vacuum calibration facility or cannot justify the effort to obtain and use a calibrated gauge. In all cases the users need to factor in the effects of environmental and operating parameters on gauge performance and the expected long-term stability of different types of gauges in evaluating the reliability of their measurements. The user of uncalibrated gauges should know what magnitude of errors can be expected from uncalibrated gauges and if certain types of gauges are more likely than others to have larger differences between specified and actual sensitivities.

The most commonly used HV gauge is the ion gauge, both of the cold cathode and hot cathode or hot filament types. Cold cathode gauges, noted for their resistance to abuse, are also noted for their instability, and almost no work has been done to characterize their properties as transfer gauges in the HV range. Therefore, when we speak of ion gauges, unless otherwise noted, we will be referring to the hot cathode type. In reporting the results of ion gauge performance tests, the variability of gauges within a type is characterized by the standard deviation of individual values about the mean. If adequate data have been obtained and the performance of the gauges are truly randomly, or more precisely, normally distributed, the standard deviation can be used to predict the performance of the set of gauges as a whole, e.g., 99.7% of the gauges of a given type will have values within a range of three times the standard deviation about the mean value. Calibrating or evaluating ion gauges is slow work and most of the studies have involved a limited number of gauges. Furthermore, as was demonstrated in one NBS study, ion gauge performance may be influenced by unknown systematic effects. In short, making statistical predictions on the basis of the data to be presented may be very risky, but the standard deviation is still a useful comparative "figure of merit."

A quite different family of gauges measures the momentum transfer between the gas molecules and a mechanical object. A gauge utilizing the momentum transfer or molecular drag on a rotating magnetically levitated ball has become commercially available within the past year. It shows promise for use as a stable HV transfer gauge.

A. Ion gauges

The simple model of an ion gauge is that of a measured current of energetic electrons colliding with gas molecules in the active region of the gauge to produce a measured current of ions. The ion current is,

$$I^{+} = KI^{-}\rho, \tag{1}$$

where K is a calibration or sensitivity factor, I^{-} is the ionizing electron current, and ρ is the molar gas density. If the temperature of the gas is known the pressure P can be determined from the density and Eq. (1) becomes

$$I^{+} = KI^{-}\frac{P}{RT}, \tag{2}$$

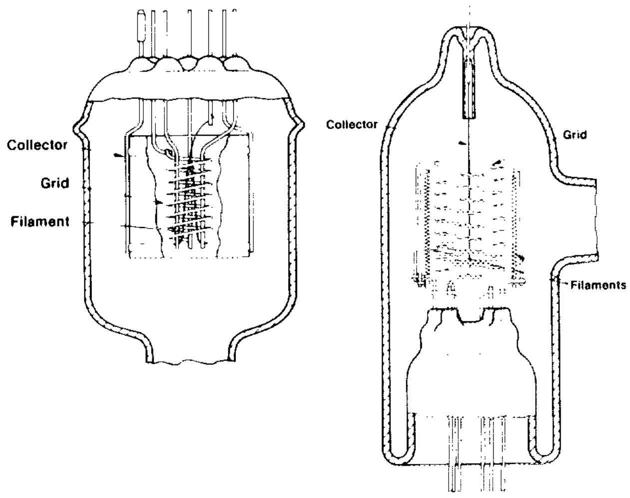
where T is the temperature and R the gas constant. It should be pointed out that the distinction between Eqs. (1) and (2) is important when the gauge is calibrated or specified for a gas at one temperature and used with a gas at another temperature, as often happens with a cryopumped system.

The two most common types of hot cathode gauges, the conventional triode and the BA gauge, are illustrated in Fig. 1. The cathode, a hot filament typically biased at 30 V above ground potential, emits an electron current, typical values ranging from 0.1 to 10 mA. The electrons are accelerated by a potential difference between the filament and grid, typical values being 150 V. The positive ions generated by electron-gas collisions are repelled by the positively biased filament and grid and collected by the anode or collector, generally maintained at ground.

The conventional triode is limited in its measurement of low pressures by soft x rays generated by electron impact at the grid. When the x rays strike the large collector photoelectrons are emitted. The photoelectrons cannot be distinguished from the ions, giving rise to an apparent pressure independent ion current. For a conventional triode this x-ray current is equivalent to a pressure of about 10^{-6} Pa. The inverted BA structure greatly reduces the x-ray cross section of the collector while retaining a high ion collection efficiency. Ion gauge performance can be described in terms of the sensitivity,

$$S = \frac{I^{+} - I_{0}^{+}}{I^{-}(P - P_{0})}.$$

I^{+} is the collector current at pressure P, I_{0}^{+} is the collector current at a pressure P_0. If P_0 is low enough I_{0}^{+} will equal the x-ray current.



Conventional Triode Gage

Bayard-Alpert Gage Opposed Filaments

FIG. 1. Schematic of the electrode structure for tubulated conventional triode and BA gauges with tungsten filaments.

The parameters K and S just defined are occasionally referred to as constants. They are anything but, depending on gas species, electron energies, electrostatic field distributions within the gauge structure, temperature, charge densities, and the surface condition of the electrodes, to name only the more obvious possibilities. These factors can change with time and conditions of use. Attempts have been made to theoretically predict ion gauge characteristics, but it is a very difficult problem and much more is to be gained by experimental investigation of factors affecting ion gauge performance. Much of the work following the introduction of the BA gauge was directed towards the low-pressure limit of gauges and is of limited relevance to the HV range. In addition, many of the gauge types investigated then are not commercially available or little used today, even in the UHV range. However, a recent review of UHV gauging[19] contains a good deal of information relevant to the high-vacuum region, as do earlier reviews.[20,21] It is interesting that the recent work in HV measurements has generated renewed interest in the conventional triode gauge.

It is worth noting that, to a first approximation, mass spectrometers or residual gas analyzers can be considered as ion gauges with a mass filter placed between the ionization region and the collector. Although more care may be taken in their construction, the residual gas analyzer suffers from most of the basic problems that afflict ion gauges, with the additional problems of variability of the mass filter's transmission probability. Although residual gas analyzers provide far more information than ion gauges, and a much better measure of the pressure when the gas composition is unknown, the uncalibrated accuracy of their sensitivity for any specified gas, and the ratio of sensitivities for different gases, will not necessarily be any better than that of an ion gauge. Indeed, the complexity of operation may make them more subject to change with time and use.

Discussions of ion gauge performance are generally concerned with the gauge structure or "gauge tube" alone. This is because it should be relatively easy to reduce errors due to control and measurement of electrical parameters to negligible levels. Our limited experience with commercial controllers does not justify that optimism. Fortunately the user can fairly readily check the relevant electrical parameters. Bias voltages can be measured with a digital multimeter. Emission currents can be monitored by measuring the differential voltage across a resistor placed in the grid line, a 1000-Ω resistor working well for 1-mA emission. The measured grid current is the sum of the filament emission current and the negative of the ion current (the creation of each ion generates an electron that is collected at the grid). The ion current does not equal 1% of the emission current until the upper limit of the HV range is reached. The ion current is generally measured with a feedback-type electrometer, which maintains the collector input very close to ground. This permits the calibration of the electrometer using a known large resistance and a voltage source to generate a known current.

1. Accuracy and stability of sensitivity

The comparisons of measured sensitivities with manufacturers' specified sensitivities, reported below from several NBS studies, were made when the gauges were new, i.e., with only a few hundred operating hours and under carefully controlled conditions. The gauges and calibration system were baked at 200 °C and the grids outgassed by resistance heating. Manufacturers' recommended bias voltages and emission currents were used unless otherwise noted. Given an option of emission currents, we chose 1 mA. Similarly, the other studies reported were generally made under ideal standards laboratory conditions. If these benign conditions were deliberately or accidentally disturbed that is usually noted. Thus, these results generally represent the best performance that could be obtained from these gauges.

Measurements of the N_2 sensitivities of three dual filament UHV-type nude BA gauges[22] found sensitivities varying from 70% to 110% of the specified sensitivity over the high-vacuum range. Significant nonlinearities were evident over this range, as well as systematic differences of 10%–15% between the sensitivities for the two filaments on a particular gauge.

Repeated calibrations of seven "broad-range" BA gauges[23] found the N_2 sensitivities to vary from 52% to 67% of their specified values. Significant nonlinearities, as a function of pressure, were evident for some gauges. This type gauge has a grid with a larger length to diameter ratio than most BA gauges, and a grounded platinum coating on the inside of the tube, both modifications intended to extend the linear operation of the gauge to higher pressures. Random variations of 5%–10% were found under some conditions of use and several gauges exhibited instabilities larger than 10%. Two of the seven gauges were essentially inoperable with hydrogen.

Probably the most widely used HV gauge is the "conventional" BA gauge. This is a tubulated gauge with a grid about 22 mm in diameter and 45 mm long and $\frac{1}{4}$-mm-diam collector. Available from almost any gauge supplier, this type of gauge can be had with three different configurations of filaments: a single thoria-coated iridium ("burn-out proof") filament, two tungsten filaments located "side by side" 8 mm apart on one side of the grid, and two tungsten filaments located on opposite sides of the grid structure. Tests results for conventional BA gauges at NBS can be categorized by the type of filament structure.

Nitrogen sensitivities for eight gauges with thoria-coated filaments are shown in Fig. 2. The mean sensitivity at 10^{-3} Pa differed by − 14% from the specified value and the standard deviation about the mean was 18%. The nonlinearities evident in the figure for some gauges were repeatable for those gauges. Day-to-day random variations in gauge sensitivity were of the order of 5%. An additional measure of the uniformity of this gauge type and the agreement with specified sensitivity is given by arrows on the left side of the figure which indicate the range of sensitivities about the specified value, indicated by the line at the base of the arrows.

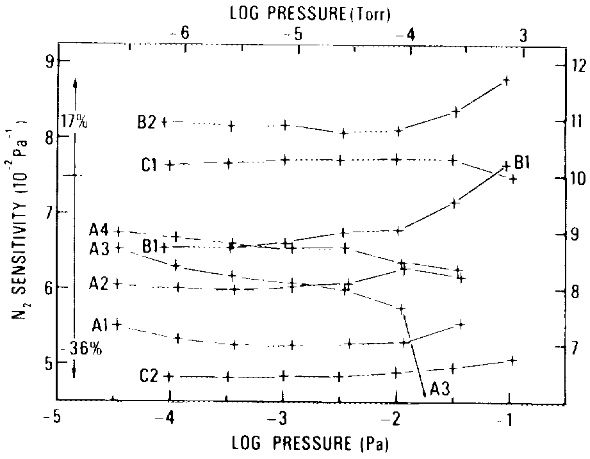


FIG. 2. Nitrogen sensitivities for eight BA gauges with thoria-coated filaments obtained from three different suppliers. Arrows on the left indicate the range of measured sensitivities about the supplier's specified value.

Nitrogen sensitivities for both filaments of four conventional BA gauges with side-by-side tungsten filaments are shown in Fig. 3. The mean sensitivity at 10^{-3} Pa differed from the specified value by 7% and the standard deviation about the mean was 9%. Evident from the figure are systematic differences between the L (left) and R (right) filament for a given gauge. We believe this is due to an asymmetry caused by a grid support structure. Day-to-day random variations were of the order of 2%, but some gauges exhibited

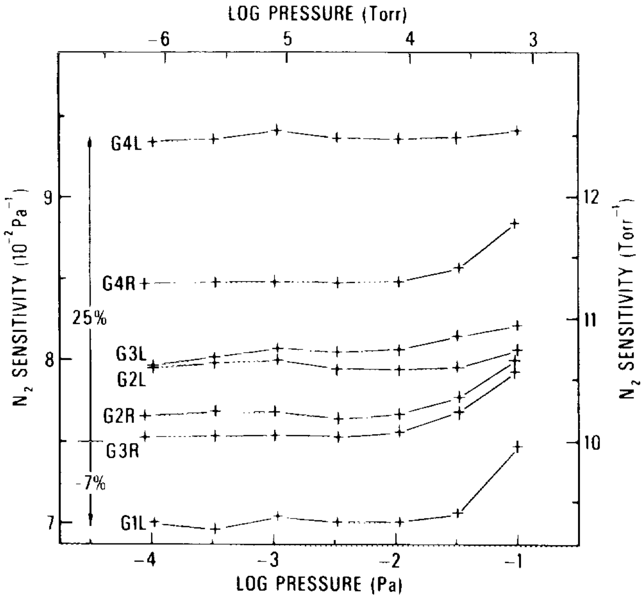


FIG. 3. Nitrogen sensitivities of both tungsten filaments of BA gauges with "side-by-side" filaments. Filament G1R would not operate. Arrows on the left indicate the range of measured sensitivities about the supplier's specified value.

an occasional instability when operated on the L filament in the upper part of the 10^{-2} Pa decade, leading to a sensitivity as low as $\frac{1}{3}$ of the normal value.

Nitrogen sensitivities for both filaments of six opposed conventional BA gauges are shown in Fig. 4. The mean sensitivity at 10^{-3} Pa differed from the specified value by -1% with a standard deviation about the mean of 5%. Day-to-day random variations in sensitivity were of the order of 1%–2%. The three different suppliers of the gauges are designated by different letters on the figures. There is an apparent systematic difference between the gauges from supplier D and F, although the small samples do not permit much significance to be attached to this difference. The gauges designated F, although visually identical to the other gauges except for supplier's brand name, took several days to reach a base reading of about 10^{-7} Pa, while the other conventional BA gauges reached their base reading when the system cooled following bake-out. Although not necessarily of consequence in the HV range, this may well indicate a different processing of the electrode materials.

A variant of the conventional BA gauge with a platinum-coated bulb and thoria-coated filament was not included in the above discussion because of unstable behavior after hydrogen exposure.

The N_2 sensitivities of 145 nude UHV-modulated BA gauges with dual tungsten filaments were found to have a standard deviation of 11% before their installation on the CERN intersecting storage ring.[24] The H_2 sensitivities for 72 of the same gauges had a standard deviation of 15%. Calibrations were performed in the 10^{-6}-Pa decade. About 300 replacement gauges calibrated at CERN in the same range had N_2 sensitivites with a standard deviation of 10% and H_2 sensitivities with a standard deviation of 11%.[25]

Five BA gauges with a single tungsten filament studied at NPL were found to have N_2 sensitivities at 5×10^{-3} Pa with a standard deviation of 14%.[26]

With allowance for the small number of gauges in the NBS and NPL studies and the quite different construction of the CERN gauges, it appears that BA gauges with tungsten filaments can be characterized by standard deviation of the order of 10%. The possibility exists that the "opposed"-type dual filament gauges are more uniform than that. Indeed, experience at NBS with two gauges of the same type as examined in the NPL study would lead us on the basis of day-to-day variations alone to expect that the "opposed" gauges would be more uniform and stable than the NPL gauges. The larger variations indicated for the thoria-coated filament gauges are, in our experience, typical. We believe this is probably due to variations in the temperature and electron emission density along the coatings.

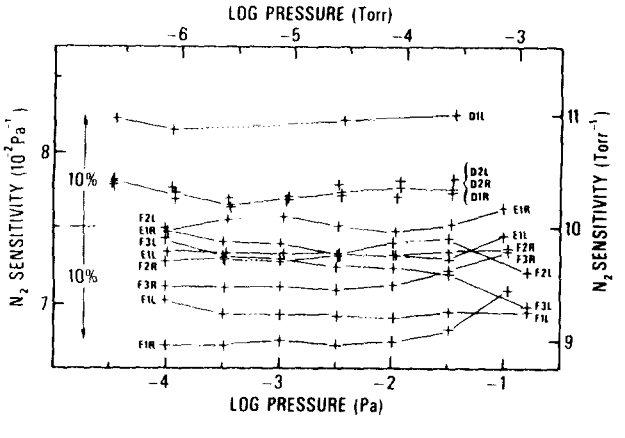


FIG. 4. Nitrogen sensitivities for both filaments of six BA gauges with opposed tungsten filaments obtained from three different suppliers. Arrows on the left indicate the range of measured sensitivities about the supplier's specified value.

Much less information is available about the long-term stability of BA gauges. The problem is complicated by the dependence of gauge stability on operating environment. The NPL study[26] systematically monitored the sensitivity of gauges over a period aggregating 1000 h of operation. One BA gauge kept under vacuum during the entire period changed by 14%. Other gauges exposed to atmosphere (filament off) showed changes as large as 25% between calibrations. Far less systematic observations during the NBS work would indicate stabilities for tungsten filament BA gauges that are significantly better than this.

Messer[16] cites a change in sensitivity of less than 5% for industrial reference gauges over a 1 yr period, but does not specify the type of gauge. In a later paper[27] he gives a range of sensitivity of 1.4% over a three-year period for a rather unusual type of BA gauge. This latter result should not be considered as typical.

After 30 yrs of relative oblivion the conventional triode ion gauge is being studied for use as a stable transfer gauge in the HV range. The cylindrical symmetry and rugged construction of the electrode structure promise simpler and more stable electrostatic fields. Experimental results have tended to confirm that these gauges, equipped with tungsten filaments, are more uniform and stable than BA gauges.

The NPL study cited earlier[26] found 14 triode gauges to have N_2 sensitivities at 5×10^{-5} Pa with a standard deviation of 5%. Nitrogen sensitivities measured between 0.01 and 0.3 Pa of 258 triode gauges at the Electrotechnical Laboratory, Japan, had a standard deviation of 6.5%.[28] NBS studied 20 triode gauges obtained from three different suppliers.[29] The nitrogen sensitivities for 15 of these gauges, operated at the manufacturer's recommended potential and 5-mA emission, are illustrated in Fig. 5. The average sensitivity differed from the specified value by 2% at 10^{-3} Pa and the standard deviation of the sensitivity was 4%. Operation at 1 mA improved the linearity of the gauges. However, calibration of five more gauges, purchased after the original study, found N_2 sensitivities entirely outside the range of values found for the original 15, differences from the mean being as large as 22%. We have been unable to determine a cause for these differences and they serve as an example of the dangers to be incurred in making statistical projections from limited data. In view of the similarity in results for the first 15 gauges and those obtained at NPL and the Electrotechnical Laboratory, we feel this last lot of gauges is somehow systematically different and not representative of this type of gauge.

In spite of the "anomolous" gauges encountered in the

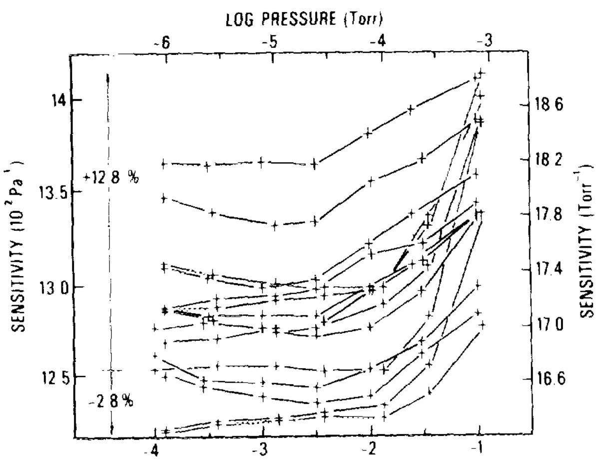


FIG. 5. Nitrogen sensitivities of 15 type-507 conventional triode gauges of the same type obtained from three different suppliers. Arrows on the left indicate the range of measured sensitivities about the supplier's specified value. Operation of these gauges with 1-mA emission, filament and grid bias of 20 and 180 V, and collector grounded improved the linearity and gave an average sensitivity of 0.136 Pa^{-1}. A separate lot of this same type of gauge was found to have sensitivities entirely outside the range shown here.

NBS work, the studies support the idea that conventional triode gauges are more uniform than BA gauges. There is also reason to believe that they will be more stable. Our experience was that typical day-to-day random variations were of the order of 1% if the gauge was conditioned beforehand with the calibration gas. Limited work at NBS with gauges of the same type as studied at NPL indicated similar random variations for that type of gauge as well. Over a longer time period, the Electrotechnical Laboratory study found an unspecified number of triode gauges to have retained their calibration over an 8 yr period to within the accuracy of the McLeod gauge standard, 3%. Operating time was not specified. The NPL study[26] found the triode gauge sensitivities to change at a steady rate that was no larger than 0.45%/100 operating hours. One of the gauges in the NBS study was operated over a period of several thousand hours, including exposure to H_2O, H_2, and O_2. Its sensitivity changed by no more than 14%.

The promising behavior of the triode gauges has prompted attempts to reduce the residual current or x-ray limit of the triode structure.[30] Reductions of about a factor of 20 have been achieved and the modified gauges had stabilities comparable to the parent gauges.[26]

A distinctly different ion gauge[31] that incorporates a shielded ion collector and an electron beam geometry has been used in one reported comparison of standards laboratories.[16] Although not in common use, this so-called JHP gauge is of interest because of the conclusion that the three gauges used in the comparison did not change their sensitivity by more than 1.7% over an 18-month period, including multiple transfers between laboratories.

2. *Relative gas sensitivities*

The sensitivity of an ion gauge will vary with gas species, largely because of varying ionization probabilities. The sensitivities for different gases are often expressed as the ratio of the sensitivity for the specified gas to that for N_2. Argon is also used in some cases as the reference gas. The premise has been made by a number of investigators that the relative sensitivities for a gauge or type of gauge will remain constant in spite of changes in the absolute gas sensitivities. A more daring supposition is that the relative sensitivities for any gauge can be correlated with some property of the gas molecule. The vacuum gauge user in search of reliable relative sensitivity values may question these suppositions since literature values vary a great deal. The variations could, of course, be due to measurement errors.

Holanda[32] reviews the experimental data up to 1973 and attempts to correlate it with different molecular properties. He concludes that the best correlation is with the ionization cross section, and the worst are with the first ionization potential and the number of electrons. Later extensive experimental investigations were conducted by Young[33] and Nakayama and Hojo.[34] These latter investigations looked at BA gauges only and included data for a number of hydrocarbons. Young correlates his results with the number of electrons, Nakayama and Hojo obtain the best correlation with molecular cross sections.

Data obtained at NBS lead us to conclude that such correlations will be useful only as a rough guide, although relative sensitivities may be stable for a given gauge. We see significant differences in the relative sensitivities depending on gauge type and definite pressure dependences. Figure 6 shows relative sensitivities for six conventional triode gauges and four BA gauges. The conventional triode data are the average of the results for six gauges of the same type.[29] Results for individual triode gauges were indistinguishable from the average. Repeated measurements on several gauges over a period of time indicated no changes in related sensitivities even though significant N_2 sensitivity changes occurred during this same time. Ar and He relative sensitivities were measured for the "anomolous" triode gauges and agreed with the data of Fig. 6 to within experimental errors even though the N_2 sensitivities were quite different. These results imply that the relative sensitivities may be constant for this conventional triode gauge structure. However, the relative sensitivities shown in Fig. 6 for the BA gauges, one with side-by-side tungsten filaments, and three with opposed tungsten filaments, not only differ from the triode data, but had significant differences from gauge-to-gauge, indicated by the error bars. Both filaments of the gauges were measured for He, Ar, and H_2. Differences between filaments for a given gauge were much less than the gauge-to-gauge differences, implying that the grid and collector structure may determine the relative sensitivity. Stability as a function of time for the BA gauges has not been checked.

The NBS data can be compared with Young's data[33] since one type of BA gauge was measured in both experiments. The two sets of values agree well at the higher pressures, where Young's data was obtained, for He, H_2, and Ar. Nakayama and Hojo's data and the NBS data agree reasonably well for Ar, and Kr, but significant differences exist for He, Ne, H_2, and Xe. We suspect these differences are due to different gauge structures.

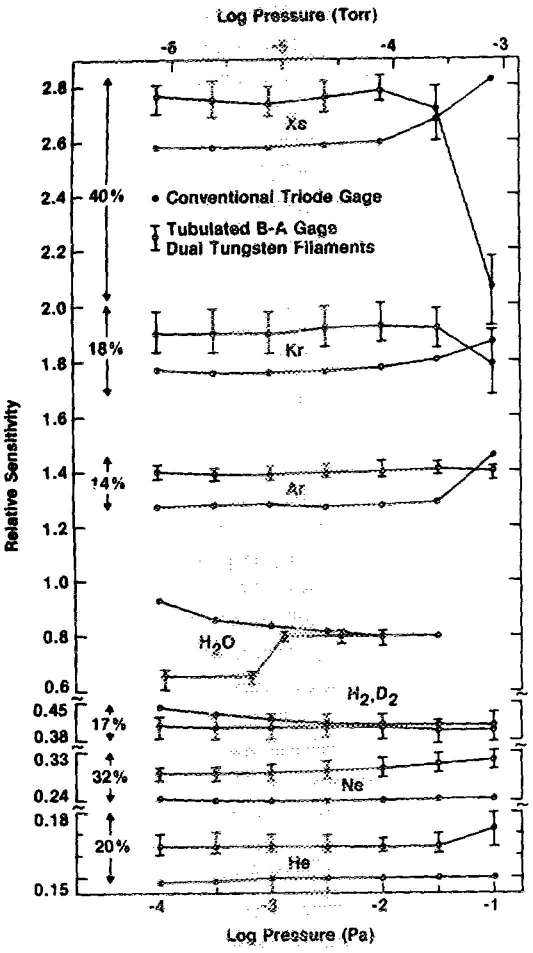


FIG. 6. Sensitivities for different gases relative to N_2. Values for the type-507 conventional triode, shown as dots, are average values. Differences between individual gauges were within the size of the dots. Values for conventional tubulated BA gauges are shown with error bars indicative of the range of values found for the four different gauges tested. The ranges of relative sensitivities for a given gas for all gauges over the entire pressure range are indicated on the left side of the figure.

3. *Variables*

The performance data presented so far were generally obtained under ideal conditions. The user often does not have this luxury and must be aware of what effect different variables will have on the reliability of the measurements. These variables may be beyond operator control, or are controllable, but only with significant effort. The effects of some variables are readily quantified, others are not and only the magnitude of possible errors can be indicated. The list of variables discussed below is not exhaustive, but does cover some of the more obvious areas of concern.

The bias voltage dependences for the conventional triode gauges studied by NBS[29] were a 0.5% change in N_2 sensitivity-per-volt change in filament bias, and a − 1.3% change-per-volt change in grid bias. Similar values were found at NBS for the triode gauges studied at the NPL.[26] Voltage coefficients for BA gauges are typically an order of magnitude less, although anamolously large voltage coefficients, comparable to the values for triode gauges, were found for some "broad-range" BA gauges.[22]

Changing the triode gauge emission current in the NBS study between 0.1 and 5 mA changed the N_2 sensitivity by ± 10% and affected the linearity of the gauges as well. The linearity and relative sensitivity for different emission currents was found to change by a few percent as a function of time and use. Changing the emission current of the BA gauges from 1 to 0.1 mA caused no significant changes in the N_2 sensitivity, but increasing it to 10 mA decreased the low-pressure sensitivity by up to 20% and caused marked high-pressure nonlinearities starting in the 10^{-3}-Pa decade. Operation of conventional BA gauges with 10-mA emission current should therefore be avoided.

Combined density and thermal transpiration effects give an ion gauge response varying inversely as the square root of the absolute temperature of the gas. Determining the gas temperature is a difficult task in a tubulated gauge, particularly the conventional triode type where the gas temperature is determined in part by the envelope temperature, and in part by the large collector, which is heated by the filament to some unknown temperature above the envelope. A careful experiment by Close, Lane, and Yarwood[35] found the gauge reading could be correlated with the square root of the triode envelope temperature, as well as several other functional forms. Work by Kleber[36] and Haefer[37] and our own work[29] found the variation to be approximately ½ that predicted by the square root of the envelope temperature. Differences can be expected due to the different geometry of the gauges studied, i.e., whether a molecule is more likely to collide with the gauge tube or with the warm collector. In a BA gauge most of the molecules probably equilibrate with the envelope, but the envelope temperature is quite asymmetric because of the asymmetric location of the filament. Haefer,[37] on the basis of data from Kleber,[36] did find a correlation with the square root of the temperature of the flange of a nude BA gauge mounted in an enclosure. Close, Lane, and Yarwood[35] found the ion current to change by 0.075% K^{-1}, approximately half what one would expect from the envelope temperature of a BA gauge.

For most room-temperature applications the temperature dependence of an ion gauge is not important. However, for applications involving cryopumps or cryopanels not only may the temperature effects be large, but the gas may be markedly non-Maxwellian. Large directional asymmetries may exist in the density and temperature of the molecular flux. To overlook these effects could introduce order-of-magnitude errors, indeed, the common concepts of pressure have little or no meaning under these conditions. Moore[38] introduced the idea of putting a vacuum gauge into an enclosure with a directional aperture and a known temperature. This "converter" samples the gas flux in a defined direction and allows the reading of the gauge to be related to the flux density. The application of converters and the problems of gauging in a cryogenic environment have been discussed in detail by Kleber[36] and Haefer.[37,39] The presence of asymmetric molecular fluxes can be present in noncryogenic environments as well. Indeed, any pumping of a vacuum chamber requires such an asymmetry and the user should take this possibility into consideration.

A magnetic field will clearly have an effect on ion gauge sensitivity. Several designs of UHV gauges deliberately use a magnetic field to increase the gauge sensitivity by orders of magnitude. Since many vacuum experiments operate in a magnetic field environment, often of varying or unknown magnitude and direction, it is surprising how little data exist on magnetic field dependences of ion gauges. Martin[40] and Normand[41] looked at the effect on both BA and conventional triode gauges in fields up to 0.2 T (2 kG) and 0.03 T (300 G), respectively. Hammond and Riviere[42] looked at conventional triodes and BA gauges with circular filaments in fields up to 0.2 T (2 kG). Hseuh[43] has looked at BA gauges in fields up to 0.006 T (60 G). Berman[44] addresses some related theoretical considerations. The results do not lend themselves to summary. The effects depend on the direction and magnitude of the field, gauge design, and pressure. Normand's data indicate little effect for fields perpendicular to the axis of a conventional triode, the other data appear not to be consistent with that conclusion. The effects are generally nonlinear in both magnetic field and pressure. The general approach of the beleaguered experimentalist has been to try and remove the gauge from the magnetic field or shield it. An example of such shielding is given in Ref. 2. Where possible the efficacy of such measures should be checked by comparing the gauge with another gauge outside the field under verified state conditions.

A major factor influencing a gauge's stability is its history. The HV situation is not so critical as in the UHV region because the surface effects are so quickly saturated. As an example, we have found N_2 pumping speeds for ion gauges operating with 1-mA emission to be less than 0.02 l/s, while values in the literature from UHV experiments are orders of magnitude larger than this. The difference probably arises because under UHV conditions clean surfaces stay clean for long periods and clean surfaces in a gauge can be very effective pumps. However, even in the HV region these surface effects cannot be ignored. Ion gauges will be unstable for several hours following outgassing until the chemical composition and adsorbed layers on the newly cleaned surfaces reach equilibrium. The gas used in a gauge can cause semipermanent changes in its sensitivity. The effects of some gases are worse than others. Young[33] observed sensitivity reactions of up to a factor of 4 in gauges operated with hydrocarbons over a period of about a day due to buildup of carbonaceous deposits on the collector. These changes could be reversed by electron bombardment of the collector, or operation with O_2. Triode gauges appear to be less effected by this than BA gauges.[26] The Blears effect, a reduction in tubulated gauge readings below the true chamber pressure, is due to heavy hydrocarbons depositing on the tubulated inlet, a process that continues indefinitely. Active gases may cause significant changes in gauge sensitivity, although it is not clear in precisely what manner. NBS[29] found that O_2 exposure appeared to reduce the sensitivity of triode gauges while NPL[26] observed increased sensitivity with O_2 exposure. Becker and Messer[45] have systematically investigated sensitivity changes related to changes in the ion collector surface properties. They find that thermal changes caused by outgassing and sputtering during high-pressure operation can have significant effects.

Even with inactive gases time must be allowed for gauge equilibrium to be reached following pressure changes. Our experience is that tungsten filament gauges accommodate to increasing N_2 pressure faster than thoria-coated filament gauges. Tungsten filament gauges will respond to a threefold pressure increase to within 0.1% of the final value within a few minutes. The response in a dirty system, or with active gases, is much longer. The response to decreasing pressures is slower by several orders of magnitude since adsorbed gases on gauge surfaces must establish equilibrium with the lowered pressures. Electron-stimulated desorption of ions from the grid of a hot cathode gauge will make a significant contribution to the ion current in a hot cathode gauge as it is pumped down. An interesting example of the differing response times to increasing and decreasing pressures is the factor-of-10 hysteresis in the indication of an ion gauge (cold cathode) aboard an upper-atmosphere satellite rotating with a 100-min period so that the gauge alternately faces into the incoming gas and back into the satellite's rarefied wake.[46] The magnitude of these effects in particular cases will depend on the gas, the gauge, and its history. Our practice is to pump a gauge two decades below the first calibration point before starting a calibration, to view with skepticism any measurement where the gauge was not first pumped one decade below the pressure measured, and to disbelieve transient measurements. If measurements must be made under less than ideal conditions the user, if possible, should try and repeat the measurement with different pretreatments of the gauge to obtain some idea of the magnitude of possible effects. Transient measurements, if possible, should be repeated at a slower speed.

Changes in geometry of the gauge due to thermal cycling or mechanical shock can affect gauge sensitivity. We believe this factor favors the rugged and symmetric conventional triode structure. Changes in the emission characteristics of the filament are of concern, particularly in thoria-coated filament gauges where the coating can be seen to completely detach from the underlying filament after long periods of use. This readily apparent change must be preceeded by undetected partial detachment of the coating and consequent changes in the spatial distribution of emitted electrons.

B. Molecular drag gauges

A variety of vacuum gauges sensitive to collisions between gas molecules and mechanical bodies have been proposed and a few built. Such a device is attractive because it can be inert, i.e., outgassing, pumping, or chemical reaction effects are eliminated or greatly reduced. Its sensitivity will depend on very stable geometric quantities, and, in some versions, on less predictable surface properties. One such gauge has been refined beyond the point of a laboratory instrument and is now commercially available. This gauge was originally proposed and built by Beams[47] and significant improvements were made by Fremerey.[48] It measures the rotational slowing of a spinning, magnetically levitated steel ball. The gas molecules, in equilibrium with the gauge housing, collide with the moving surface of the ball, reach some degree of equilibrium with that surface, and are reemitted with an average net momentum transfer to the ball. The momentum transfer depends on the speed of the ball's surface, the temperature, molecular weight, and pressure of the gas, the roughness of the ball's surface, and the degree of equilibrium achieved between the gas molecules and the ball. The last two factors depend on the gas and the ball's surface and are characterized by a tangential momentum-accommodation coefficient. The gas pressure can be determined if the rotational frequency and its rate of change can be measured, and the gas temperature and molecular weight and the accommodation coefficient are known. The accuracy of the pressure measurement will be primarily determined by the random noise in the measurement and the accuracy and stability of the accommodation coefficient. In our laboratory in a quiet basement using an 8-min integration time, we obtain random noise levels equivalent to about 10^{-6} Pa. This makes the gauge useful throughout the HV range up to about 1 Pa where gas–gas collisions became significant. The gauge can be used above this pressure,[49] but it becomes increasingly nonlinear. The uniformity and stability of the accommodation coefficient of different balls and different gases has been investigated at PTB[27,50] and work is continuing in this area at several of the national standards laboratories. The PTB work found the argon accommodation coefficients for one ball to vary by less than 1% over a period of 3 yrs. The accommodation coefficients varied by no more than a few percent for four balls from a given lot (4.0-mm-diam stainless-steel ball bearings were used). The accommodation coefficient for the rare gases, N_2, H_2, O_2, CO_2, CO, and CH_4, varied by no more than 4% from one another for a given ball.

The uniformity and stability of this molecular drag gauge using commercial ball bearings is somewhat surprising since the accommodation coefficient will depend on the microscopic details of surface finish[51–53] and chemical composition. In our use of this type of gauge at NBS[12] we have had the accommodation coefficient for specific balls increase by up to 5%. An accidental air bake at 200 °C for several hours increased the values by 1.5%–2%. Abrasion of the surface during a demagnetizing process undoubtedly increased their accommodation coefficients. Exposure to gases such as hydrogen may effect the accommodation coefficient as well. We have not found the 4.5-mm balls supplied with commercial units to be as uniform as the lot reported on in Ref. 50. Further data will have to be acquired on the long-term stability, effects of use and abuse, and possible beneficial effects of pretreatment of the balls. However, it appears evident that only the best of ion gauges, if any at all, will have a stability approaching that of this gauge. In addition, the uniformity from gauge-to-gauge and the predictability of the relative sensitivity for different gases cannot be matched by any ion gauge. It is in many ways well suited for calibration and high-accuracy measurements where the slow speed of response is not a limiting factor.

IV. APPLICATION OF VACUUM GAUGES

As noted, the reliability of vacuum measurements can be comprised in a variety of ways. Beyond the limitations of the instrumentation and its calibration, additional errors will be introduced if the gauge measures something other than what the user thinks it is measuring. One common source of error occurs when the gas composition is unknown or different from what is expected. Outgassing products, from the gauge and the system, or leaks, may give a gas composition yielding a gauge sensitivity quite different from that assumed by the user. Two other examples given below illustrate the general problem.

The "quick-connect" or "O" ring compression fitting commonly used to connect tubulated gauges to vacuum systems will generate an air leak by diffusion through the "O" ring. This leak will not show up during the usual helium leak test unless time is allowed for the helium to diffuse through the elastomer and generally will not significantly affect system performance. However, if the gas leaking in scatters from the connector into the gauge before it goes into the vacuum chamber it can cause a pressure in the gauge offset from that in the chamber. We modified such a connector by boring out the shoulder that serves as a stop for the end of the gauge tubulation. Removal of this shoulder should reduce the probability that the gas leaking in will be scattered 180° back into the gauge. Even with the modification the diffusion leak caused an offset equivalent to a nitrogen pressure of 2×10^{-5} Pa.

Specified pump speeds are the basis for spending large sums of money and are an important parameter in vacuum engineering calculations. Users that verify the speed of pumps often find that their measured values differ significantly from the manufacturers' specified value, and are almost always lower. This does not necessarily imply dishonesty or incompetence on the part of the manufacturer. The difference often has to do with the location and orientation of the vacuum gauge on the test dome used for the speed measurement. It is not universally understood that the commonly used expression,

$$S = Q/P, \tag{4}$$

where S is the pump speed, Q is the gas flow into the test dome, and P is the pressure in the test dome, applies only under certain conditions, such as are found when the test dome is large compared to the pump inlet. The cost of large test domes lead to the drafting of several procedures for pump speed measurements using test domes of the same diameter as the pump inlet. Numerous theoretical studies, many of them cited by Sharma and Sharma,[54] have been conducted to determine gauge location in small domes that will give pressures equivalent to those in a large test dome. However, these small domes are a compromise, differences still exist between the speeds measured with different test domes, and those differences can be greatly exaggerated if the test dome is improperly modified or if the location and orientation of the gauge inlet is changed. Sharma and Sharma[55] obtained gauge readings differing by a factor of 2 for different locations within a given test dome. Experimenters incorporating an apparently innocuous modification in the test dome, or using a more "convenient" gauge location, have obtained pump speeds far exceeding the theoretical maximum conductance of the pump inlet.

V. CONCLUSIONS

It has often turned out to be the case that errors in HV primary standards are unexpectedly large. However, several standards, mostly of the static or dynamic expansion type, have been carefully assessed and can confidently claim errors of only a few percent, and in a few cases, of a percent or slightly less.

The errors of uncalibrated ion gauges and the instabilities of calibrated gauges vary widely and can be increased substantially by failure to control operating parameters, improper conditioning of the gauge and vacuum system, history of the gauge, and failure to insure that the gauge senses the pressure that is supposed to be measured. Of the commonly used ion gauge types, recent data favor the conventional triode gauge as the most stable and reproducible, followed by the conventional tubulated BA gauges with tungsten filaments. The BA gauge design common in the USA with dual tungsten filaments on opposite sides of the grid may have a performance approaching that of the conventional triode. The use of thoria-coated filaments seems to degrade the performance of the gauges, as does the use of platinum coatings on the inside of gauge tubes. Operation of BA gauges with 10-mA emission current causes significant shifts in sensitivity and large high-pressure nonlinearities. UHV-type nude gauges appear to have significant nonlinearities in the HV range at 4-mA emission. Ion gauge relative sensitivities for different gases appear to be constant to within a few percent for a particular gauge, and constant at that level for a given type of conventional triode gauge. However, they definitely do vary between different types of gauges. Under the best of conditions errors in ion gauge measurements can be reduced to a few percent. It is not possible to establish an upper limit, but order-of-magnitude errors have been repeatedly demonstrated.

The molecular drag gauge utilizing a magnetically livitated spinning steel ball has demonstrated long-term instabilities on the order of 1% under carefully controlled conditions. Under more intensive-use conditions these gauges have been observed to change by up to 5% over a 2 yr period. Although more detailed evaluation is required, it appears that the uniformity, stability, and predictability of the sensitivities for different gases appear to be significantly better than can be expected from an ion gauge.

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