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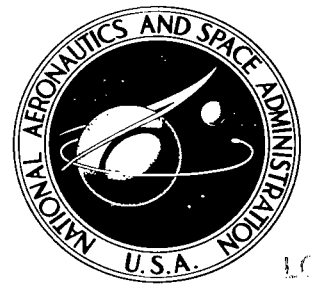

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EVALUATION OF A VOLUME-RATIO CALIBRATION SYSTEM FOR VACUUM GAGES FROM 10^{-6} TO 10^{-3} TORR

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • NOVEMBER 1965

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SUMMARY

An evaluation was made of a volume-ratio calibration system in the range from 10^{-6} to 10^{-3} torr. An error analysis showed that the pressures could be produced with a limit of error ranging from about 6 percent at 10^{-6} torr to 1 percent at 10^{-3} torr. An analysis was also made of the effects of ionization gage pumping, outgassing, ultimate pressure, and adsorption. Finally, commercial ionization gages were calibrated in the system. Large differences in calibration factors were observed between different gages along with various nonlinearity effects; however, the reproducibility of a typical ionization gage calibration curve was found to be about 3 percent.

INTRODUCTION

A system based on the volume-ratio method as a primary standard of pressure measurement was to be evaluated in the range from 10^{-6} to 10^{-3} torr. The question to be answered was whether the expansion of a gas from a small volume into a large volume can be relied upon to create accurately known pressures in this range.

The volume-ratio calibration technique is referred to as the Knudsen method, the expansion method, the method of pressure division, and the static system, and has been used successfully by various investigators (refs. 1 to 4).

Reference 1 used this method to introduce small known quantities of impurities into pure gas samples. Reference 2 calibrated a thermistor pressure gage in the range from 10^{-2} to 10 torr with a limit of error of about $\pm 1\frac{1}{2}$ percent. Reference 3 calibrated a

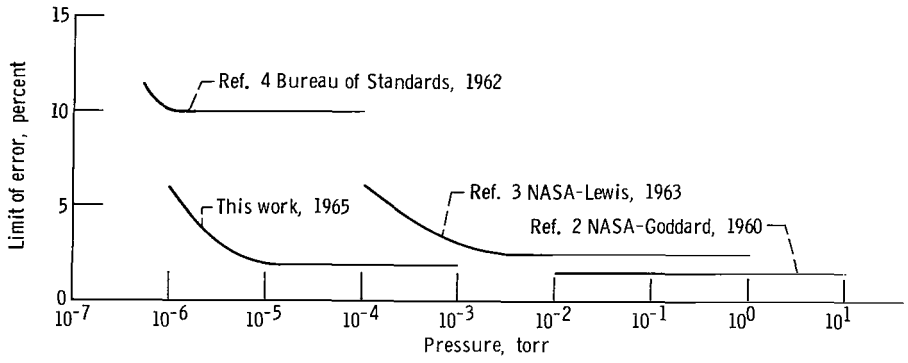


Figure 1. - Recent history of volume-ratio calibration system progress.

Pirani pressure gage from 10^{-4} to 1 torr with a limit of error of about ±3 percent. Reference 4 evaluated a system in the range from 2×10^{-7} to 10^{-4} torr and concluded that its overall limit of error was ±10 percent from 10^{-6} to 10^{-4} torr.

The vacuum systems utilized by these investigators are diverse. Reference 2 used a long, thin, 17-liter brass cylinder (information obtained by personal communication with J. Ainsworth); reference 3, a bell-shaped, 2000-liter mild-steel tank; reference 4, a glass sphere of about 10 liters; and the present work, a 1444-liter stainless-steel tank. Figure 1 summarizes the pressure range and limit of error results. A physical description of the calibration system with an analysis of its accuracy and representative samples of calibrations of commercial hot-filament ionization gages are presented herein. This report is part of a research program in vacuum measurement being conducted at the Lewis Research Center.

CALIBRATION SYSTEM

Principle of Operation

The volume-ratio method has been called the static system of pressure measurement to indicate that the test chamber is a closed system. It is differentiated from dynamic systems which are so called because of a continuous flow of gas through the test chamber, which is also continuously pumped.

In principle, the designation "static system" is accurate. An arbitrary quantity of the gas enclosed in a small volume is expanded into the test chamber. This test chamber is a closed system and the gas comes to equilibrium in its new surroundings. Its pressure p_T can be computed to be

$$p_T = (p_i - p_f)V_r \tag{1}$$

where p_i = initial small-volume tank pressure, p_f = final small-volume tank pressure, and V_r = volume ratio = $\frac{\text{small volume}}{\text{test chamber volume}}$, according to Boyle's law, assuming isothermal conditions and an initially perfect vacuum in the test chamber.

In the practical case, a closed vacuum system is far from static. Gas molecules continuously enter the closed chamber through leaks, evaporation, permeation, diffusion, and desorption. Under certain conditions, the test gas whose pressure is to be measured can be permanently adsorbed on the inner surfaces. Ionization gages in the system pump the gas as they measure its pressure. It is these dynamic effects which must be carefully considered in the evaluation of the volume-ratio calibration system.

Equation (1) can be rewritten to account for the dynamic effects on the test gas by writing the expression for test chamber pressure at a particular time t as

$$p_T = \left[(p_i - p_f)V_r - \left(\frac{dp_T}{dt}\right)_{gage} \cdot (t) - p_a\right]_{test\ gas} \tag{2}$$

where $\left(\frac{dp_T}{dt}\right)_{gage}$ = rate of change of test gas pressure due to gage pumping, and p_a = equivalent pressure reduction caused by adsorption of the test gas by the wall; and it is assumed that the test gas is introduced into the system at t = 0.

The rate of change of pressure due to gage pumping can be assumed to be constant for small changes in the pressure of the system. The effect of adsorption is not expressed as a function of time because at the pressure levels of concern in this report, adsorption can be assumed to occur instantaneously.

Finally, the equation can be written to include the dynamic effects of residual gases by expressing the total test chamber pressure at time t as

$$p_T = \left[(p_i - p_f)V_r - \left(\frac{dp_T}{dt}\right)_{gage} \cdot (t) - p_a\right]_{test\ gas} + \left[\sum p_{o,R} + \sum \left(\frac{dp_R}{dt}\right)_{outgassing} \cdot (t)\right]_{residual\ gases} \tag{3}$$

where $p_{o,R}$ = partial pressure of each residual gas in the system at time t = 0 (valve

closure); $\sum p_{o,R} = p_o$ = ultimate pressure of the system; and $\left(\frac{dp_R}{dt}\right)_{outgassing}$ = rate of change of partial pressure of each residual gas due to the summation of the effects of leaks, evaporation, permeation, diffusion, and desorption (henceforth the total of these effects will be called outgassing rate). The effect of gage pumping and adsorption of the residual gases is considered negligible because the partial pressure of residual gas in the system will always be a small percentage of the partial pressure of the test gas.

Though the outgassing is a function of the previous history of the vacuum tank, this system has been experimentally analyzed to show that the rate becomes essentially constant about 100 hours after the system has been exposed to atmospheric pressure.

The magnitudes of the individual terms in equation (3) will be discussed later.

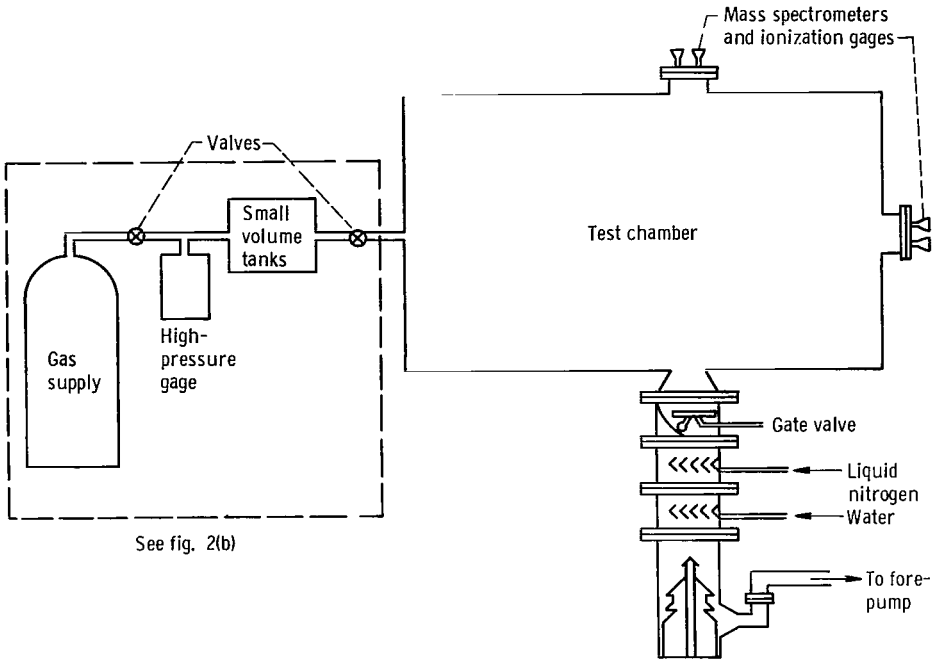
Description of System

A schematic drawing of the vacuum system is shown in figure 2. The test chamber is a 4-foot-diameter, 4-foot-long cylindrical stainless-steel tank with a volume of 1444 liters. The pumping system consists of a 10-inch fractionating diffusion pump using DC-705 pump fluid and two mechanical pumps, with separate roughing and forepump lines. The diffusion pump is rated at 4200 liters per second and the mechanical pumps at 15 liters per second each.

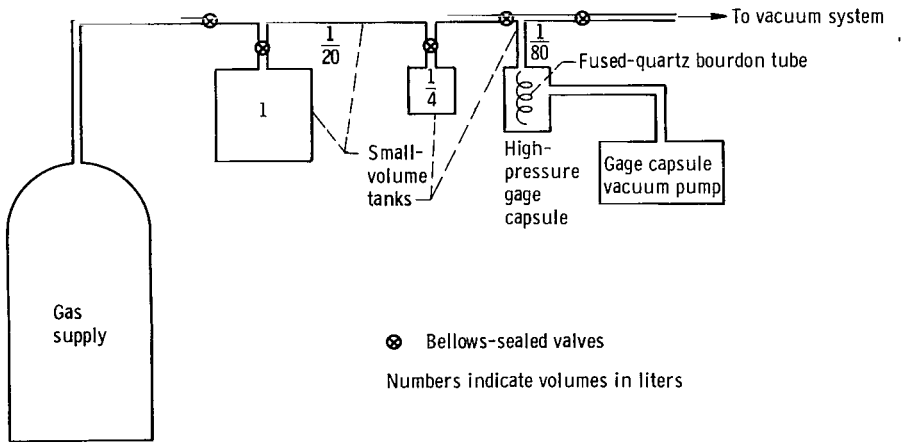
The system contains a water-cooled baffle and a liquid-nitrogen-cooled baffle mounted below a pneumatically operated 10-inch gate valve. The actual pumping speed above this valve is about 600 liters per second.

The test chamber is equipped with six 8-inch flanges sealed with gold O-rings for mounting experimental equipment. In addition, the tank contains a 31-inch-diameter flange of the same type to provide for human access to the interior of the tank. The ultimate pressure reached in the tank is 3×10^{-9} torr without bakeout. The test chamber is equipped with a Farvitron electrostatic mass spectrometer for analysis of rapidly varying processes in a qualitative sense, and a Veeco mass spectrometer (60° sector magnetic field) for high resolution of mass peaks and quantitative analysis of partial pressures. Ionization gages are attached to the chamber flanges by means of copper-gasketed flanges.

Four small-volume tanks are used with nominal volumes of 1/80, 1/20, 1/4, and 1 liter. The two smallest volumes are made from 1/4-inch-diameter stainless-steel and copper tubing, and the two largest volumes are stainless-steel cylinders. Connections are made with 1/4-inch tubing and bellows-sealed valves. A fused-quartz bourdon tube



(a) Volume-ratio calibration system.



(b) Details of small volume tanks.

Figure 2. - Schematic drawing of vacuum system.

pressure gage with a range of 0 to 800 torr is used to measure the small-volume tank pressures. The capsule surrounding the bourdon tube is evacuated to approximately 1×10^{-3} torr to provide for absolute pressure indication.

Experimental Procedure

The test chamber is initially evacuated to its ultimate pressure p_o by the diffusion pump. Ionization gage readings are constant after an appropriate time interval following gage outgassing (1/2 to 1 hr). The small-volume tank is evacuated and a quantity of the test gas is then transferred into it from the gas supply, creating an initial pressure p_i which is measured by the fused-quartz bourdon tube pressure gage. The test chamber is then sealed off from the diffusion pump by means of the 10-inch valve and a measurement of elapsed time begins at this moment. The rate of rise of pressure is observed for a sufficient time to establish the outgassing rate with adequate accuracy. An arbitrary amount of the test gas is transferred into the test chamber from the small-volume tank. A waiting period of about a minute allows the gas to come to equilibrium with its new surroundings. The final pressure in the small-volume tank, p_f, is then recorded; ionization gages in the test chamber are read and the time of the reading is noted. Tank wall and room temperatures are recorded to assure that isothermal conditions are maintained. This procedure may be repeated for an arbitrary number of gas transferences.

RESULTS AND DISCUSSION

Accuracy of Basic Components

Volumes. - The basic components of a volume-ratio calibration system are the volumes and the high-pressure gage. Four small-volume tanks were used whose nominal volumes were 1/80, 1/20, 1/4, and 1 liter. The 1 and 1/4 liter volumes were calibrated by filling with water and weighing. The 1/20 and 1/80 liter volumes were then compared to those volumes by a gas expansion method near atmospheric pressure. This method involved measuring the pressure resulting from mixing the contents of the known and unknown volumes after the smaller volume had been filled to a known pressure and the larger volume had been evacuated. The volume of the 1444-liter test chamber was similarly determined using an auxiliary calibration tank of about 150-liter volume. The volume of the test chamber obtained from 14 repetitions of this experiment was found to be 1444 liters ±0.2 percent limit of error. A volume determination based on physical measurements fell within this tolerance. The results of the volume determinations are summarized in table I.

TABLE I. - CALIBRATION SYSTEM VOLUMES

Description of volume	Nominal volume, liters	Volume, liters	Method of determination	Limit of error, percent
Small-volume tanks				
Stainless-steel cylinder	1	1.053	Weight of water	±0.2
Stainless-steel cylinder	1/4	2.536×10^{-1}	Weight of water	±0.2
Copper and stainless-steel tubing	1/20	4.95×10^{-2}	Gas expansion comparison to stainless-steel cylinders	±0.5
Stainless-steel tubing	1/80	1.30×10^{-2}	Gas expansion comparison to stainless-steel cylinders	±1.0
Test chamber	1444	1.444×10^{3}	Gas expansion comparison to auxiliary calibration tank	±0.2
Auxiliary calibration tank	150	1.567×10^{2}	Weight of water	±0.1

The temperatures of the volume tanks were recorded during these volume determinations and during the subsequent use of the volume-ratio system for the calibration of ionization gages. All of these experiments assume isothermal conditions. Temperature differences no greater than 2° F between the small and large volumes were observed during the tests. Also, the test chamber itself has variations in wall temperature from point to point no greater than 2° F. A temperature difference of 1° F between the small and large volume results in a 0.2 percent error in the volume-ratio calibration system calculation of p_T.

Pressure gage. - The pressure gage used to measure the high pressures in the small-volume tanks is a fused-quartz bourdon tube pressure gage with a 0 to 800 torr range. The gage has a resolution of 4×10^{-3} torr. The capsule surrounding the tube was evacuated to approximately 1×10^{-3} torr. Although hysteresis and drift are negligibly small, the gage has 0.5 percent of full scale nonlinearity, so that careful calibration over the entire range was required.

The gage was calibrated against: (1) an air deadweight tester in the range from 50 to 800 torr; (2) a dibutylphthalate manometer in the range from 10 to 70 torr; (3) and a thermal-conductivity vacuum gage of the type reported in reference 3 in the range from 10^{-1} to 4 torr. From calibrations (1) and (2), the probable error of the gage was determined to be less than ±0.1 percent of the reading above 10 torr. From calibrations (2) and (3), the repeatability of the gage was determined to be about $\pm4\times10^{-3}$ torr.

Figure 3 is a graph compiled from these volume and pressure gage accuracy computations (including temperature effects). The interpretation of the graph is as follows: a pressure of 1×10^{-6} torr can be created in the test chamber with a limit of error of about

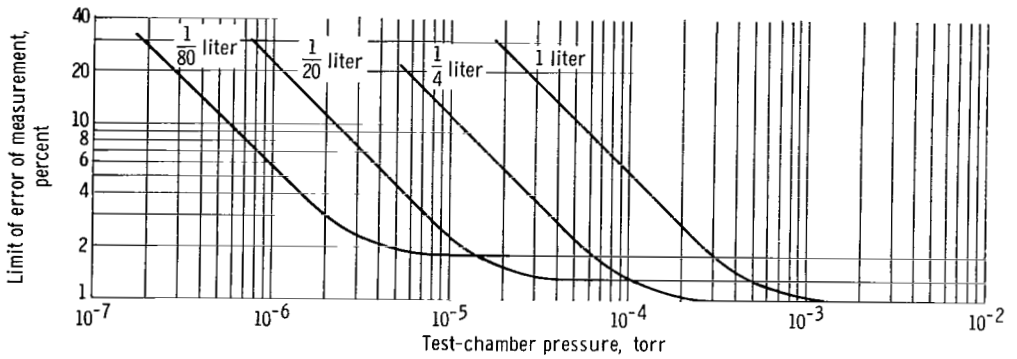


Figure 3. - Effect of accuracy of volumes and high-pressure gage on accuracy of volume-ratio system.

6 percent by using the 1/80-liter volume in conjunction with the bourdon tube pressure gage, while this same pressure created with the 1/20-liter volume would result in a 22-percent limit of error. At the upper end of the calibration range of this experiment (1×10^{-3} torr), any of the four volumes can be used to create the test chamber pressure with a limit of error no greater than about 2 percent.

Analysis of Dynamic Effects

Gage pumping. - In the operation of the volume-ratio system, a known quantity of gas is transferred into the test chamber in which one or more ionization gages are being operated. A period of time then elapses to allow the gas to come to equilibrium with its new surroundings. Experience has shown that about 1 minute is more than sufficient to achieve this equilibrium. It must, therefore, be established that the ionization gage does not pump a large percentage of this gas before this pressure is measured.

The general equation for the rate of change of pressure in a closed system due to gage pumping (in the absence of any other dynamic effects) is

$$\left(\frac{dp_T}{dt}\right)_{gage} = -\frac{S}{V}(p_T - p_o) \tag{4}$$

where S = overall gage pumping speed (electrical and chemical), V = volume of the test chamber, and the other terms have been defined in equations (1) to (3).

Since the pressures being measured are much greater than p_o, equation (4) becomes

$$\left(\frac{dp_T}{dt}\right)_{gage} = -\frac{S}{V}p_T \tag{5}$$

With $V = 1444$ liters and assuming a pumping speed of 0.1 liter per second for nitrogen gas, which is a representative value for many gages, the pumping rate becomes 0.004 p_T per minute, or less than 1/2 percent change in pressure per minute at any pressure level. Experimental measurements indicate that the rate is actually less than this value. The effects of gage pumping for hot filament gages in this experimental system are therefore negligible, since a reading was obtained in a time of approximately 1 minute.

Outgassing rate. - Gas can enter into a closed vacuum system in many ways. Among these are diffusion, permeation, evaporation, physical leaks, desorption of previously adsorbed gas from the inner walls, and so-called virtual leaks or mechanical pockets of gas within the system. The sum total of all these sources is represented by the outgassing rate.

The outgassing rate of the closed system as measured by a calibrated ionization gage was about 2×10^{-8} torr per minute equivalent nitrogen pressure. (Since the composition of this gas is not precisely known and varies with time, the ionization gage reading is expressed in terms of a gas for which the calibration is known. In this case nitrogen is used because it is the test gas used in the experiments.) This outgassing rate can also be expressed in different units as 5×10^{-12} torr-liters per second per square centimeter of surface area, or 5×10^{-7} torr-liters per second. These figures refer to the outgassing rate which results after about 50 to 100 hours following any exposure of the tank to atmospheric pressure. It has become essentially a constant after such waiting periods.

It is not implied that waiting periods of this length are absolutely necessary to successfully operate the volume-ratio system. After a 24-hour waiting period, the outgassing rate is already less than 1×10^{-7} torr per minute so that high accuracy calibrations could be performed in the 1×10^{-5} to 1×10^{-3} torr range. However, it is rare for an ionization gage calibration facility of any type to be operated less than 24 hours following the exposure of the facility to atmospheric pressure.

The actual composition of the residual gas has been observed with a mass spectrometer to be principally carbon monoxide, with smaller amounts of water vapor and hydrocarbons. Since this is an unbaked system, the chief source of gas is probably the desorption of backstreamed oil vapors from the pumping system which had previously adsorbed on the inner walls.

Since a calibration point can be obtained 1 minute after the valve to the system is closed, the contamination of the test gas due to outgassing is at most 2 percent in the calibration range of the system. Because the outgassing rate is a predictable constant, corrections to the data can be made for its effects.

Ultimate pressure. - Superimposed on the contamination of the test gas due to outgassing is the residual gas in the system at the moment the valve is closed. This corresponds to an equivalent nitrogen pressure of about 2×10^{-8} torr. Although the ultimate pressure of the system, with valve open, is about 3×10^{-9} torr, the act of valve closure

(due to the movement of the piston of the 10 in. gate valve) introduces a small quantity of gas to account for the difference. The combined effect of contamination of the test gas due to outgassing and a nonzero ultimate pressure is less than 5 percent of the total pressure at the low pressure limit of the experiment (1×10^{-6} torr), less than 1/2 percent at 1×10^{-5} torr, etc.

Adsorption Effects

Equation (2) contains a term to correct for a permanent adsorption of the test gas on the walls of the chamber. No such effects have been observed in the operation of the volume-ratio calibration system using nitrogen as the test gas.

In the 10^{-6}- to 10^{-3}-torr pressure range, the minimum formation time for adsorption of a monolayer of gas at the walls of the test chamber is from 3×10^{-1} to 3×10^{-4} seconds. Ionization gage calibration data presented in this report were obtained in times of the order of minutes. Thus, adsorption in these pressure ranges may be assumed to occur instantaneously. The result of adsorption would be a nonlinearity in the calibration curve of an ionization gage which would be greatest at the lowest pressure levels. This would result in an error in the calibration curve that would disagree in a like manner when compared to other standards. It would also be expected that large scatter in the data would result if the adsorptive capacity of the walls was not constant. Since the adsorptive capacity of the walls would be a critical function of the previous history of the system, and the calibration data was obtained over a period of about 3 months, variations in capacity would be expected. Instead, none of these effects were observed. The calibration data will be discussed in a later section of the report.

Attempts were made to observe long-term adsorption effects in the 10^{-6}- to 10^{-3}-torr pressure range on the assumption that the walls did not have a maximum adsorptive capacity. A sample of nitrogen gas introduced into the closed system was allowed to remain for times up to 24 hours. Changes in pressure during this time were entirely attributable to the outgassing rate.

Attempts were made to observe adsorption effects at pressures in the 10^{-8}-torr range where monolayer formation times are in the order of 3 to 30 seconds. Figure 4(a) represents the behavior with nitrogen, a gas which should not exhibit large adsorption effects in an unbaked vacuum system whose walls would already contain considerable adsorbed gas; figure 4(b) represents the behavior with water vapor, a gas which might exhibit multilayer adsorption effects under these same conditions.

Section 1 of the curve of figure 4(a) indicates the outgassing rate of the system immediately after the system was closed. Section 2 depicts the effect of a substantially instantaneous introduction of a small quantity of nitrogen into the system. Section 3 is the

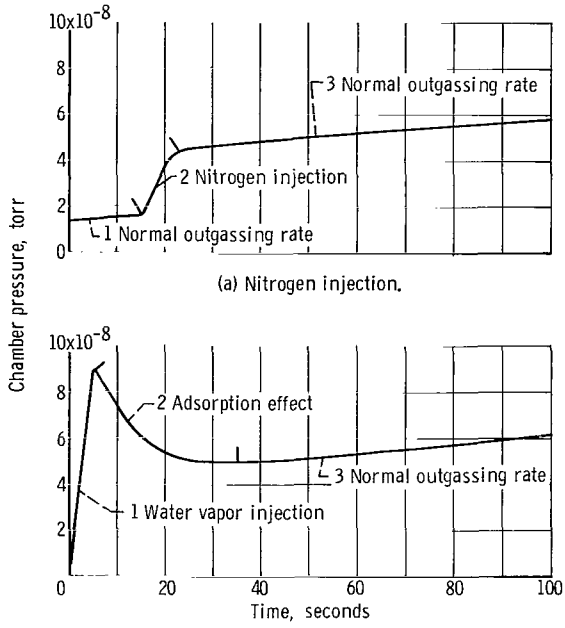


Figure 4. - Variation in chamber pressure with time for two different gas injections.

return of the system to its original outgassing rate within about 5 seconds. Simultaneous observation of the nitrogen content with the mass spectrometer confirmed that nitrogen content followed the pattern shown in figure 4(a).

Section 1 of the curve of figure 4(b) is the result of an introduction (over a 5-sec interval) of a mixture of gases containing a large amount of water vapor. Section 2 indicates the subsequent decrease in the pressure of the system. Simultaneous observation with the mass spectrometer during this period showed a corresponding pattern of decreasing water vapor content, presumably representing adsorption of water vapor by the tank walls. Section 3 represents the return of the system to its normal outgassing rate.

The following reasons are given for the phenomena observed:

(1) Because the system has not been baked, the wall condition of the tank can be characterized as saturated by at least a monolayer of gas. Under these conditions, the probability of adsorption of nitrogen is very small. Quantitatively, if any adsorption of nitrogen gas in this system is present, it is small compared to the outgassing rate.

(2) A more active gas such as water vapor will exhibit multilayer adsorption effects under these same conditions.

Comparison of the Calibration System to Other Methods

Independent comparisons were made between the volume-ratio calibration system and (a) a commercial McLeod gage and (b) commercial calibrated leaks.

a. McLeod gage. - The McLeod gage had a range of 1×10^{-7} to 4×10^{-3} torr. The comparison was made in the range from 1×10^{-5} to 1×10^{-3} torr. In this range, the McLeod gage has a readability of 2 percent or better (based on 0.25 mm reading accuracy). The McLeod gage was read with a cathetometer and also directly from the manufacturer's scale; no difference in results was noted. The comparison was made between the McLeod gage and an ionization gage that was calibrated against the volume-ratio method both before and after this experiment.

Figure 5 shows the results of the comparison. The term "pressure, volume-ratio

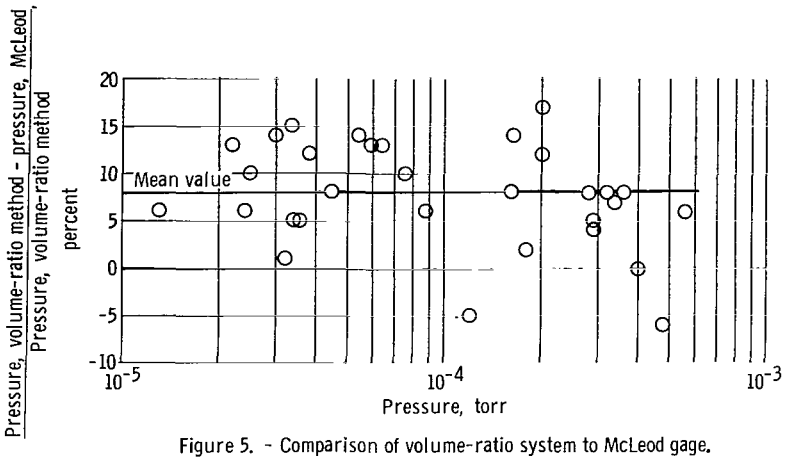


Figure 5. - Comparison of volume-ratio system to McLeod gage.

TABLE II. - COMPARISON OF VOLUME-RATIO SYSTEM TO CALIBRATED LEAKS

Leak rate, Q, torr-liters/sec (a)		Difference, $\frac{Q_m - Q_N}{Q_N}$, percent
NASA, Q_N	Manufacturer, Q_m	
2.9×10^{-5}	3.0×10^{-5}	+3
1.00×10^{-4}	1.02×10^{-4}	+2
3.46×10^{-4}	3.40×10^{-4}	-2
1.06×10^{-3}	1.02×10^{-3}	-4
3.30×10^{-3}	3.25×10^{-3}	-2

[a] System outgassing rate, 5×10^{-7} torr-liters/sec

system" refers to the use of the ionization gage as a transfer standard. The mean value of the difference between the two standards was about 8 percent, and the probable error of a single observation was about ±4 percent. Although the McLeod gage was used with reasonable care, no attempt was made to develop the art and technique required to realize the full potential of the instrument.

b. Calibrated leaks. - Two ionization gages that were previously calibrated against the volume-ratio method were compared against five commercially produced calibrated leaks. The technique used was to measure the rate of pressure rise in the closed test chamber with the ionization gages, with dry air at atmospheric pressure applied to the leak. Leak rates were then computed from the ionization gage data and compared to the manufacturer's values. The leaks ranged in size from about 3×10^{-5} to 3×10^{-3} torr-liter per second. The manufacturer calibrated the devices against a McLeod gage by the same rate of pressure-rise technique with an uncertainty of ±10 percent.

Table II compares the computed leak rates with those given by the manufacturer, and the percentage difference between them. Note that the outgassing rate of the system is less than 2 percent of the smallest calibrated leak. A correction has been applied for its effect. The Q_N term represents an average of the data obtained from the two ionization gages used in the experiment. The mean value of the difference between the two standards was no greater than 4 percent.

Calibration of Ionization Gages

To demonstrate the volume-ratio calibration system a number of hot-filament ionization gages of the type in common use were calibrated in the pressure range from 10^{-6} to 10^{-3} torr. The ionization gage tubes and their control units were commercially produced devices operated according to manufacturer's specifications with no modification of the instrumentation.

In the hot-filament gage, electrons emitted from the filament collide with molecules of the gas whose pressure is to be measured, resulting in ionization of the gas. The meter reading of the gage control unit is a measure of the ion current thus produced.

The number by which to multiply this meter reading in order to convert the meter reading to pressure is variously called the gage factor, multiplication factor, or calibration factor. The control units of the gages calibrated were constructed to read correctly for nitrogen. Therefore, a calibration factor of unity means that the gage is reading correctly, since all of these calibrations were performed in nitrogen. The calibration factor will be a constant if the following conditions are met:

(1) The rate of electron emission is constant.

(2) The average path length traveled by the electrons is constant, which implies constant potentials and a rigid gage structure.

(3) A constant fraction of the ions is collected.

(4) The gas composition remains constant.

(5) The temperature of the gage remains constant.

Figures 6 to 10 represent the calibration of five commercially produced hot-filament ionization gage tubes and their respective control units against the volume-ratio calibration system. The data consists of a total of 350 points. The probable error of a single observation from the mean value is about ±3 percent with slight variations from one gage to another. The total scatter of the data is about ±8 percent. The data were obtained over a span of three months with no significant systematic effects observed in the test gages. The average readability of the typical ionization gage meter is about 3 percent. It can be seen from the figures that the average reproducibility of the calibration factor of a typical gage is near this value.

The following general comments can be made concerning figures 6 to 10:

(1) The calibration factor of a "gage" (tube plus control unit) for a given gas can be appreciably different from that given by the manufacturer (e.g., fig. 8). This difference can be attributed to variations in the internal structure of the gage from one tube to another, or to inaccuracies of the control unit.

(2) A discontinuity in calibration factor can occur at the point of control unit range change, obviously attributable to the control unit (e.g., figs. 8 and 10).

(3) The calibration factor may vary with pressure.

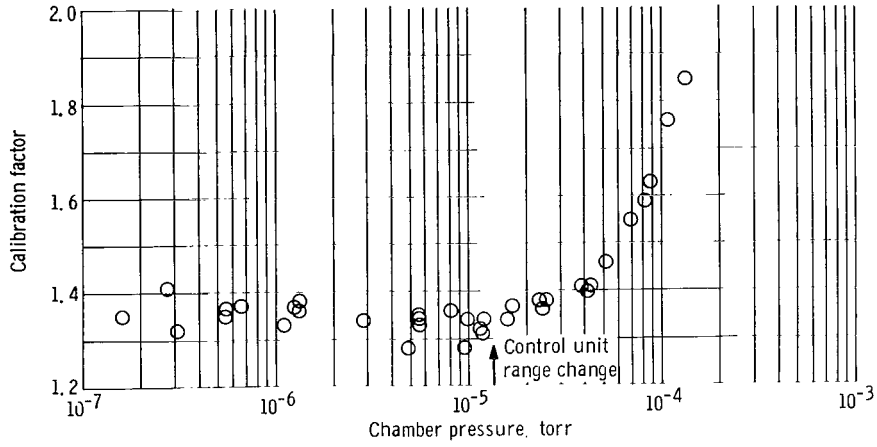


Figure 6. - Calibration of ion gage A.

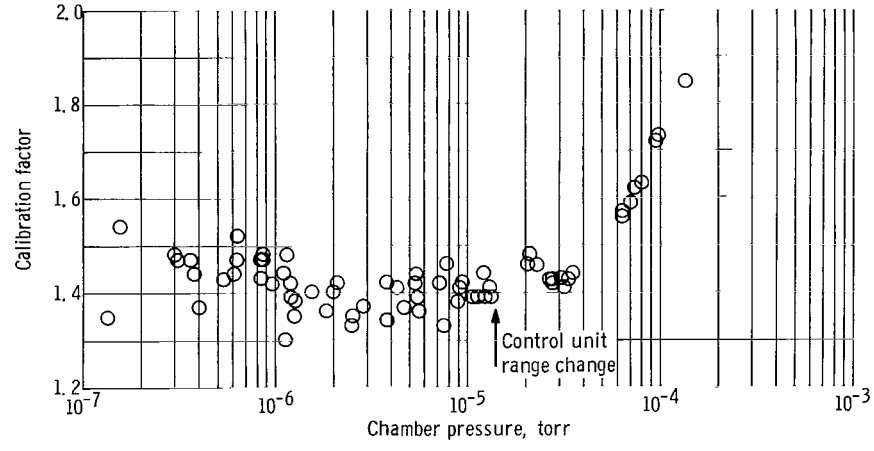


Figure 7. - Calibration of ion gage B.

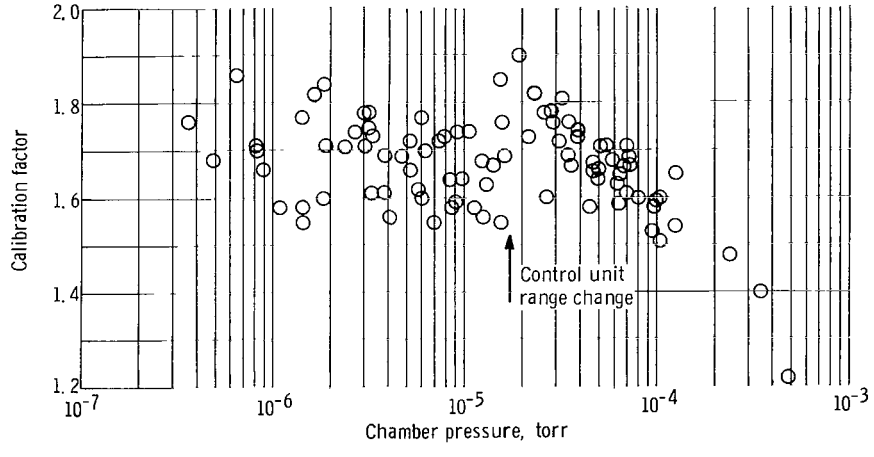


Figure 8. - Calibration of ion gage C.

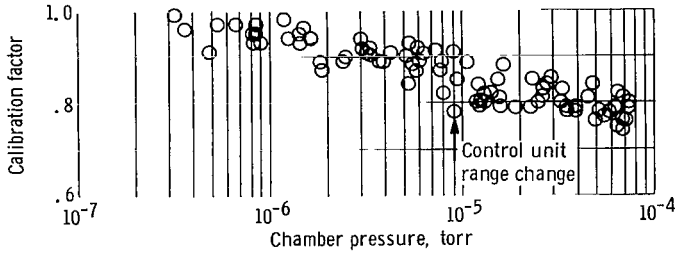


Figure 9. - Calibration of ion gage D.

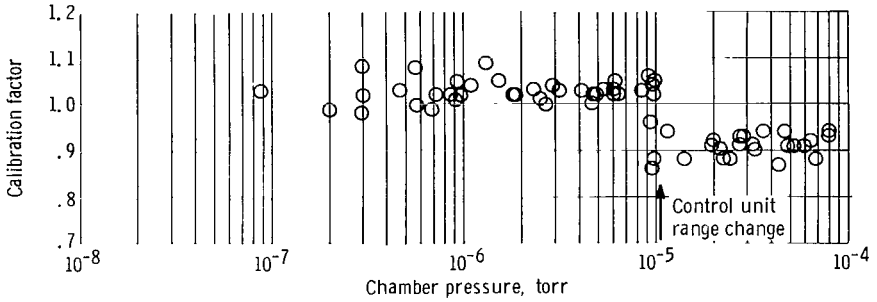


Figure 10. - Calibration of ion gage E.

(a) Gages A and B (figs. 6 and 7) were Bayard-Alpert types in which emission current was held constant over the entire pressure range. These gages showed pronounced nonlinearity above 10^{-4} torr, where the mean free path of the gas becomes comparable to gage-tube dimensions; the phenomena can be attributed to recombination or scattering of positive ions before they reach the collector (i. e., a decreasing fraction of ions is collected).

(b) Gages D and E (figs. 9 and 10) were Bayard-Alpert types in which emission was reduced tenfold in the 10^{-4} to 10^{-3} torr decade. These gages showed very little nonlinearity up to 10^{-3} torr. Both recombination and scattering of positive ions would be reduced proportionately by lower emission currents.

(c) Gage C (fig. 8), which was of conventional triode configuration, and was operated at constant emission current on all ranges, also showed pronounced nonlinearity above 10^{-4} torr. Space-charge effects may be present.

The conclusions that may be drawn from these observations are: For applications where no stringent accuracy requirements exist, the manufacturer's calibration factors may be used; for 10- or 20-percent accuracy, an average experimentally determined calibration factor for the entire pressure range of interest may be used; for accuracies better than this, the calibration factor must be determined at many pressure levels within the range of interest. References 5 and 6 have observed these same behavior patterns of commercially-produced ionization gages.

SUMMARY OF RESULTS

An evaluation has been made of a calibration system based on the volume-ratio method as a primary standard for pressure gage calibration in the range from 10^{-6} to 10^{-3} torr. The test chamber was a 1444-liter stainless-steel cylinder. An error analysis showed that the pressures could be produced with a limit of error ranging from about 6 percent at 10^{-6} torr to 1 percent at 10^{-3} torr.

An analysis was also made of the effects of ionization gage pumping, outgassing, ultimate pressure, and adsorption of the test gas. Gage pumping of nitrogen gas was shown to be negligible due to the large volume of the test chamber. The outgassing rate of the system caused no greater than a 2 percent contamination of the test gas, and this effect was correctable. The ultimate pressure of the system also caused a contamination of the test gas which was no greater than 2 percent and was also correctable. Adsorption effects of nitrogen gas were found to be negligible, at least as compared to the outgassing rate. This was probably due to the fact that the wall conditions of the unbaked test chamber can be characterized as saturated for the purposes of nitrogen adsorption. The sum total of these effects do not, therefore, alter the uncertainty figures previously mentioned. Also, a comparison of the volume-ratio method to a McLeod gage and to calibrated leaks were in agreement with these figures to the accuracies that the comparisons were performed.

To demonstrate the volume-ratio calibration system, five commercial hot-filament ionization gages were calibrated in the 10^{-6}- to 10^{-3}-torr pressure range in nitrogen gas. The results of 350 data points showed the reproducibility of the ionization gages to be about 3 percent, which is approximately equal to the readability of the gage meters. However, calibration factors appreciably different from those given by the manufacturer and various nonlinearity effects were observed.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 17, 1965.

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