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EVALUATION OF A VOLUME-RATIO SYSTEM FOR VACUUM GAGE CALIBRATION FROM 10^{-8} TO 10 TORR

by Raymond Holanda

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ABSTRACT

An evaluation was made of a volume-ratio calibration system in the range from 10^{-8} to 10 torr. An error analysis showed that the pressures could be produced with a limit of error of about $\pm 1\frac{1}{2}$ percent between 10 torr and 10^{-6} torr, and increasing from $\pm 1\frac{1}{2}$ percent at 10^{-6} torr to ±4 percent at 10^{-8} torr. An analysis was made of the effects of volume-ratio and reference-pressure-gage inaccuracies, temperature effects, ionization-gage pumping, ultimate pressure, transfer-volume-cycle gas generation, outgassing, and adsorption. Sample calibrations of vacuum gages are provided. The upper and lower pressure limitations of this system are discussed.

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SUMMARY

An evaluation was made of a volume-ratio calibration system in the range from 10^{-8} to 10 torr. An error analysis showed that the pressures could be produced with a limit of error of about $\pm 1\frac{1}{2}$ percent between 10 torr and 10^{-6} torr, and increasing from $\pm 1\frac{1}{2}$ percent at 10^{-6} torr to ±4 percent at 10^{-8} torr. An analysis was made of the effects of volume-ratio and reference-pressure-gage inaccuracies, temperature effects, ionization-gage pumping, ultimate pressure, transfer-volume-cycle gas generation, outgassing, and adsorption. Sample calibrations of vacuum gages are provided. The upper and lower pressure limitations of this system are discussed.

INTRODUCTION

An accepted technique for creating accurately known pressures in the vacuum region is to expand a known mass of gas into a previously evacuated and sealed vacuum chamber. The mass of gas is known by a measurement of its initial temperature, pressure, and enclosing volume, and a knowledge of its composition. The final pressure in the vacuum chamber can then be determined if the volume of the vacuum chamber and its temperature are known.

The method at the present time lacks a generally accepted name. Investigators have variously referred to it as Knudsen's method (refs. 1 and 2), the gas expansion method or technique (refs. 2 and 3), the incremental mass addition principle (refs. 4 and 5), Knudsen's expansion system (ref. 6), Knudsen's method of pressure division (ref. 7), the static expansion method (refs. 8 and 9), and the volume-ratio calibration system (refs. 10 and 11). Until a uniform name is adopted, it should be sufficient to know that all these names refer to the same technique.

Figure 1 shows the results obtained by various experimenters using the volume-ratio technique, and represents this author's best estimate of their error analysis; all

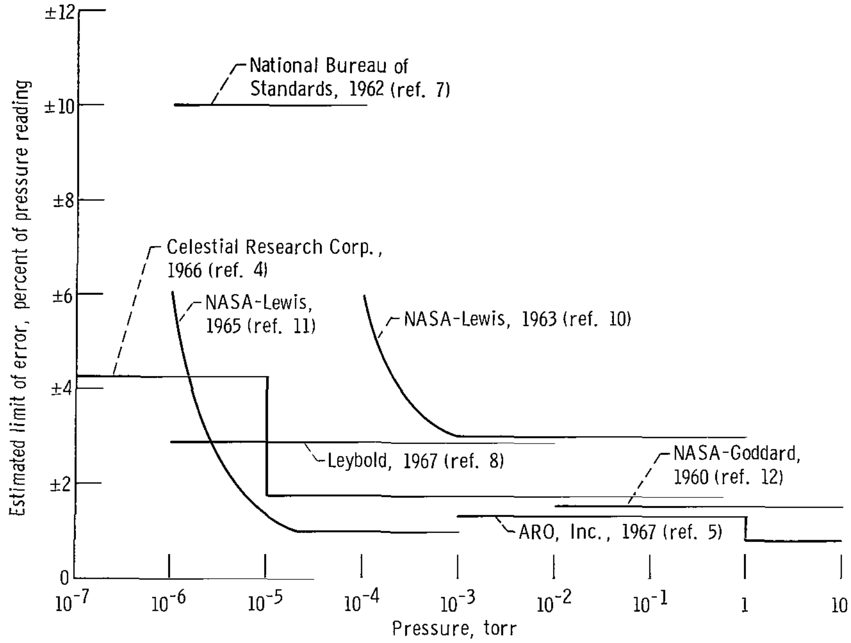


Figure 1. - Recent history of volume-ratio calibration system progress.

the investigators (refs. 1 to 12) did not present their data in a manner that could be displayed on this graph. This body of work was performed under a wide variety of experimental circumstances, with successful results obtained using many different gases, various system shapes, sizes, and materials of construction, and diverse reference gages and volume ratios.

This report represents an extension of the pressure range in both directions from this author's previous work (refs. 10 and 11). The final result is a single system that can be used to create pressures over a range of nine decades with accuracies comparable to other standards in the field. The calibration system spans the range from 10^{-8} to 10 torr, using reference pressures from 0.07 to 800 torr and volume ratios from approximately 10^{4} to 10^{7}. Nitrogen was the gas used in these experiments. A physical description of the calibration system, with an analysis of its accuracy and representative samples of gage calibrations, is presented. The effects of volume-ratio and reference-pressure-gage inaccuracies, temperature differences, ionization-gage pumping, ultimate pressure, transfer-volume-cycle gas generation, outgassing, and adsorption are discussed, along with the upper and lower pressure limitations.

The work described in this report is part of a research program in vacuum measurement being conducted at Lewis.

SYMBOLS

N	number of repetitions of test-gas transfer cycle
P	pressure
S	gage pumping speed
t	time
V	volume

Subscripts:

o	ultimate
pg	gage pumping
r	test chamber
s	source volume
si	source volume, initial
sf	source volume, final
tr	transfer volume

GENERAL PRINCIPLE OF VOLUME-RATIO CALIBRATION SYSTEM

The apparatus used for the volume-ratio calibration system is pictured schematically in figure 2. First, assume that all volumes are evacuated, that all valves are closed, and that the system is isothermal. The test chamber is at its ultimate pressure $P_{r,o}$. The source volume is filled to an initial pressure P_{si} with a test gas. The valve between the source volume and transfer volume is opened to transfer an increment of test gas into the transfer volume, and the valve is then closed. Next, the valve between the transfer volume and the test chamber is opened, transferring the increment of test gas into the test chamber, and then this valve is closed. After N repetitions of this cycle, the test-chamber pressure $P_{r,N}$ is

$$P_{r,N} = P_{r,o} \frac{P_{si} - P_{r,o}}{1 + \frac{V_{tr}}{V_s} + \frac{V_r}{V_s}} \left\{ 1 - \left[\frac{1}{\left(1 + \frac{V_{tr}}{V_r}\right) \times \left(1 + \frac{V_{tr}}{V_s}\right)} \right]^N \right\} \tag{1}$$

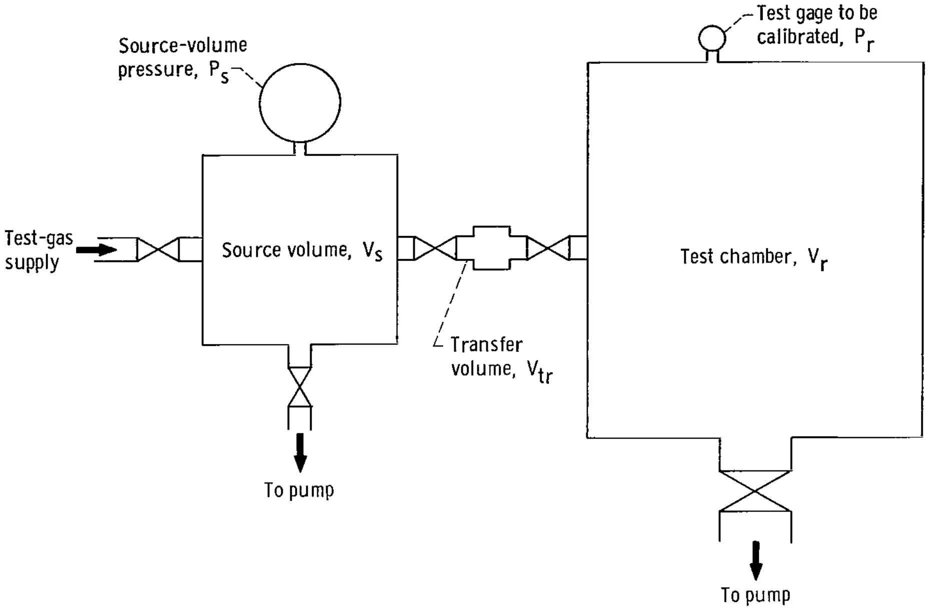


Figure 2. - Block diagram of volume-ratio calibration technique.

This equation is derived in reference 1. If $V_{tr}/V_s << 1$, $V_{tr}/V_r << 1$, and $P_{r,o}/P_{si} << 1$, equation (1) reduces to

$$P_{r,N} \cong P_{si} \frac{V_{tr}}{V_r} N + P_{r,o} \tag{2}$$

which is a useful form for approximate calculations. The fractional error of this approximation is of the order of

$$(N + 1) \frac{V_{tr}}{V_r} \left(1 + \frac{V_r}{V_s}\right)$$

A method of calculation which eliminates the need for knowing V_s and leads to an alternative to equation (1) can be presented. The final pressure in the source volume, after each increment of test gas in transferred from the source volume to the transfer volume, is $P_{sf,N}$; it is identical to $P_{tr,N}$ and to $P_{si,N+1}$. By measuring this value after each pressure increment, an exact equation can be written that is simpler than equation (1) and does not require a knowledge of V_s:

$$P_{r,N} = \frac{V_r P_{r,N-1} + V_{tr} P_{sf,N}}{V_r + V_{tr}} = \left(P_{r,N-1} + \frac{V_{tr}}{V_r} P_{sf,N}\right) \times \frac{1}{1 + \frac{V_{tr}}{V_r}} \tag{3}$$

If $V_{tr} << V_r$, a convenient form of this equation for the purposes of approximate calculation is

$$P_{r,N} \cong P_{r,N-1} + \frac{V_{tr}}{V_r} P_{sf,N} \tag{4}$$

The fractional error of this approximation is V_{tr}/V_r.

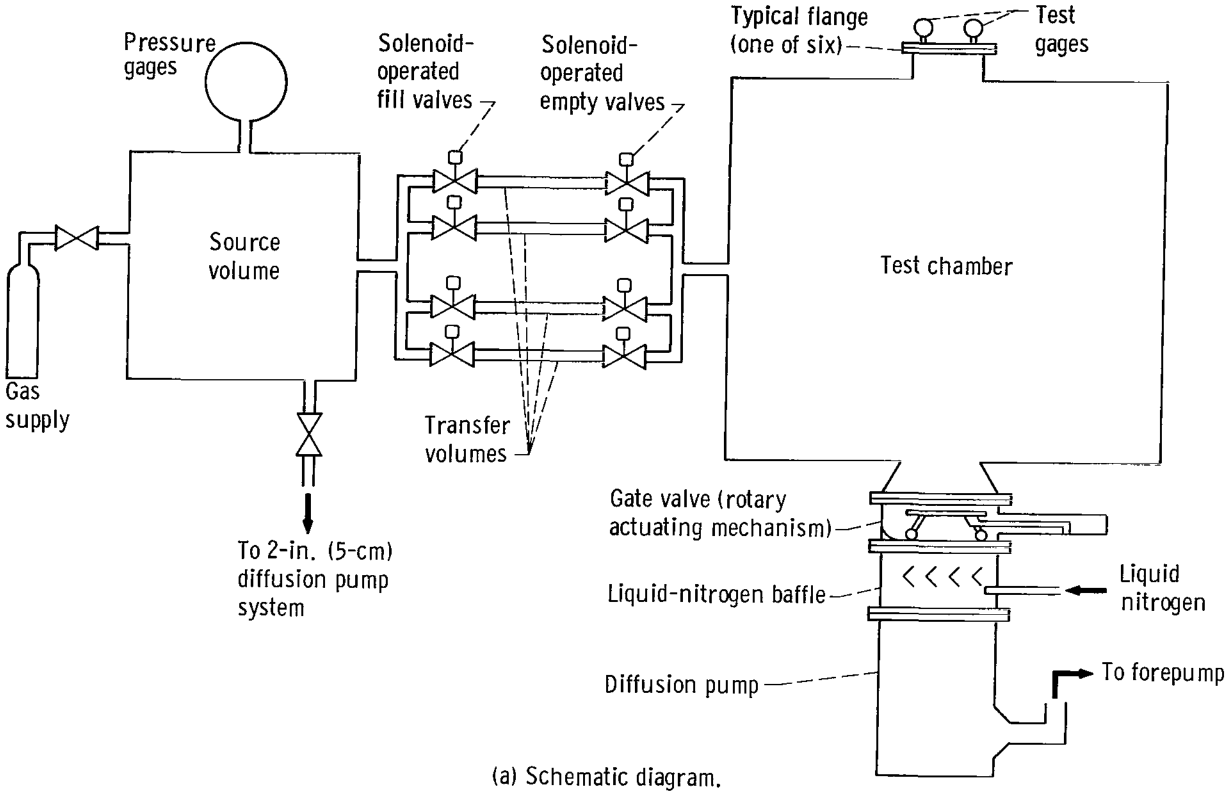
The choice between equations (1) and (3) depends on many factors, such as the ease of measurement of V_s compared with $P_{sf,N}$, the computational aids available to the experimenter, and the quantity of data to be processed. Also, if the approximation formulas leading from equations (1) and (3) are going to be used, an error analysis of each individual system would show which formula led to the best approximation. Equation (3) was used for the work performed in this report.

DESCRIPTION OF SYSTEM

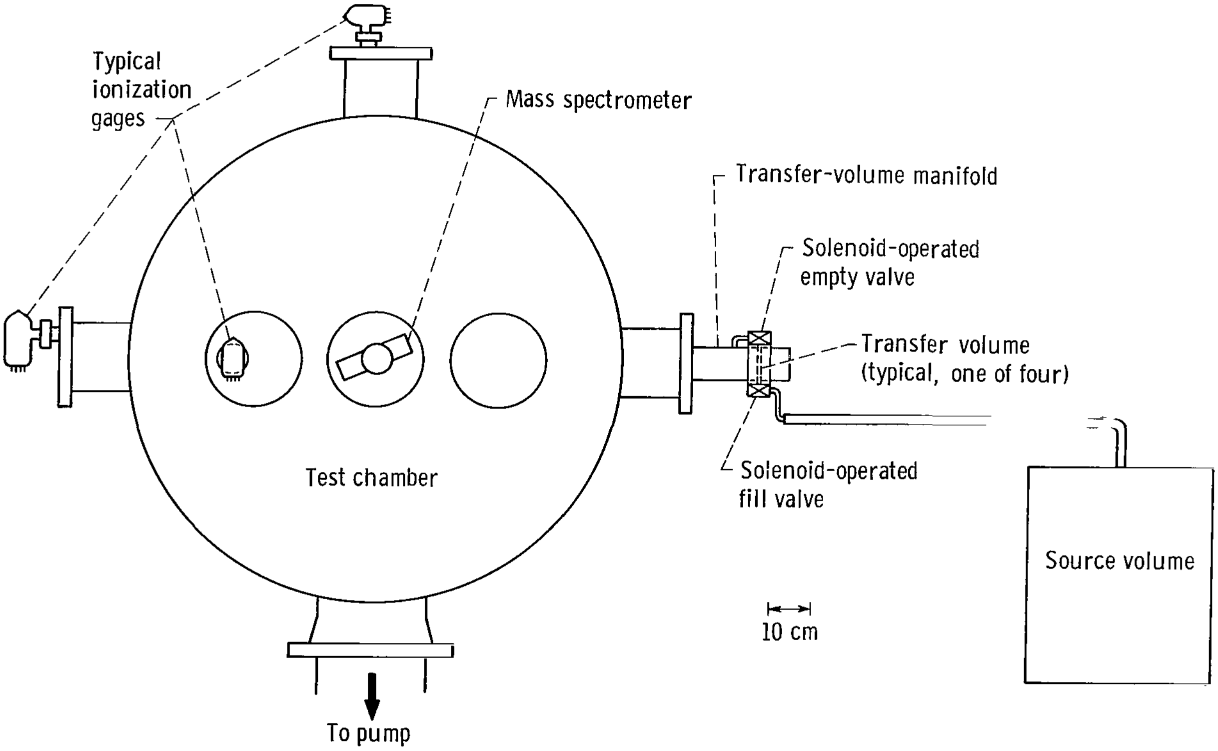
A schematic drawing of the system is shown in figure 3(a). The test chamber is a 1.2-meter-diameter, 1.2-meter-long stainless-steel tank with a volume of 1444 liters. The system is evacuated with a 10-inch (25-cm) fractionating diffusion pump rated at 4200 liters per second and using DC-705 pump fluid. The system contains a chevron-type liquid-nitrogen-cooled baffle with an antimigration barrier.

The 10-inch (25-cm) gate valve operates with a rotary actuating mechanism. The rotating shaft has a double O-ring seal which is evacuated on both sides to minimize the entrance of gas into the system during valve actuation. The valve, baffle, and pump are of stainless-steel construction with copper or aluminum gasketing on all exterior seals; the interior seal on the valve gate is made with a viton gasket.

The test chamber is equipped with six 8-inch (20-cm) flanges sealed with gold O-rings for mounting experimental equipment. One end of the tank has a 31-inch (79-cm) diameter gold O-ring sealed flange to provide for human access to the interior of the tank during initial welding and cleaning. Permanently installed on the tank is a mass spectrometer for measurement of partial pressures. Space is available to mount eight ionization gages at a time for total-pressure measurement and gage intercomparison. The test chamber is covered with a heating mantle which has 400° C bakeout capability.



(a) Schematic diagram.



(b) Scale drawing of tanks, test gages, and transfer-volume assembly.

Figure 3. - Volume-ratio calibration system.

Four stainless-steel transfer-volume tanks are used with nominal volumes of 0.2, 2, 20, and 200 milliliters. These volumes are isolated by solenoid valves to provide for automatic injection of measured quantities of gas into the test chamber. The gas is supplied to the transfer volumes from a large reservoir referred to as the source volume. Connected to the source volume are a test-gas supply, a 2-inch (5-cm) diffusion pumping system for evacuation prior to the introduction of the test gas, and the reference-pressure gages that measure the pressure of the gas being supplied to the transfer volumes.

The transfer volumes, solenoid valves, and source volume were a standard commercially available assembly. Figure 3(b) is a scale drawing that shows the size and location of the various volumes and test gages.

EXPERIMENTAL PROCEDURE

The test chamber (see fig. 3(a)) is initially evacuated to its ultimate pressure $P_{r,o}$ (about 3×10^{-10} torr) by the diffusion pump. The solenoid valve between the transfer volume and the test chamber is open so that the transfer volume is also evacuated to $P_{r,o}$. The source volume is initially evacuated by means of the 2-inch (5-cm) diffusion pumping station and then sealed off and filled with a pure test gas to the pressure level P_{si} desired for the experiment. The calibration run proceeds in the following sequence:

(1) The solenoid valve between the transfer volume and the test chamber is closed, isolating the transfer volume.

(2) The gate valve between the pump and the test chamber is closed, isolating the test chamber.

(3) The outputs of the ionization gages on the test chamber are recorded for a short period of time to determine the outgassing rate of the closed test chamber.

(4) The solenoid valve between the source volume and the transfer volume is opened, filling the transfer volume with the test gas.

(5) This solenoid valve is then closed, followed by the opening of the solenoid valve between the transfer-volume tank and the test chamber; thus, a measured quantity of test gas is transferred into the test chamber. The ionization-gage readings and the source-volume pressure $P_{sf,N}$ are recorded.

This procedure may be repeated for an arbitrary number of gas transferences.

As an alternative to this procedure, the operator has the option at any point in the calibration of switching to any of the other transfer-volume tanks in the system. These transfer volumes are a factor of 10 different in size from one to the next. By generating data points in sequence from the smallest to the largest volume, four decades of pressure steps can be created in a single calibration run with a given source-volume pressure.

ERROR ANALYSIS OF SYSTEM

There are many factors which influence the accuracy of this type of calibration system. The main factors are inaccuracy of the transfer-volume and test-chamber-volume measurements, inaccuracy of the reference-pressure gage in measurement of the gas pressure in the source volume, temperature effects, effect of ionization-gage pumping, effect of nonzero ultimate chamber pressure, gas generation during transfer-volume valve cycling, test-chamber outgassing rate, and adsorption by the test-chamber surfaces. Each of these factors is treated separately in the following sections.

Volume-Ratio Determination

The following experiment was performed to determine the transfer-volume to test-chamber-volume ratios. The volume-ratio technique was used at pressures high enough so that pressure gages of high accuracy (0.1 to 1 percent limit of error) could be used to measure the pressures in the source volume and the test chamber. Then the volume ratio was computed from Boyle's law. This experiment is identical in every way to the actual operation of the volume-ratio method as a calibration system with the exception that the volume ratio rather than the test-chamber pressure is the unknown.

A 0- to 800-torr aneroid gage, described in the next section, was used as the source-volume pressure gage, and a 0- to 3-torr capacitance-type diaphragm gage was used as the test-chamber pressure gage. This capacitance gage was used as a transfer standard to provide traceability of the volume-ratio calibration system to a deadweight tester (primary standard). First, the capacitance gage was calibrated against the deadweight tester in the pressure range from 0.3 to 3 torr. In this range the deadweight tester has a limit of error from ±0.25 to ±0.025 percent. The capacitance gage is a differential-pressure instrument; therefore, it could be used with a reference pressure of 1 atmosphere for calibration against the deadweight tester and with a reference pressure of "zero" when mounted on the test chamber, provided the instrument indication was adjusted each time to read zero when the differential pressure was zero.

The calibrated capacitance gage was then mounted on the test chamber, and the largest transfer volume was used to transfer the test gas into the system, using the technique described in steps (1) to (5) of the section EXPERIMENTAL PROCEDURE. From this experiment, the volume ratio was computed. This experiment was then repeated for the other three transfer volumes. The volume ratios and their calculated limits of error are shown in table I. The results obtained in several repetitions of these experiments fell within these error limits.

TABLE I. - CALIBRATION SYSTEM VOLUMES AND VOLUME RATIOS

	Volume ratio		Volume, liters
	Ratio of transfer volume to test chamber volume, V_{tr}/V_r	Limit of error, percent	
Transfer volume:			
1	1.44×10^{-7}	±2	2.07×10^{-4}
2	1.43×10^{-6}	±1	2.06×10^{-3}
3	1.38×10^{-5}	±1	1.99×10^{-2}
4	1.36×10^{-4}	±1	1.96×10^{-1}
Test-chamber volume	---------	--	[a]1444

[a]Limit of error, ±0.2 percent.

The accuracy of volume-ratio determination in this work is poorer than the accuracy of volume-ratio determination in 1965 (ref. 11). While the present work relied on pressure measurements and Boyle's law, the earlier work used absolute weighings of water-filled volumes. Absolute weighing could not be used in the present experiment because the volumes were very small cavities in a very massive and complex structure.

The loss in accuracy produced by the present technique, compared to the technique of reference 11, has been accepted in order to gain the following advantages:

(1) The high-pressure gage is no longer attached to the transfer volume. Greater flexibility and accuracy in choice of the gage is therefore possible.

(2) It is possible to create pressures two decades lower than those created previously.

In addition, the volume of the test chamber was determined to be 1444 liters ±0.2 percent limit of error by an experiment described in reference 11. From the volume ratios and the test-chamber volume, the transfer volumes were computed to have the values shown in table I. The volume ratios V_r/V_s and V_{tr}/V_s were not determined because equation (3) rather than equation (1) was used for the work performed in this report.

Reference-Pressure Gages

Two pressure gages are mounted on the source volume to measure the pressure of the gas being introduced into the small volumes. A 0- to 800-torr aneroid gage with a limit of error of ±0.1 percent of full scale was used to measure source pressures in the range 80 to 800 torr. This gave a limit of error of ±1 percent or less to these pressure measurements. A 0- to 10-torr capacitance gage with a limit of error of ±(0.02 percent of full scale plus 0.15 percent of reading) was used to measure source pressures in the range 0.07 to 10 torr. This gave a limit of error of less than ±1 percent for pressure measurements in the range 0.3 to 10 torr. (The figures quoted for these two gages are manufacturer's specifications verified by acceptance calibrations.) A separate calibration of the capacitance gage by the volume-ratio method for the pressure range 0.07 to 0.3 torr showed a maximum deviation of 3 percent in this range (see fig. 6).

Note that there is a gap in the pressure-measurement capability, from 10 to 80 torr. Since this gap is less than one decade and the transfer volumes span four decades, there is no corresponding gap in calibration capability. Although the aneroid gage actually covers the range 10 to 80 torr, its limit of error in this range is greater than 1 percent; and, therefore, the gage is not used as a standard in this range.

Temperature Effects

The volume-ratio calibration system theory as developed in equations (1) to (4) is based on the assumption of an isothermal system. Temperature differences between the volumes cause pressure errors to occur, the fractional value of which is equal to the fractional difference in temperature. Near room temperature, this is approximately 0.3 percent per $^\circ$C.

The manufacturer of the transfer-volume apparatus reports in his operations manual that, on the basis of the manufacturer's experiments, existence of local heating effects on the transfer volumes due to solenoid-valve operation can cause wall temperature variations of $\pm 2^\circ$ C from an average value. In addition, room-temperature variations did not cause more than $\pm 1^\circ$ C variation in wall temperatures of the source volume, transfer volume, and test-chamber volume. At the rate of ±0.3 percent pressure error per degree of temperature difference, a ±0.9 percent limit of error due to temperature effects can occur in this system.

Ionization-Gage Pumping

The effect of gage pumping as it applies to the accuracy of the calibration system is negligible in the test chamber used, because of its large volume. The general equation for the rate of change of pressure in a closed system as applied to gage pumping is

$$\frac{dP_{r,pg}}{dt} = -\frac{S}{V_r}(P_{r,N} - P_{r,o}) \tag{5}$$

where

S	gage pumping speed
V_r	volume of test chamber
P_r	test-gas pressure
$P_{r,o}$	ultimate pressure of system

The typical value for the pumping speed S for nitrogen gas of standard commercial hot-cathode ionization gages is about 0.1 liter per second. With $V_r = 1444$ liters, $dP_{r,pg}/dt = -0.004\ p_{r,N}$ per minute. This is about a 0.1 percent change in pressure, due to gage pumping, per gage during the 15-second time interval required to generate a data point. No more than three gages are run simultaneously in this test facility for intercomparison of gages; normally, gage calibrations are run individually. For vacuum systems of 50 liters or smaller, gage pumping would be a significant factor in the operation of a volume-ratio calibration system. However, references 2 and 3 have shown that the problem of gage pumping even in small chambers can be handled satisfactorily.

Ultimate Pressure

The ultimate pressure of the tank with the valve open can vary from 1×10^{-9}- to 1×10^{-10}-torr equivalent nitrogen pressure, depending on the previous history of the tank in regard to bakeout and exposure to atmospheric pressure. (Since the composition of this gas is not precisely known, the ionization-gage reading is expressed in terms of a gas for which the calibrations are known. The term "equivalent nitrogen pressure" is used because nitrogen is the test gas used in these experiments.) For the purposes of calibration, the figure of interest is the ultimate pressure of the system at the time the 10-inch (25-cm) gate valve seals the test chamber just prior to a calibration run. The gas generated by the valve-closing process degrades the ultimate pressure for calibration purposes to about 3×10^{-9} torr. This degraded ultimate pressure is measured for every test run, and this value would be $P_{r,N-1}$ in equation (5) in the absence of effects, such as outgassing, which could change the pressure with time.

Transfer-Volume-Cycle Gas Generation

The injection of a quantity of gas into the test chamber was described in steps (1) to (5) of the section EXPERIMENTAL PROCEDURE. The same problem of gas evolution arises from the opening and closing of the small solenoid valves as occurs due to the movement of the 10-inch (25-cm) gate valve. Moving parts in a vacuum system cause gas to be evolved by surface "scrubbing," and the act of creating a seal of a gasket to a surface "squeezes" gas from the gasket.

An experiment was performed to determine the amount of residual gas generated by the small-volume injection cycle. The source volume was pumped by a diffusion pump to a pressure less than 10^{-4} torr. This pressure level makes the quantity of test gas negligible compared to the residual gas evolution resulting from valve cycling. Each transfer volume was then cycled, and the pressure rise in the test chamber was measured with an ionization gage. In the worst case, the pressure rise due to transfer-volume-valve cycling for any volume was 5 percent of the smallest pressure step generated by that volume. With a maximum deviation of ±20 percent in the repeatability of the gas evolution caused by this valve cycling, a ±1 percent random uncertainty is the worst uncertainty encountered in this calibration system as a result of this effect. This uncertainty occurs at the low pressure limit of the calibration system (1×10^{-8} torr) and rapidly diminishes to a negligible effect at higher pressures. The systematic portion of this gas evolution is corrected for by subtracting it from the ionization-gage reading.

Outgassing Rate

The outgassing rate of the closed test chamber as measured by a calibrated ionization gage is approximately 4×10^{-9} torr per minute equivalent nitrogen pressure. It is the total of the combined effects of diffusion, permeation, evaporation, desorption, and leaks. This outgassing rate can also be expressed as 1×10^{-7} torr-liters per second or 1×10^{-12} torr-liters per second per square centimeter of surface. These values are typical for baked-stainless-steel vacuum systems (ref. 13) and refer to the outgassing rate which results after 50 to 100 hours of pumping following any exposure of the tank to atmospheric pressure. The rate has become essentially constant after such pumpdown periods. These numbers represent a fivefold reduction in outgassing rates over previous values reported for this system (ref. 11). This reduction was achieved by mild bakeout at 150° to 300° C and the use of improved system components (valve, baffle, and pump).

The composition of the residual gas evolved has been observed with a mass spectrometer to be primarily carbon monoxide, water vapor, and hydrocarbons. This composition is typical of diffusion-pumped systems using silicone-based pumping fluids.

Since a calibration point can be obtained in 15 seconds, the contamination of the test gas due to outgassing is at most 10 percent of the smallest pressure step of 1×10^{-8} torr. (Correction for the effect of outgassing is made by measuring the test-chamber pressure rise for a short period of time before every test run.)

Adsorption Effects

References 4 and 11 state that in the experiments performed in these vacuum systems, no adsorption of the test gas was observed. These statements apply to data obtained in the pressure range from 10^{-8} to 10^{-3} torr. (Ref. 11 refers to nitrogen gas, while ref. 4 does not state specifically to which of the various test gases this statement applies.) The results of the present experiments reiterate these conclusions with certain qualifications, discussed later in this section, extending the pressure to which this statement applies to the 10^{-9}-torr range. The reasons for this lack of adsorption are given in the following paragraphs.

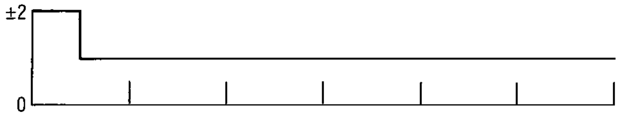
Despite the fact that the system has been baked, the wall condition of the test chamber can be characterized as saturated by at least a monolayer of residual gas. This saturation exists because monolayer formation times are still quite short (approx. 1 hr) at the ultimate pressures of this vacuum system, and cooldown times following bakeout are long (approx. 1 day). Thus, a clean-wall condition free from a single monolayer of residual gas is never achieved in this system for calibration purposes. Therefore, the sticking coefficient of a gas such as nitrogen on a surface will be that associated with surfaces of one or more monolayers of previous surface coverage. References 14 and 15 give negligible sticking coefficients for nitrogen on tungsten under these circumstances, and the results of references 4 and 11 and the present work indicate that the sticking coefficients for nitrogen gas on stainless steel are also negligible. A more active gas such as water vapor will exhibit multilayer adsorption effects under these same conditions.

Assume now that a portion of some surface within the vacuum system is made clean (free from even a single monolayer of gas) just prior to a calibration run. Then, when the test gas is introduced, the sticking coefficient for nitrogen on clean surfaces applies to this portion of the surface. Reference 16 states that values of sticking coefficient between 0.1 and 1.0 are typical for clean surfaces. Under these circumstances, the effects of adsorption on the calibration system should be observed. Just such a situation arises when an ionization gage is outgassed extensively immediately before a calibration run, and the existence of adsorption under these circumstances has been observed in these experiments. Therefore, following any extensive gage outgassing, it is necessary to wait sufficiently long for a stable monolayer to form on gage surfaces, or to create such a stable surface by means of purging with the test gas (artificial acceleration of the monolayer formation time by creating a higher system pressure).

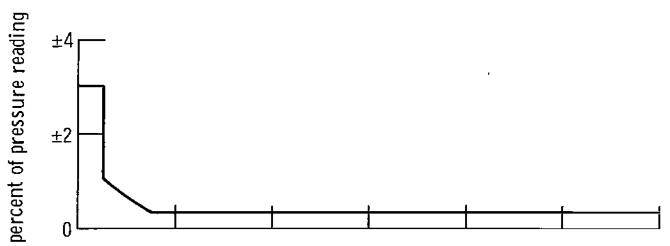
Summary of Error Analysis

The systematic errors in this calibration system are due to ionization-gage pumping, ultimate pressure, outgassing, and transfer-volume-cycle gas generation. Techniques

(a) Temperature effects.

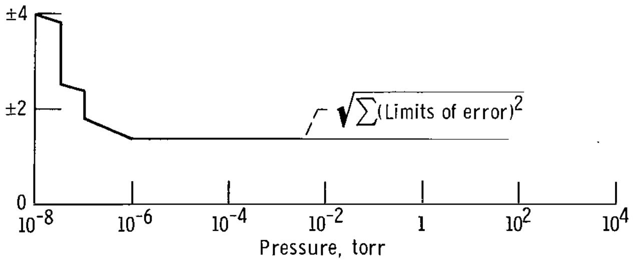


(b) Volume-ratio determination.



(c) Pressure-gage error.

(d) Residual gas effects.



(e) Total limit of error.

Figure 4. - Summary of random errors for volume-ratio calibration system.

have been shown in the text to correct for these effects where they are not negligible.

Figure 4 summarizes the random errors discussed in the preceding sections of the report. Figure 4(e) shows the square root of the sum of the squares of the limits of error of figures 4(a) to (d); this represents the overall limit of error to be expected. At any test-chamber pressure level, the combination of reference-pressure level and transfer-volume size has been chosen so that the sum of the errors from these two sources is a minimum.

The graph shows the limit of error of the system to be about $\pm 1\frac{1}{2}$ percent from 10^{-6} to 10 torr, and to increase from $\pm 1\frac{1}{2}$ to about ± 4 percent from 10^{-6} to 10^{-8} torr. The volume-ratio and temperature uncertainties predominate at the higher pressures, while errors due to residual gas effects are increasing most rapidly at the lower pressure limit of 10^{-8} torr and therefore can be considered the primary obstacle to extension of the technique to lower pressures in this particular system.

The limit of system error in the range of $10^{-5} < P < 10^{-3}$ torr is higher than the corresponding limit in this author's 1965 work (ref. 11) because of the poorer accuracy of volume-ratio determination. This deficiency is compensated by the ability to extend the lower limit of calibration capability by two decades.

RESULTS AND DISCUSSION

The pressure range of the calibration system has been stated to be from 10^{-8} to 10 torr. This range is obtained in the following manner.

Upper Limit of Pressure Range

By using the highest source-volume pressure (800 torr) and the largest transfer volume (about 0.2 liter), the largest pressure increment can be generated in the test chamber (0.11 torr). Nine repetitions of this pressure increment give a test-chamber pressure of approximately 1 torr, and 91 repetitions are required to generate the upper

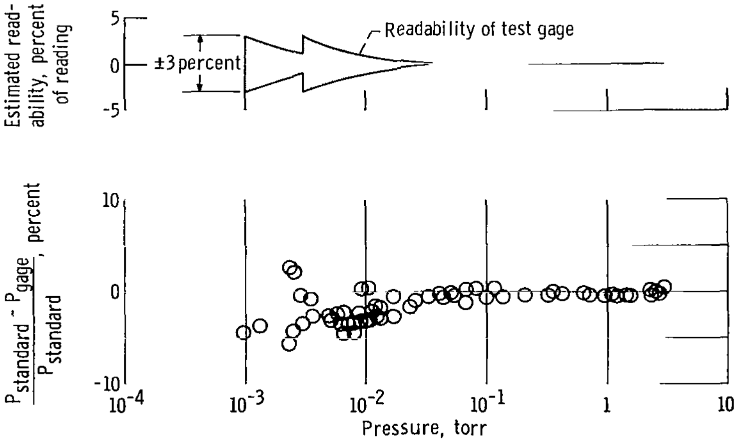


Figure 5. - Calibration curve of 0- to 3-torr differential capacitance-type diaphragm gage.

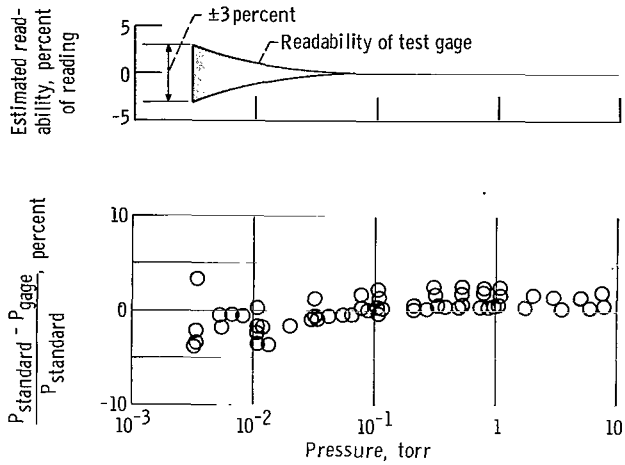


Figure 6. - Calibration curve for 0- to 10-torr differential capacitance-type diaphragm gage.

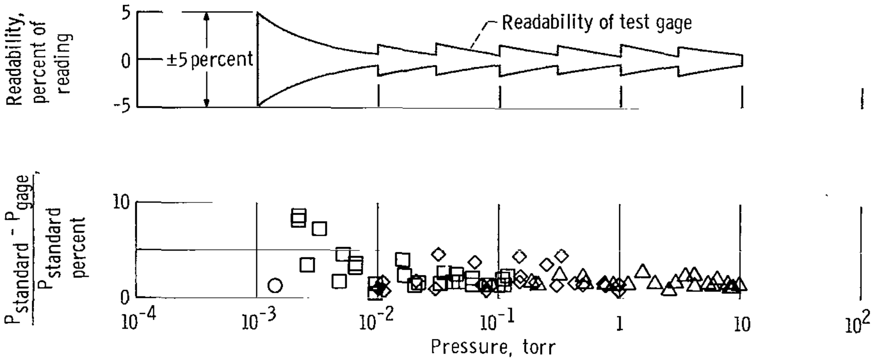


Figure 7. - Calibration of 0- to 100-torr absolute capacitance-type gage in range 10^{-3} to 10 torr. (Different symbols refer to different calibration runs.)

pressure limit of the calibration system of 10 torr. At the rate of four increments per minute, it takes 23 minutes to generate the 10-torr limit. Note that this upper pressure limit is not really a limit imposed by physical restraints. It is, rather, a choice by the operator as to the point where it becomes too time-consuming to achieve higher pressures, because the number of pressure increments required to produce a meaningful pressure increase is increasing exponentially. Also, calibration techniques of higher accuracy become available at this point.

Figures 5 to 7 demonstrate the use of the volume-ratio calibration system in the higher pressure ranges (10^{-3} to 10 torr). The calibrations are for capacitance-type diaphragm pressure gages with ranges of 0 to 3, 0 to 10, and 0 to 100 torr, respectively. The scatter in the data is within the limit of error of the calibration system at the high pressure levels of these calibration curves. At the low pressure levels, the scatter corresponds roughly to the estimated readability of the capacitance gage, as can be seen by comparing the scatter to the readability limits shown in these figures.

Lower Limit of Pressure Range

By using the smallest source-volume pressure (0.07 torr) and the smallest transfer volume (about 2×10^{-4} liter), the smallest pressure increment (1×10^{-8} torr) can be generated.

This limit is set by the cumulative effect of many error sources in the system, although the single factor that sets an absolute lower limit on the calibration system is the ultimate pressure. The following discussion relates to the problem of how close to the ultimate pressure one can approach and still have a reliable calibration system.

The sum total of the residual gas contamination in the test chamber, in the time required to generate the smallest pressure increment of 1×10^{-8} torr, is about 5×10^{-9} torr (3×10^{-9} torr is due to the ultimate pressure of the test chamber, less than 1×10^{-9} torr is due to transfer-volume-cycle gas generation, and 1×10^{-9} torr is due to the outgassing rate. See previous sections on these subjects for further details.) The question that is immediately raised by these figures is whether an ionization gage can be calibrated for a particular test-gas pressure increment when the residual gas in the test chamber amounts to 50 percent of this increment. The answer can be had by referring to figure 8, which is a graph of test-chamber pressure as indicated by the ionization gage being calibrated against time during the introduction of a test-gas sample. Outgassing, transfer-volume-valve-cycle gas generation, and test-gas sample introduction, added to the initial ultimate pressure, are shown to result in the test-chamber total pressure. By measuring only the pressure increment instead of the total pressure, the only uncertainty due to residual gas effects is caused by the transfer-volume-valve cycle. This was previously stated to be 0.5×10^{-9} torr ±20 percent, resulting in about 1 percent uncertainty in the pressure increment due to residual gas effects for the 1×10^{-8}-torr data point. It is assumed that the test gas acts independently of all residual gases as far as the ionization gage is concerned at these levels of free-molecule flow. Results based on the linearity of ionization-gage calibration curves have shown this assumption to be valid.

At test-gas pressure increments of 1×10^{-7} torr and above, the percentage of residual gases in the test gas is 5 percent or less, and the random error in their measurement quickly becomes negligible. Figure 9 shows sample calibration curves of hot-filament ionization gages. The results are similar to those previously reported (ref. 11) with total scatter of about 10 percent.

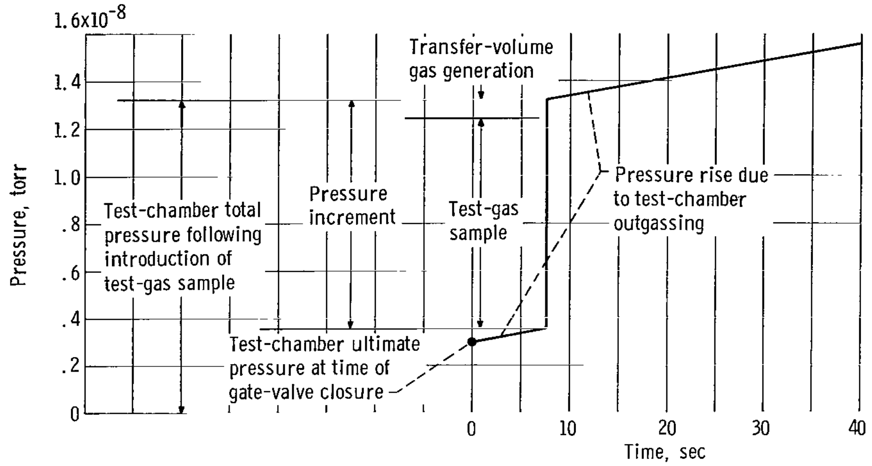


Figure 8. - Typical example of ionization-gage output while generating 1×10^{-8}-torr data point.

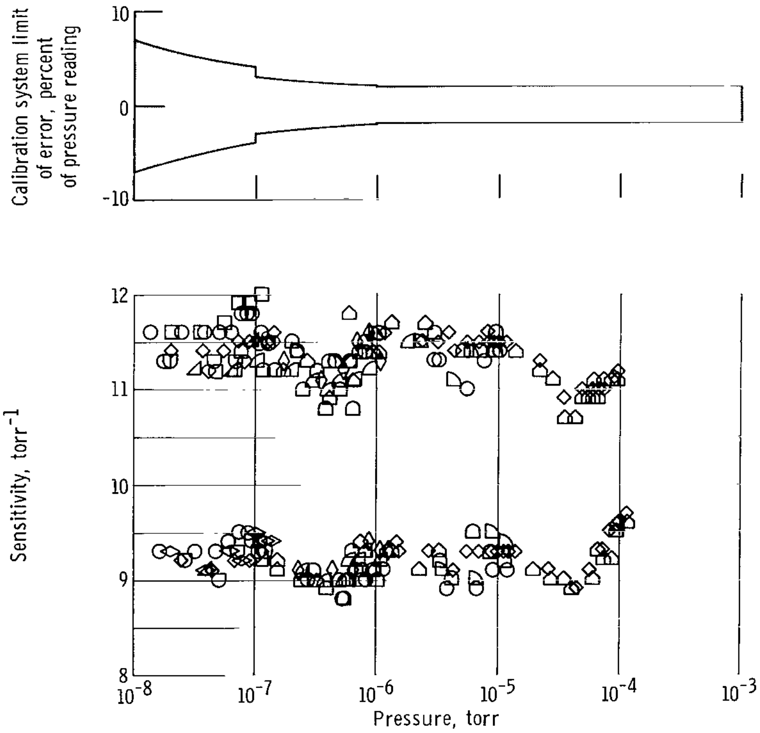


Figure 9. - Calibration curves for Bayard-Alpert-type hot-filament ionization gages (nitrogen gas). (Different symbols refer to different calibration runs.)

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 1×10^{-8} to 10 torr. An error analysis showed that the pressures could be produced with a limit of error ranging from about ±4 percent at 1×10^{-8} torr to about $\pm1\frac{1}{2}$ percent at 10^{-6} torr and above.

Traceability of the calibration system to a deadweight tester exists by means of the experiment used to determine the volume ratios and the calibration of the reference-pressure gages.

The error due to temperature differences between the volumes can be as large as ±0.9 percent because the transfer volumes contain a heat source in the form of the solenoid valves used to seal them, and because of ambient-temperature fluctuations.

Ionization-gage pumping effects are negligible because of the large volume of the test chamber.

The residual gas in the test chamber at the time the test chamber is sealed (ultimate pressure) and the outgassing rate of the system are directly measured before each calibration; therefore, correction can be made for these effects.

An experiment was performed to determine the amount of gas generated by the process of transferring the test gas into the system (transfer-volume-cycle gas generation). It was determined that this effect introduces a limit of error of ±1 percent at the low pressure limit of 1×10^{-8} torr.

Adsorption effects in this system for nitrogen gas were not observed when the walls of the test chamber were saturated with at least a monolayer of gas.

Sample calibrations of vacuum gages are provided.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 9, 1969,
124-09-19-06-22.

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