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(NASA Contract NASS-270)

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION Goddard Space Flight Center Washington, D.C.

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SUMMARY

An ultra-high vacuum type metal and glass system has been constructed for the purpose of calibrating vacuum gauges in the pressure region from 760 to 10^{-7} torr. The high vacuum portion of the system is bakeable at temperatures up to 450° C.

A mercury manometer serves as a pressure standard in the region from 760 to 20 torr, and three specially designed, bakeable McLeod gauges with overlapping pressure ranges serve as pressure standards in the region from 20 torr to 1×10^{-4} torr. Theoretical accuracies of 1 percent or better are expected for the McLeod gauge readings in this pressure region. Pressures from 1×10^{-4} torr to 10^{-7} torr region can be measured, but the accuracy of the readings depends on adsorption and outgassing effects.

Results pertaining to the reproducibility of measurements and comparisons of various gauge readings are presented. A pressure rise measurement technique is used to determine how the vacuum gauges and other system components are behaving.

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INTRODUCTION

In connection with the design and development of high vacuum gauges and mass spectrometers, it is necessary to have some means of accurately calibrating these instruments for pure gases and mixtures. A specialized system has been constructed to fulfill this requirement and, at the same time, provide a means for studying such processes as ionization gauge pumping and outgassing, adsorption, temperature effects, etc. In this way, the system is used not only to calibrate gauges, but to improve calibration procedures as well.

The problems of pressure gauge calibration have not received extensive treatment in the literature. Such a treatment requires integration of information about pressure gauges, pumping systems, gas flow, adsorption and desorption, etc. Dushman, for example, (Reference 1) treats each of the subjects mentioned above rather extensively, but says little or nothing about calibration work. The recent book on pressure measurement by Leck (Reference 2) does have a short (6 page) chapter on gauge calibration. Guthrie and Wakerling (Reference 3) also devote some space to a discussion of what the various vacuum gauges actually measure. Each of the three books just mentioned provide references to individual papers concerning vacuum gauges and vacuum measurement.

Only a very few vacuum gauge calibration systems have been discussed in the literature. A description of a demountable type vacuum gauge calibration system is given in a paper by LeBlanc (Reference 4). His system used waxed and greased connections and was not intended for use at pressures below 10^{-5} torr. Alpert (Reference 5) has developed a null-reading absolute manometer that can be used with a pressure reduction technique to measure

low pressures in a closed system. Such an arrangement has not attained wide usage.

The high vacuum gauge calibration system described in this paper is basically a glass-metal, ultra-high vacuum system, all essential vacuum components of which may be baked out at temperatures up to 450°C. This type of design was chosen to minimize the contamination of introduced gases and to facilitate the studies of adsorption, desorption, pumping, etc., previously mentioned.

Vacuum gauge calibrations from pressures of 760 torr to pressures below 10^{-5} torr can be performed with this apparatus. A mercury manometer and a set of three mercury McLeod gauges are the primary pressure standards. These units have been developed specially for this purpose.

Preliminary tests of system components and system operation have been made and some operating procedures have been established. A description is given of a pressure rise method of testing vacuum gauges and low pressure phenomena.

A great deal of work remains to be done to develop calibration system components and techniques for measuring low pressures. There is a definite need for new low pressure standards and for careful evaluation of existing standards and the methodology of their use. It is hoped that the system described herein can be used to make contributions in this area of research.

SYSTEM CONSTRUCTION

The calibration system is shown schematically in the block diagram of Figure 1. Figure 2 is an over-all view of the calibration system in operation. Figure 3 is a close-up showing some of the detailed construction. In connection with the description that follows, it will prove helpful to refer to the block diagram and the two photographs that follow.

The entire system is mounted on a three section, modular-type electronics cabinet. The major portions of the ultra-high vacuum system (which includes the McLeod gauges) are mounted within the Veeco motorized lift, model FH high temperature oven. The oven, featuring interior dimensions of 23" x 18" x 20", is an integral part of the system. Within the oven are two parallel stainless steel mounting bars, $3'' \ge \frac{1}{2}'' \ge 22\frac{1}{2}''$, which are located just above the insulated oven base and are spaced about 9 inches apart. Granville Phillips type C ultra-high vacuum (UHV) valves are fastened to these mounting bars, and a large part of the vacuum system is connected to, and supported by, the valves. The McLeod gauges are seated in plaster molds which rest on a mounting plate located between, and fastened to, the mounting bars. In this way, the components within the oven are all supported by the same rigid sturcture to minimize relative movements due to mechanical and thermal stresses. Tightening the UHV valves is one such mechanical stress. The major stresses are caused by temperature gradients that build up during the bakeout period.

The pumping portion of the vacuum system is fairly conventional. It consists of a Welch Model 1400B 21 liter/min. mechanical fore-pump and an H.S. Martin Company model M-40112 80 liter/sec., three jet, water-cooled, glass, mercury diffusion pump. The fore-pump is mounted in a tray that

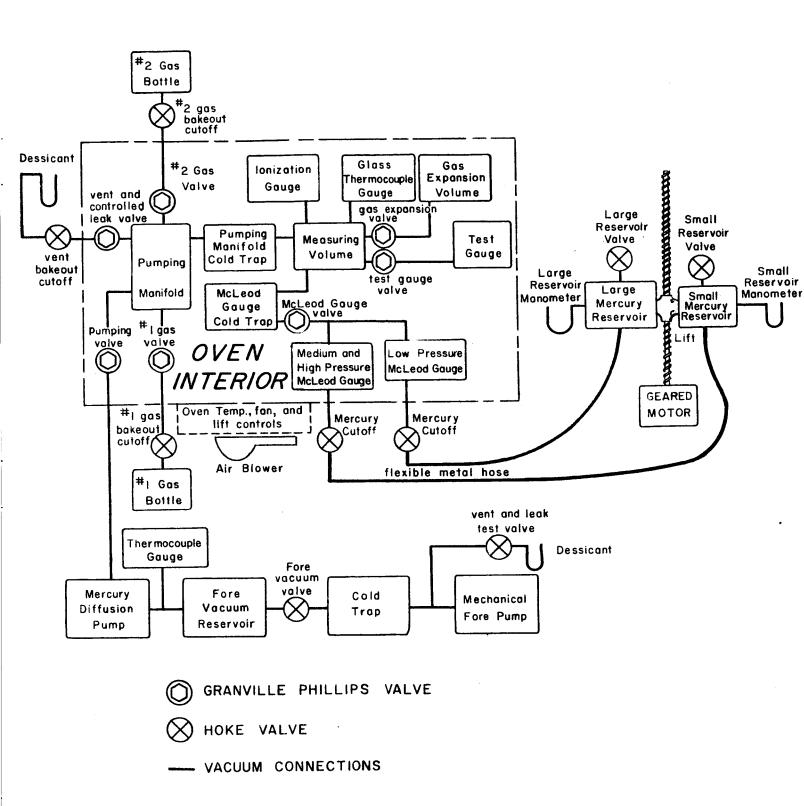


Figure 1. Block Diagram of Vacuum Gauge Calibration System

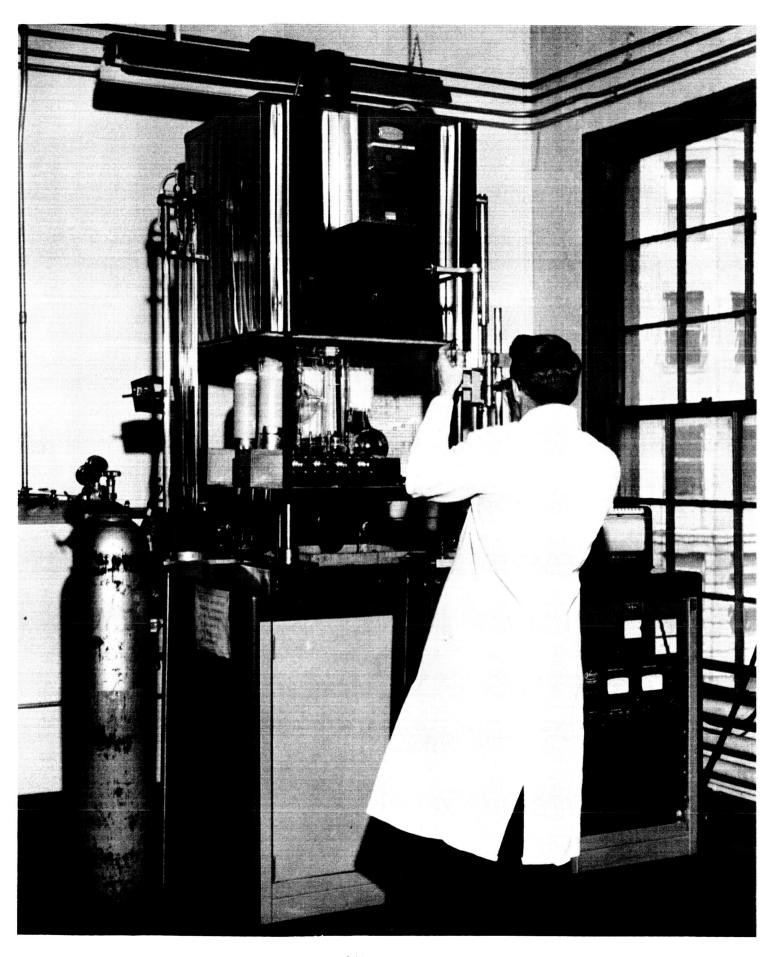


Figure 2. Overall View of the Calibration System

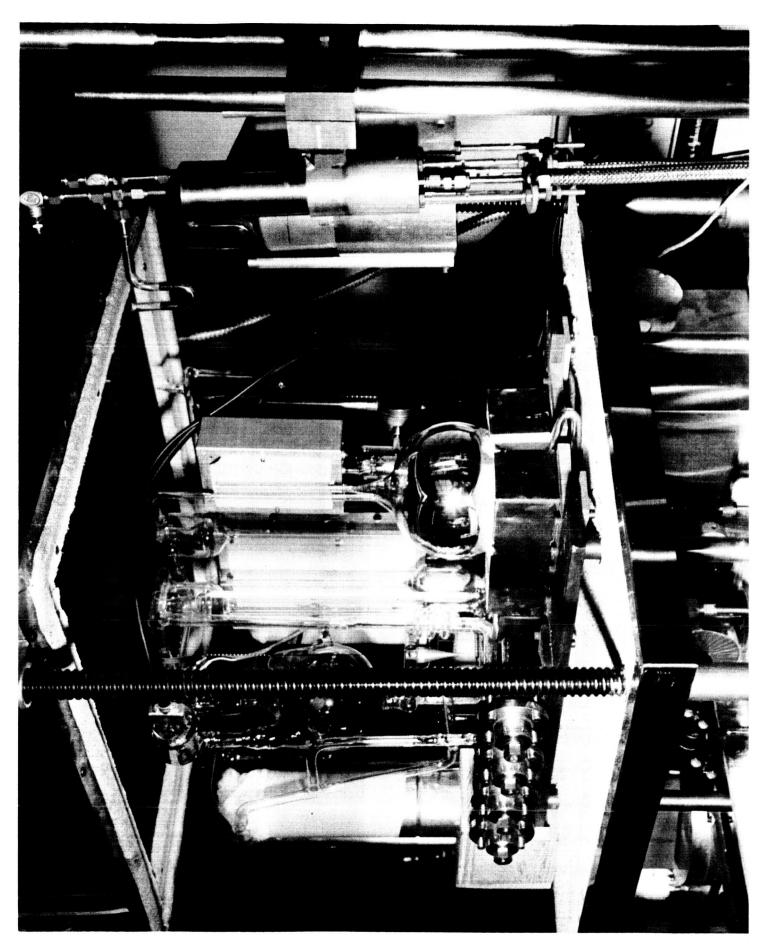


Figure 3. Close-up View of Oven Interior and McLeod Gauge Mercury Reservoirs

rests either on the cabinet caster brackets or else is supported directly from the floor by means of four corner jackscrews. When supported from the floor, the mechanical pump is effectively isolated from the cabinet, being connected to the vacuum system only by a length of rubber tubing. The vibration minimization afforded by this construction is essential for optimum performance of the mercury manometer and McLeod gauges.

A fore-vacuum cold trap (dry ice), a valve, and a large glass reservoir separate the mechanical and diffusion pumps. The reservoir makes it possible to turn off the mechanical pump completely when required.

On the high vacuum side of the diffusion pump there is the conventional liquid nitrogen cold trap. The main pumping line runs from this trap straight up through the insulated oven base and connects to the UHV pumping valve. The pumping valve connects to the manifold which, in turn, has connections to two reagent grade, 1 liter gas bottles and a large gas cylinder (tank gas in Figure 1). The latter connection can be easily broken for the purpose of venting the system. It should be noted that each of the three connections just mentioned is made through a pair of valves rather than a single valve. The three bakeout cutoff valves are Hoke model 411 diaphragm type valves. They are located at the left hand side of the calibration system below the insulated oven base.

The pumping manifold is connected to the measuring volume through the pumping manifold cold trap. This trap acts to keep condensable materials originating in the pumping, venting and gas sample portions of the system. out of the measuring volume. The measuring volume is a 1 liter, three neck spherical flask having a Veeco RG-75 ionization gauge tube attached to one neck, a Hastings Raydist model DV-17, 0 to 1000 micron pressure range, glass

envelope thermocouple gauge attached to the second neck, and the third neck available for gauges to be calibrated. The thermocouple gauge is used to give continuous qualitative indication of pressure within its range.

The McLeod gauges are mounted in plaster molds within the oven, and their pumping tubulations are connected together and lead into the measuring volume through the McLeod gauge valve and the McLeod gauge cold trap. The mercury manometer also leads into the McLeod gauge cold trap via the mercury manometer valve.

The UHV test gauge valve can be used to connect the measuring volume directly to a gauge being tested. This valve has the glass tubulation sealed when it is not connected to a gauge, so that the valve can be left open during bakeout.

A UHV gas expansion valve joins a small calibrated volume to the measuring volume. In use, the calibrated volume is filled with a gas at a known pressure. The remainder of the system (having a known measured volume) is then pumped down to a low background pressure, after which the gas in the calibrated volume is allowed to expand into the remainder of the system. By monitoring the new pressure in the system, it is possible to measure adsorption and perform calibration work.

The mercury cutoff valves for the low pressure and dual medium and high pressure McLeod gauges are located below the oven enclosure where they may be kept cool with an air blower during the bakeout period.

An Ealing model 6165 cathetometer is used to read the positions of the mercury columns in the McLeod gauges and the manometer. The cathetometer is mounted on a metal stand that is equipped with four casters for easy mobility and four jackscrews for supporting the stand in a fixed

position. The stand may be rigidly joined to the calibration system cabinet by means of an angle iron bracket. To aid in reading the McLeod gauges and manometer, illumination boxes are appropriately positioned on the system and controlled by a switch at the cathetometer. Filling and emptying of the McLeod gauges is also controlled by an electrical switch at the cathetometer.

Mercury is brought into and taken out of the McLeod gauges by raising and lowering vacuum-tight, stainless steel reservoirs that are coupled to the McLeod gauge via flexible metal hoses. Mercury can be introduced into these reservoirs through small valves located at the tops of the reservoirs. Just below these valves are located closed-end mercury manometers to indicate the state of vacuum existing above the reservoir mercury.

A motorized lift is used to raise and lower the mercury reservoirs. The lift is a modified version of a commercially available bell jar lift. It makes use of a 1/15 horsepower double reduction gear motor that turns a saginaw screw. A saginaw nut, which is firmly attached to two parallel upright steel rods, rides up and down on the screw. The two rods are constrained to move vertically by fixed-position ball bushings. A horizontal rectangular bar, which may be adjusted in position, is clamped to the steel rods near their upper end. Two heavy aluminum reservoir holders are suspended from the rectangular bar by T-slot bracket arrangements so that they may be easily removed. Brackets are also provided to suspend the reservoir holders at a fixed level below the mercury cutoff valves. This is done so that the mercury of the McLeod gauges can be lowered below the cutoff valves and thus isolated from the vacuum system during bakeout.

There are four control panels on the calibration system. One is for control of the mechanical and diffusion pumps; the second is a modified Veeco control unit for the ionization gauge (provision has been made to reduce the ionizing electron current to very small values and to measure these currents); the third is a standard five-position Hastings Raydist control unit for the thermocouple gauges; and the fourth panel controls the speed with which the mercury reservoirs are raised or lowered. These four units are in addition to the oven temperature, fan, and lift controls that are on the oven itself.

PRESSURE STANDARDS

The mercury manometer is the fundamental primary pressure standard of the calibration system. For pressures above 20 torr, the manometer is used directly as a standard. In the pressure region just below 20 torr, the manometer is used to check the pressure readings of the high pressure McLeod gauge. The high pressure McLeod gauge range overlaps that of the medium pressure McLeod gauge, so that the latter gauge readings may also be referenced to those of the manometer. Finally, the medium pressure McLeod gauge range overlaps part of the range of the low pressure McLeod gauge, so that these readings also may be referred to those of the manometer.

The mercury manometer is of the conventional U-tube variety, and is constructed of 3/4 inch I.D. precision-bore glass tubing. Each leg of the manometer is connected via a Hoke 440 bellows type vacuum valve to the mercury manometer valve within the oven. Mercury was carefully vacuum distilled into the manometer so that no trace of gas bubbles along the glass walls is evident. The manometer is kept continuously under high vacuum except when it is in use.

The manometer is read with a cathetometer which is equipped with a micrometer screw for accurately measuring distances up to 30 millimeters. The smallest division on the micrometer scale represents 5 microns, and estimates to the nearest micron may be made. For a constant pressure in the system, a series of measurements of the mercury column height difference (with the columns being hand tapped between readings) yielded standard deviations of about 10 to 20 microns. Deviations less than this are obtained if the columns are not hand tapped.

The three McLeod gauges were constructed to cover the pressure range from 20 torr down to the 10^{-7} torr region, with pressures between 10^{-4} torr and 20 torr being measured with 1 percent accuracy or better. Past experience has indicated that one could not hope to satisfy more stringent low pressure requirements at the present state-of-the-art.

The McLeod gauges were designed in such a way that they could be mounted within the oven enclosure and baked out at high temperature with the other system components. This requirement limited the gauge dimensions as indicated in Figures 4 and 5. The medium and high pressure gauges were combined in a single unit to conserve space and permit one mercury reservoir to fill both gauges simultaneously.

The high pressure McLeod gauge measures pressures from 20 torr to 2.00 x 10^{-1} torr with a calculated accuracy of 1 percent, and lower pressures with correspondingly less accuracy. The medium pressure McLeod gauge measures pressures from 4.00 x 10^{-1} torr to 4.00 x 10^{-3} torr with 1 percent accuracy, and lower pressures with less accuracy. The low pressure McLeod gauge measures pressures from 8.00 x 10^{-3} to 8.00 x 10^{-5} torr with a calculated accuracy of 1 percent, and lower pressures with less accuracy.

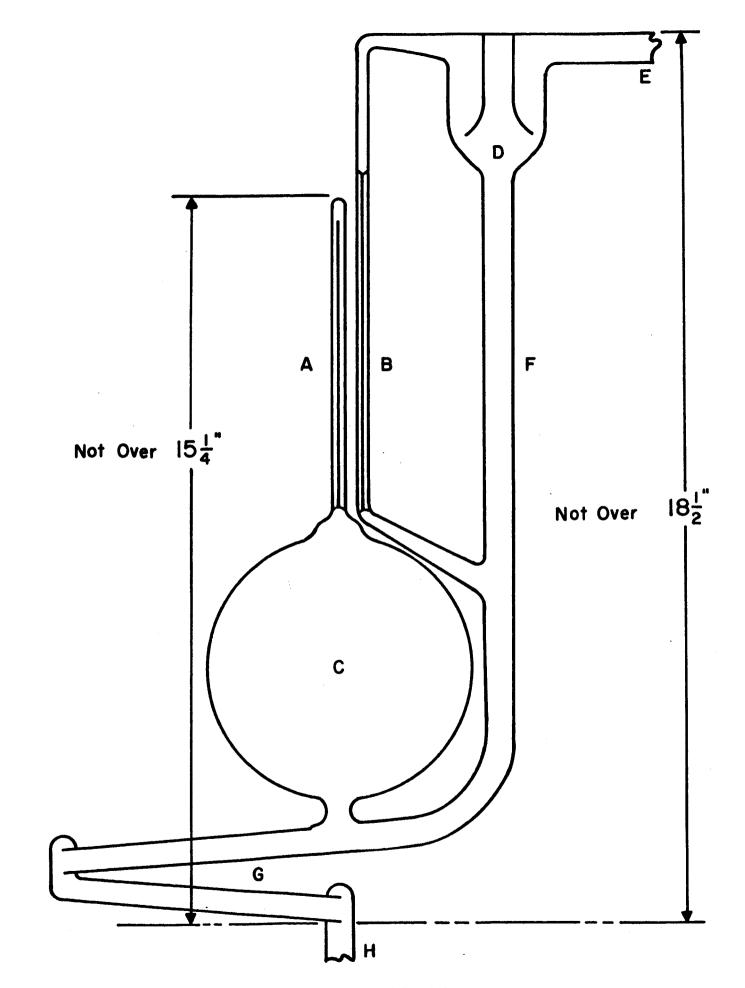
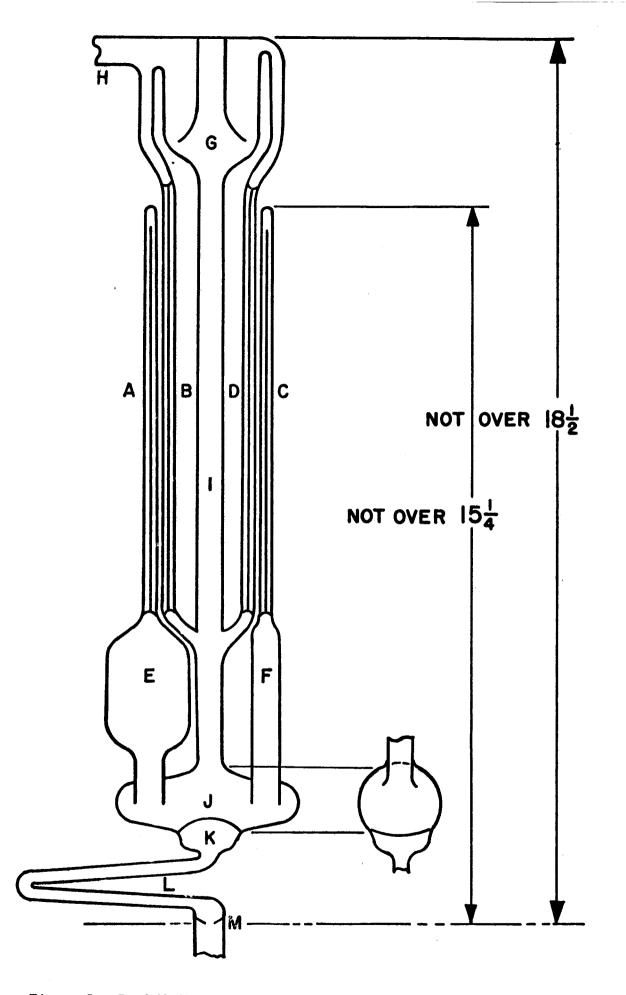
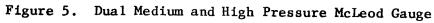


Figure 4. Low Pressure McLeod Gauge





The dimensions of the latter gauge are such, that in square law operation, 1 mm corresponds to a pressure of 2×10^{-7} torr. The deliberate overlapping of the 1 percent accuracy pressure ranges of the three gauges is a feature which enables gauge measurements to be compared, thus revealing systematic errors that depend on gauge geometry.

Mercury is vacuum distilled into the two McLeod gauge stainless steel reservoirs and is kept under vacuum continuously to minimize outgassing during operation. The mercury surface is always clean and bright, and there have never been any gas bubbles visible at the mercury glass interface.

The heights of the McLeod gauge mercury columns are normally measured to 0.05 mm with the cathetometer. Differences between the heights of the columns that do not exceed 30 mm can be measured to a few microns with the micrometer vernier adjustment of the cathetometer.

OPERATING PROCEDURES

Starting with the complete calibration system at atmospheric pressure, the system is evacuated in the following manner in order to keep it as clean as possible. First, dry ice is placed on the fore-vacuum cold trap dewar and the mechanical pump is turned on. When the forepressure reaches a value of the order of 10 microns, both the pumping valve and the fore-vacuum isolation valve are closed, the dewar is removed from the forevacuum trap, and the trap is warmed up to drive off the vapors that have condensed there. When the trap is clean, the dewar with dry ice is again placed in position, and the isolation valve is opened. The mercury diffusion pump is turned on, and the diffusion pump cold trap is half-filled

with liquid nitrogen. The pumping valve is opened, and the ov n portion of the system is pumped down. When the glass thermocouple gauge reads essentially zero, the ionization gauge is turned on so that the system pressure can be monitored continuously. When the pressure in the system is sufficiently low, bakeout may be commenced. After bakeout and cooling, the diffusion pump cold trap is filled to the top with liquid nitrogen, and the system is ready for use.

After the bakeout period, the system is usually allowed to pump down to a limiting pressure of about 2×10^{-7} torr. At this point, liquid nitrogen is introduced into the dewars that surround the pumping manifold cold trap and the McLeod gauge cold trap. The level of the liquid nitrogen in these dewars should be kept fairly constant.

With the system at a low pressure, the gas bottles can be opened, and the tubing up to the now closed Granville Phillips gas valves can be filled with gas. By closing the gas bakeout cutoff valves, small amounts of gas are isolated between the valves and can be used for calibration work.

The Granville Phillips ultra-high vacuum valves can be used to admit gas into the system from low leak rates to very high leak rates. Unfortunately, fine adjustment of the leak rate with these valves is difficult. Settings could be only roughly reproduced with the aid of a torque wrench. New Granville Phillips series 9100 variable leaks are being used to replace these gas valves.

The normal calibration procedure consists of the following steps: The system is first pumped down and baked out at a temperature of between

300 and 450°C for one hour or more. System pumping is continued until the pressure in the measuring volume, as read by the ionization gauge, is at a level that is well below the pressure at which calibrations are to be made. At this time, a continuous flow of the calibrating gas is established so that the lowest calibration pressure exists in the measuring volume.

A series of pressure readings is taken with both the appropriate pressure standard McLeod gauge and the gauge being calibrated. The gas flow is then increased step by step to obtain higher pressures in the measuring volume and the measuring procedure is repeated at each step. It is important that sufficient time be allowed for the entire system to reach its new higher pressure each time the gas flow is increased.

SYSTEM AND COMPONENT TESTS

Only a limited amount of experimental work has been carried out to date with this system. Most of the work has been concerned with testing the operation of the various system components -- especially the McLeod gauges, mercury manometer, the continuous flow of various gases through the measuring volume at different pressure levels, and the procedures for baking the system and pumping down to low background pressures.

Background pressures as low as 5×10^{-9} torr in the measuring volume, as read by the Veeco ionization gauge, have been achieved by using only a moderate bakeout at 300° C for one hour, followed by overnight ion gauge pumping. The system has also been baked overnight a few times at 450° C with no difficulty. Background pressures of 2×10^{-7} torr, as read by the ionization gauge, are routinely established in the measuring volume (with the McLeod gauges and their mercury in the system) without any bakeout.

Continuous flows of pure, reagent grade nitrogen have been established in the system corresponding to pressures in the low 10^{-7} torr region, as read by the ion gauge. Steady flows of prepurified nitrogen from the high pressure cylinder and pressure regulator have been obtained, corresponding to system pressures as high as 20 torr, as read by the manometer. The diffusion pump is turned off, of course, during these relatively high pressure measurements.

A series of experiments were performed to test the operation of the McLeod gauges. The gauges were filled at various speeds, and the optimum rate of filling, as determined from observation of the mercury motion in the capillaries, was established. Experiments were made to determine the effect of hand tapping and mechanically vibrating the capillaries at low frequencies of the order of 60 cycles per second. This matter is not settled yet, but it appears that the vibration technique, while yielding more reproducible readings, tends to raise the mercury to nonequilibrium positions, from which it may not recover in the case of small bore (1 mm dia. or less) capillaries. Since hand tapping gives rise to poor reproducibility of readings, the only acceptable method appears to be a controlled filling of the capillaries under conditions of minimum vibration. For the high pressure gauge with its 2 mm dia. bore capillary, the equilibrium position of the mercury columns is reached in a matter of seconds. For the medium and low pressure gauges with their small bore capillaries, the equilibrium position is attained after several minutes.

The initial comparison of the readings of the mercury manometer with those of the high pressure McLeod gauge was made by bleeding laboratory air into the system through a filter, desiccant, external liquid

nitrogen cold trap, and a Vactronic Vari-Vac adjustable leak. The results of these measurements are given in Table I. The increase in the error for pressures of about 6 torr and higher are believed to be due to poor illumination of the mercury menisci in the manometer, when the levels become separated by this amount. This comparison is considered only a preliminary one.

The preliminary comparison between the readings of the high and medium pressure McLeod gauges was performed with a continuous flow of prepurified nitrogen from the high pressure cylinder through an external liquid nitrogen cold trap and the Vari-Vac adjustable leak. Results of this comparison are summarized in Table II. The medium pressure gauge gave consistently lower readings than the high pressure gauge by amounts that are definitely greater than the systematic and experimental errors of the two gauges. It is possible that adsorption and desorption effects are responsible for this systematic difference in the readings.

When medium and low pressure McLeod gauge readings were taken of four different pressures of pure nitrogen in a continuous flow fashion, the same sort of results were obtained as discussed above. In this case, the low pressure gauge yielded readings that were 8 to 10 percent below those obtained with the medium pressure gauge. These readings are shown in Table III.

There are at least three possible explanations for these results. The first is that water vapor is present in the McLeod gauges and is being condensed out by the low pressure gauge. The second is that the nitrogen is being adsorbed in the gauges -- to a greater extent in the low pressure gauge. The third explanation is that outgassing from the mercury more

TABLE I

COMPARISON OF MERCURY MANOMETER AND HIGH PRESSURE MCLEOD GAUGE READINGS FOR A CONTINUOUS FLOW OF DRIED LABORATORY AIR

(Each pressure value below is an average of five readings)

AVERAGE PRESSURE VALUES		STANDARD DEVIATIONS OF AVERAGE PRESSURE VALUES		
Mercury Manometer Pressure P M	McLeod Gauge Pressure P HPG	Error (<u>P_{HPG}-P_M)</u> P _M	Mercury Manometer Deviation	McLeod Gauge Deviation
1.423 mm Hg	1.420 mm Hg	-0.21%	0.009 mm Hg	0.003 mm Hg
2.300	2.301	+0.04	0.037	0.003
3.385	3.386	+ 0. 03	0.019	0.007
5.826	5.779	-0.81	0.016	0.015
9.562	9.721	+1.67	0.016	0.038
5.561	15.756	+ 1.25	0.009	0.010

TABLE II

COMPARISON OF HIGH PRESSURE AND MEDIUM PRESSURE MCLEOD GAUGE READINGS FOR A CONTINUOUS FLOW OF PREPURIFIED NITROGEN

(Each pressure value below is an average of five readings)

AVERAGE PRESSURE VALUES			STANDARD DEVIATIONS OF AVERAGE PRESSURE VALUES	
High Pressure Gauge Reading P HPG	Medium Pressure Gauge Reading P MPG	Error (P <u>MPG⁻PHPG</u>) P _{HPG}	High Pressure Gauge Deviation	Medium Pressure Gauge Deviation
0.088 mm Hg	0.0855 mm Hg	- 2.89%	0.001 mm Hg	0.001 mm Hg
0.177	0.174	2.08	0.002	0.0 02
0.294	0.290	1.50	0.001	0.001
0.363	0.350	3.58	0.003	0.00 3

TABLE III

COMPARISON OF MEDIUM PRESSURE AND LOW PRESSURE McLEOD GAUGE READINGS FOR A CONTINUOUS FLOW OF PURE NITROGEN

Medium pressure gauge reading P Mpg	LOW PRESSURE GAUGE READING P LPG	ERROR (P _{LPG} - P _{MPG}) P _{MPG}
1.46 x 10 ⁻³ mm Hg	1.32 x 10 ⁻³ mm Hg	- 9.59%
3.51	3.21	8.55
3.76	3.45	8.25
4.15	3.81	8.20

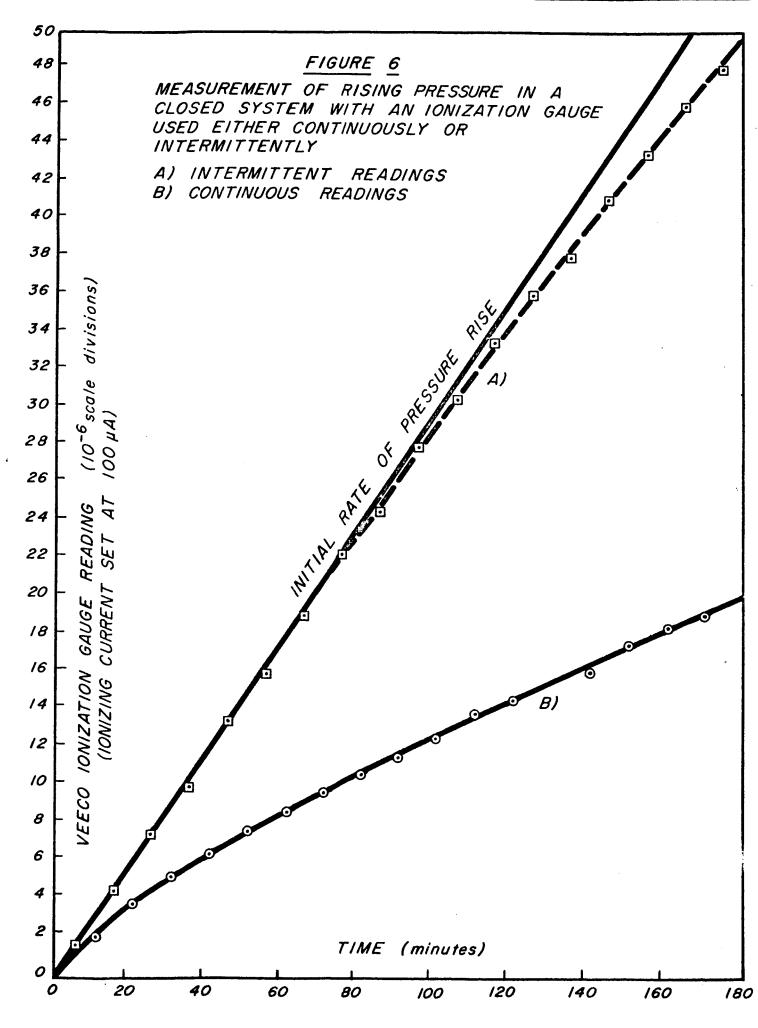
effectively increases the pressure in the smaller medium pressure gauge.

PRESSURE RISE MEASUREMENT TECHNIQUE

It may be of interest to describe briefly a technique which we are currently using for diagnostic purposes, that is, to help us determine how the gauges and other system components are behaving. In this technique the system is first baked and then pumped down to a low background pressure. With the pumping valve open, a small gas flow from a l liter gas bottle is established in the pumping manifold, and the equilibrium pressure in the measuring chamber is noted. At a convenient time, the pumping valve is closed and the pressure is allowed to rise in the system. If the leak is molecular, the pressure in the system will rise in an exponential fashion, the initial phase of which is practically linear. During this period of rising pressure, various measurements are made with the different gauges.

Measurements of the rising pressure as made with an ionization gauge are shown in Figure 6. Curve A represents the readings obtained via intermittent use of the ionization gauge. The gauge is turned on for 15 seconds every 10 minutes. The measured pressure rise is linear during the first 60 minutes. By way of contrast, curve B represents the readings obtained with the ionization gauge on continuously. Although the ionizing electron current was maintained at the low value of 100 microamperes in each case, the effect of ionization gauge pumping is strikingly evident.

When McLeod gauges are included in the closed system in which the pressure is rising, one can measure the evolution of condensables from these gauges by taking intermittent ionization gauge readings with and without a liquid nitrogen cold trap separating the McLeod and ionization gauges

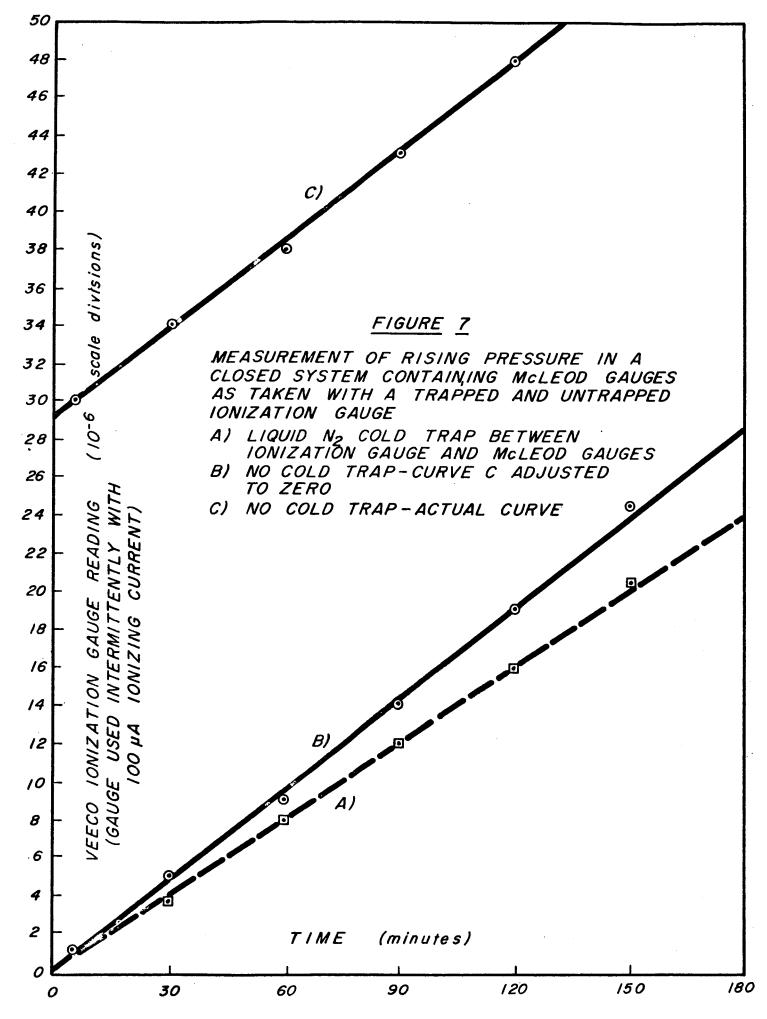


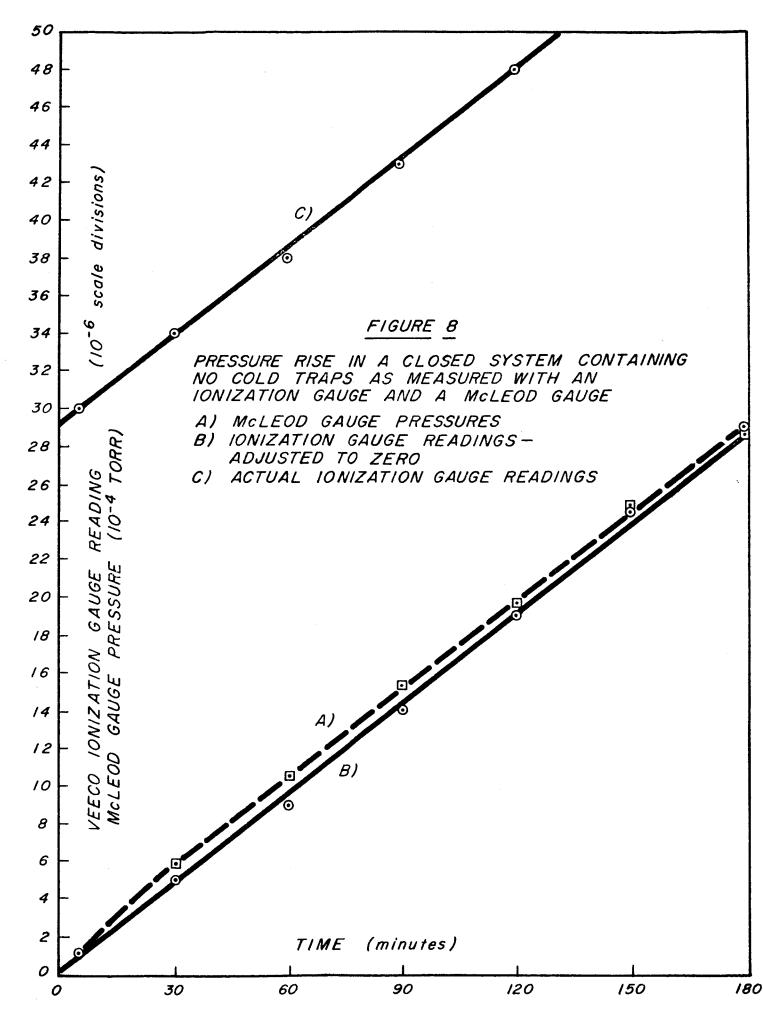
as shown in Figure 7. Without the cold trap, the ionization gauge measures the partial pressures of the mercury vapor, water vapor, and other condensables as well as the nitrogen pressure, as shown in curve C. The rate of rise of this total pressure, however, can be compared with that of the pure nitrogen rate of pressure rise.

Using the pressure rise technique, the readings of an ionization gauge can be compared with those of a McLeod gauge. Such a comparison is shown in Figure 8. No cold traps were present in the system. This particular experiment showed that the total rate of pressure rise of both condensable and noncondensable gases was measured to be the same with a McLeod gauge and an intermittently operated ionization gauge. In this case both the ionization and McLeod gauges measure the pressure of the condensables as well as the pure nitrogen. The ionization gauge also measures the mercury vapor pressure, and this must be subtracted out in order to compare the readings. When the evolution of condensable gases is small enough, a cold trap would be used to keep mercury vapor out of the ionization gauge during this comparison of gauge readings.

ACKNOWLEDGMENTS

I would like to express my thanks to Dr. R.F.K. Herzog for his many contributions to this project and to A. Suprenard and F. Robert for technical assistance in the construction and maintenance of the system. This work was performed under the sponsorship of the National Aeronautics and Space Administration.





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