

A DETAILED EXAMINATION OF THE PRINCIPLES OF ION GAUGE CALIBRATION

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Abstract

As ionization gauges are adapted to a wider variety of applications, including in particular space research, the calibration accuracy becomes more important. One of the best standards for calibration is the McLeod gauge. Its use must be better understood and better experimental methods applied for satisfactory results. These details are discussed. The theory of the ionization gauge itself is often simplified to the point that a gauge "constant" is often determined in terms of a single measurement as:

$$K = \frac{1}{p} \left(\frac{I_{+}}{I_{-}} \right)$$

Experiments described show that, in three typical gauges of the Bayard-Alpert type, K is not a constant but depends on both p and I_. The best calibration range in electron current is generally less than 5×10^{-6} amp. Significant changes in K with pressure take place in the calibration range of 10^{-4} to 10^{-3} mm. Explanations are offered for the results observed in nitrogen, argon, and helium.



Foreword

Recent developments in vacuum practice have made it necessary to re-examine the basic principles of the operation and calibration of Bayard-Alpert ionization gauges. Some time in the near future it is our intention to continue similar studies to apply to gauges of other designs. Because of the mutual interest in this subject on the part of the M.I.T. Research Laboratory of Electronics and the National Research Equipment Corporation, this study was undertaken jointly both in terms of equipment and personnel time.

The results of this study were presented in brief at the Meeting of the American Vacuum Society in Cleveland, Ohio, on October 12, 1960. It is anticipated that this paper will appear as a part of the Transactions of that meeting when that is prepared sometime in 1961. In case this paper is accepted for the Transactions, then this technical report is a preprint now being made available to those requesting it. Because of the joint interest exhibited by the Research Laboratory of Electronics and the N.R.C. Equipment Corporation, the same technical report is being made available by these two organizations.



INTRODUCTION

Ionization gauges serve many purposes, some of which do not require accuracy of calibration. Some gauges are used to observe relative changes in the vacuum conditions and give atom densities or pressures that are qualitative with the error as large as a tenfold uncertainty. These gauges can be used with the manufacturers' nominal gauge constant. If it is desired to know the atom densities within 5 per cent or better, many details concerning ion gauge calibration must be given careful consideration.

It is the purpose of this paper to discuss some aspects of the physics of ion gauge operation and calibration so that the user who is interested in accuracy will be more critical concerning his methods of calibration and gauge operation than he might have been otherwise.

The standard with which we have the greatest confidence and familiarity is the mercury-column McLeod gauge. Some discussion of its use will be covered. The ionization gauges examined experimentally were: (a) the M.I.T. modified Bayard-Alpert gauge designed by Nottingham and reported in the 1954 vacuum symposium (1), (b) the N.R.C. modified Bayard-Alpert gauge, very similar to the M.I.T. gauge except that it has a conducting coating on the interior glass wall instead of a screen grid, and (c) a standard Westinghouse gauge, type WL-5966. This latter gauge differs from the other two in two important respects which are: (a) the glass wall is permitted to take up that potential for which the net charge to the glass wall is zero, and (b) there are no enclosing structures at the ends of the cylindrical, grid-like electron collector as in the other two gauges. This lack of a grid end structure permits a considerable fraction of the ions produced within the ionization region to escape out of the ends and become neutralized at the glass wall. Some discussion will be given concerning the influences of these design features.

THE McLeod GAUGE

Pressure measurements with a McLeod gauge depend on an application of Boyle's law for gases. The resulting equation is:

$$p = \frac{a}{V_0} (h' - h_0) (\Delta h)$$
(1)

In this equation Δh is the difference in the mercury levels in the open and closed capillaries. This quantity is directly measurable and indicates the pressure difference between the gas compressed in the closed capillary and that in the open capillary. The capillaries must be clean and of equal and uniform cross section. The quantity $(h' - h_o)$ represents the distance, expressed in millimeters, between the mercury surface and the "effective" top of the closed capillary. Before a McLeod gauge can be used for accurate measurements, the location of the effective top of the capillary must be determined experimentally. The area of cross section of the capillaries is denoted by a, the total volume of gas trapped off by the closed capillary and the main bulb of the McLeod gauge by V_o. A consistent system of units is obtained if the area is expressed in square millimeters, the volume in cubic millimeters, and the distance measurements in millimeters. In that case, the pressure will be expressed in millimeters of mercury.

To determine the effective end of the closed capillary, gas pressure is introduced at some arbitrary and unknown value. The distance h' is measured from an arbitrary fiducial line near the top of the closed capillary. A convenient point is the top external surface of the glass that closes this capillary. As the gas in the closed capillary is compressed, three or more readings of Δh and the corresponding h' values can be observed. This set of readings can be related by the following equation:

$$h' = h_{o} + \frac{pV_{o}}{a} \left(\frac{1}{\Delta h}\right)$$
(2)

which shows a linear relation between the observable quantities h' and $(1/\Delta h)$. A plot of h' as a function of $(1/\Delta h)$ should yield a straight line with an intercept at h_0 . For each arbitrarily chosen pressure, the data should yield the same value of h_0 , within the limits of experimental error. If systematic differences occur, the indications are that the capillary is either dirty or nonuniform.

For accuracy it is impractical to attempt to use a McLeod gauge by directly viewing the column heights against a simple ruled scale. The alternative is to use a good cathatometer with a good telescope which can be sighted with high accuracy on the top of the mercury miniscus. Even though the capillaries from which the McLeod gauge was constructed were presumably of uniform bore, a necessary preliminary test must be carried out to show that the mercury rise in the two columns is precisely the same, thus giving a Δh of zero at all points along the capillary when the residual gas pressure is in the upper vacuum range of the order of 10^{-8} mm or better. Experience shows that even with clean mercury and clean capillaries frictional forces between the mercury column and the glass can cause very serious random errors in the readings. These errors can be minimized by a very vigorous tapping of the capillaries, after which the value of Δh under the high vacuum conditions will become zero at all positions or at least follow a systematic pattern of very small differences. The observer must remember that column differences under these conditions of one or two tenths of a millimeter will introduce important errors in the use of the McLeod gauge. The determination of h, with accuracy is not easy. If the capillaries are tapered, a systematic error may show as a reproducible nonlinearity. Repeated measurements will give an indication of the random errors that must be expected.

After h_0 has been determined, then the effective length of the gas-filled part of the closed capillary which in Eq. 1 is $(h' - h_0)$ may be identified by h and Eq. 1 rewritten as:

$$p = \frac{a}{V_0} h(\Delta h)$$
(3)

In actual gauge use, it is generally advisable to make the observation with



Fig. 1. Nomographic chart for conversion of McLeod gauge measurements to gas pressure for a gauge with $(a/V_0) = 2.43 \times 10^{-6}$.

h approximately equal to (Δ h) but it is not always possible to stop the in-flow of mercury with such accuracy that these two quantities are precisely equal to within a tenth of the millimeter. The nomographic chart illustrated as Fig. 1 is applicable to a gauge with a value of (a/V_0) of 2.43 x 10⁻⁶. The method of construction involves the choice of the simple logarithmic scales identified by "h" and " Δ h" in the figure. The center scale located halfway between the two lines has a scaling of two orders of magnitude for the same scale distance as one order of magnitude in the h scales. The center scale is displaced with respect to the others so that the straight line that joins the corresponding unit points will fall at the corresponding pressure point, in this case 2.43 x 10⁻⁶. This chart is very helpful in the determination of McLeod gauge pressure from the observed h and Δ h.

IONIZATION GAUGE THEORY

In the ideal ionization gauge the gas is bombarded by an electron current i_{\perp} and produces at a positive ion current i_{\perp} as given by the following equation:

$$\frac{i_{+}}{i_{-}} = K'_{o} p \tag{4}$$

In the practical ionization gauge, not all of the electron emission current is effective in producing ionization and not all of the ions produced are collected. In order to express these relations arithmetically, the following definitions are written:

$$i_{+} = \frac{I_{+}}{\beta}$$
(5)
(6)

In these equations I_{-} and I_{+} are the <u>observed</u> emission and ion currents which are related to the effective electron currents and the true ion currents by means of the coefficients a and β . Note specifically that the practical way of observing the total emission current is not to insert a meter in the electron collecting electrode but to insert the meter in the cathode circuit so as to read the direct current electron emission from the filament. The ion current, observable as I_{+} , is measured by inserting the meter in the ion collector circuit. A typical circuit arrangement is shown in Fig. 2.



Fig. 2. Circuit used with Bayard-Alpert ionization gauges.

Here the electron accelerating voltage is V_{fg} . The bias of the filament with respect to the ion collector is V_f and the bias of the screened grid, if there is one, with respect to the ion collector is V_s . Unless otherwise specified this V_s is zero but for special purposes it could be either plus or minus. Equation 5 indicated that only a fraction of the total emission current is really effective at producing ions which are collected. Thus a is expected to be a number equal to or less than one, since some of the electrons may either go to the exposed parts of the glass wall and there neutralize ions or they may go directly to the electron-collecting screen or they may even go into the ionization region, become attached to atoms to form negative ions which in turn recombine with positive ions and therefore do not register as effective ionizing agents. It is to be anticipated then that a is not a constant but could depend on both the pressure and the electron emission current as well as the presence of a partial pressure of atoms with a high electron affinity.

Electrons which leave the filament and are accelerated to the grid produce some



Fig. 3. Ionization efficiency P. for various gases as determined by K. T. Compton and C. C. Van Voorhis, Phys. Rev. 27, 724 (1926).

ions which never go to the ion collector but are accelerated directly to the screen or the conducting glass wall in the cases of the M.I.T. and the N.R.C. gauges or may go directly to the glass wall itself as in the Westinghouse gauge. When the mean-free path of the ions is long compared with the dimensions of the ionization region, then a considerable fraction of the ions produced there is accelerated out the ends of the ionization region of the Westinghouse gauge and become neutralized at the glass walls. Thus it is to be anticipated that the β of Eq. 6 will be less than one and it will be pressure-dependent and electron-current dependent. Its pressure dependence in the Westinghouse gauge should be greater than that of the M.I.T. and N.R.C. gauges.

Under low-pressure, low-current conditions the idealization expressed by Eq. 4 may come near to being realized. In that case, we can recognize K'_0 as the product of the effective ionization efficiency factor \overline{P}_i and the average electron path length (L_e) before the electron is collected. Nominally, P_i is an experimentally determined number dependent only on the electron energy for a particular gas. Typical values of this quantity are shown in Fig. 3 which presents data published by Compton and Van Voorhis. Note that for nitrogen, the value of P_i is 11 at 100 volts. This number means that on the average, a 100-volt electron traveling through nitrogen at 1 millimeter pressure and at 0° C will produce an ion in a distance of (1/11) cm. The concentration of nitrogen atoms under this standard condition is 3.54 x 10^{16} atoms per cc.

An ionization gauge really indicates atom concentration and not pressure and yet it

is calibrated directly in terms of pressure when a McLeod gauge is used as the reference standard. Thus in terms of fundamental data of the type illustrated in Fig. 3, the effective value of the ionization efficiency would be $P_i(273/T)$ averaged in some way over the distribution in electron energy within the ionization space. The gas temperature is $T^{O}K$. It would be practically impossible to work out this averaging quantitatively since within the ionization space electron energies range from 0 to V_{fg} . Because of the very sharp gradient of potential in the immediate neighborhood of the ion collector, a very large fraction of the region is characterized by electrons of nearly full energy and therefore it is to be anticipated that \overline{P}_i might very well be only 10 or 15 per cent less than the measured value of P_i appropriate to electrons having the full energy of about 100 volts which they might receive upon acceleration toward the electron-collecting grid. Under these idealized conditions, we have:

$$K'_{o} = L_{e} \overline{P}_{i}$$
(7)

As the pressure increases to the point that the electron mean-free path is comparable with the dimensions of the ionization gauge, we must expect the average electron path L_e to decrease. A still further complication is that the effective ionization efficiency \overline{P}_i could increase as the pressure increases since it would be energetically possible and can be demonstrated experimentally that a single electron can produce on the average as many as 2 or 3 ions before it is collected.

We may assemble all of the factors that relate ionization gauge performance to pressure as applicable in the calibration region of a McLeod gauge which is within a range of pressure of 10^{-5} to 10^{-1} mm (Torr). Equations 8 and 9 show this assembly. The observable quantities I₊ and I₋ combine with the pressure p as shown in Eq. 9 to yield the effective gauge "constant" K which in turn can be related to the other factors mentioned.

$$\frac{I_{+}}{I_{-}} = \alpha\beta L_{e}\overline{P}_{i} p = Kp$$

$$\left(\frac{I_{+}}{I_{-}}\right)\frac{1}{p} = K = \alpha\beta L_{e}\overline{P}_{i}$$
(8)
(9)

All of the factors in Eq. 9, namely, a, β , L_e , and \overline{P}_i are dependent on both the pressure p and the electron emission current I_. Thus K, instead of being a constant of the gauge, is actually a function of these other factors and becomes an observable quantity. Experiments have been undertaken and partially completed on the observed variation of K with pressure and electron current. The next section of this report will show the preliminary results and the following section will offer tentative explanations.

OBSERVED DEPENDENCE OF K ON ELECTRON CURRENT

Calibrations have been carried out using the three gases: helium, argon, and nitrogen. In general, the pressure range used for these studies extended from 10^{-4}



Fig. 4. Ion current as a function of electron current observed on three different gauges operated at the same pressure of nitrogen.

to 10⁻¹ mm. One of the basic requirements that must be imposed for accurate gauge measurements is that the ion current should be directly proportional to the electron current. For two of the gases studied * and the three gauges investigated, the electron current range for which direct proportionality exists is surprisingly low. Under the highest pressure conditions, good linearity exists only in the current range less than 5 microamperes. As the gas pressure is reduced, the maximum electron current seems to increase inversely with the square root of the pressure. Marked differences are found among the three gauges in that the Westinghouse gauge shows good linearity with current over a considerably wider range than that observed in the N.R.C. and the M.I.T. gauges. Typical curves that show the nonlinearity for nitrogen are given in Fig. 4 and similar observations with argon are given in Fig. 5.

In the experiments, the time required for each reading was only a few seconds and the electron current was maintained at the indicated value for just the time required for reading. Checkpoints were made very frequently and, in particular, at the beginning and the end of each run. The changes in gas pressure were always found to be less than two per cent. Over the low current range, it was therefore possible to choose an ion-current electron-current ratio which was independent of the electron current itself. An equation of the form of Eq. 9, applicable specifically to the low-current range can be written as:

$$\left(\frac{I_{+}}{I_{-}}\right)\frac{1}{p} = K_{o} = \left[a_{o}\beta_{o}L_{eo}\overline{P}_{io}\right]$$
(10)

If the product of the quantities given in the square brackets of Eq. 10 were independent of the pressure, then K_0 would be a constant and could properly be used as the gauge

*The helium studies have not yet been completed.



Fig. 5. Ion current as a function of electron current observed on three different gauges operated at the same pressure of argon.



Fig. 6. Gauge "constant" K_o at very low electron current as a function of pressure for argon.



Fig. 7. Gauge "constant" K_0 at very low electron current as a function of pressure for nitrogen and helium.

constant for a particular gas. The fact that ${\rm K}_{\rm O}$ is not constant is illustrated in the next section.

GAUGE SENSITIVITY UNDER LOW ELECTRON CURRENT OPERATION

The most interesting way to present these data is to plot both the pressure and the observed value of K_0 on logarithmic scales. A very good reason for presenting $\log_{10} K_0$ rather than the value of K_0 itself is that <u>relative</u> changes and <u>relative</u> differences for the various gauges are more graphically displayed. Figure 6 shows data for the three different gauges with argon as the gas studied.

The experimental data for nitrogen and helium are summarized by the curves in Fig. 7. Although the log scale applicable to nitrogen is the same as that used for argon, note that the log scale for helium, given at the right side of the figure, has been displaced one order of magnitude. Thus the values of K_0 for helium are practically 10 times smaller than the values for nitrogen, whereas those for nitrogen are only slightly smaller than those for argon. These results are in quantitative agreement with the relative ionization efficiency curves observed by Compton and Van Voorhis and illustrated here as Fig. 3.

INTERPRETATIONS

Most of the main features of the results given in Figs. 4 through 7 are not difficult to interpret, at least semi-quantitatively. The nonlinear performance with electron current follows a pattern except for minor details that is precisely what one would expect from an increased rate of recombination between ions produced in the ion space and electrons. Normally, direct recombination between electrons and ions has a small probability of occurrence. It is therefore assumed that the electrons become attached either to the neutral gas atoms present under study or to some impurity atoms or molecules present in extremely small concentration and yet having a sufficiently high electron affinity to capture a very large fraction of the electrons produced in the ionization process. The reason why such noticeable differences exist between the gauges is explainable in terms of their different design.

All of the curves of K_0 as a function of pressure shown in Figs. 6 and 7, are seen to pass through a maximum. In the M.I.T. and N.R.C. gauges, this rise as the pressure is increased may be attributed to either or both of two effects. One relates to the increase in the effective ionization efficiency of a single electron. This is the factor P_i . Under high-pressure conditions, the ion current has been observed to be as much as double the electron emission current. This means that, on the average, individual electrons produce at least two positive ions before the electrons are removed from the ionization region. This multiple ionization should begin gradually as the pressure increases and may account for part of the rise in K_0 . A second factor, less easy to evaluate except by the alteration on the actual gauge structure relates to the factor β_0 of Eq. 10. A certain fraction of the ions generated inside of the electron collecting grid and yet very close to the spaces between the grid wires are accelerated out by the charged glass wall and the outer grid and therefore escape detection at the ion collector. If, as the pressure is increased, a smaller fraction of these ions escape, that would correspond to a small increase in β_0 which would in turn reflect itself as a change in K_0 . That the location of this maximum on the pressure scale is sensitive to the collision cross-section is demonstrated by the fact that the maxima for argon and nitrogen occur at approximately the same pressure whereas the maximum for helium occurs at a pressure nearly 10 times greater.

The fact that the curves for the Westinghouse gauge in all cases rise with a steeper slope on the low pressure side is taken to be direct evidence related to the ion loss out of the open-ended grid structure. The extra increase in β_0 results from a reduction in the loss of ions out of the open ends as the pressure is increased. The indications seem to be that under low pressure conditions nearly 40 per cent of the ions are lost in that gauge structure.

CONCLUSIONS

These studies, preliminary though they are, indicate that for calibration purposes, ion gauges must generally be operated in the 1 to 10 microampere range of electron current and that the systematic variation in K_0 with pressure in the best calibration range, namely, 10^{-4} up to 10^{-3} mm must be understood in order that the most suitable value of K_0 may be used in the very low pressure range. Finally, the well-known importance of the gas composition is clearly illustrated in that as one achieves better and better vacua the fraction of the residual gas that is helium generally increases. Under these circumstances, the apparent vacuum might be considerably better than the true value. Under many circumstances this would not be important but with the increased interest in space technology, improved methods of vacuum determination are of very great importance.

Reference

1. W. B. Nottingham, Vacuum Symposium Transactions, 1954, p. 76.







