GCA TECHNICAL REPORT NO. 63-8-N

74 p.

N64 11328

CODE-1

NASA CR 52877

OTS PRICE

XEROX $ 2.60 ph

MICROFILM $ 2.42 mf.

EXTENSION OF THE LOW PRESSURE LIMIT OF MCLEOD GAUGES

WALLACE S. KREISMAN

FINAL REPORT

CONTRACT NO. NAS1-2217

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

LANGLEY RESEARCH CENTER

LANGLEY FIELD

HAMPTON, VIRGINIA

MARCH 1963

GEOPHYSICS CORPORATION OF AMERICA BEDFORD, MASSACHUSETTS

(NASA CR-52877 ; GCA Technical Report ~~No.~~ t. — 63-8-N 63-8-N) OTS: on Cover

t: EXTENSION OF THE LOW PRESSURE
LIMIT OF MCLEOD GAUGES Final ...

W. Kreisman Mar. 1963 74 p refs

FINAL REPORT

(NASA Contract No. NAS1-2217)

March 1963

3599487 | GEOPHYSICS CORPORATION OF AMERICA
Bedford, Massachusetts

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Langley Research Center
Langley Field
Hampton, Virginia

TABLE OF CONTENTS

Section	Title	Page
	LIST OF ILLUSTRATIONS	ii
	ABSTRACT	iii
I	INTRODUCTION	1
II	THEORY OF MULTIPLE COMPRESSION	4
III	METHODS OF STORING GAS	8
	A. System and Equipment	8
	B. Float Value	12
	C. Mercury Freezing Technique	16
	D. Electromagnetic Mercury Valve	19
	E. Magnetic Sphere Valve	23
	F. Comparison of Storage Methods	28
IV	LOW PRESSURE MULTIPLE COMPRESSION MCLEOD GAUGE SYSTEM	31
V	EXPERIMENTAL RESULTS	38
	A. Improved Precision in Measuring Pressures within the Normal Range of the Gauge	38
	B. Improved Accuracy in Measuring Pressures at the Lower Limit of a McLeod Gauge's Range	45
	C. Measurement of a Gas Pressure below the Lower Limit of a McLeod Gauge	56
	D. Adsorption of Nitrogen Gas in the Closed End Capillary of a McLeod Gauge	60
VI	SUMMARY AND CONCLUSIONS	63
	ACKNOWLEDGEMENTS	68
	REFERENCES	69

LIST OF ILLUSTRATIONS

FIGURE	TITLE	PAGE NUMBER
1.	Photograph of the experimental McLeod gauges and vacuum system used to test various methods of gas storage.	9
2.	Close-up photograph of the experimental McLeod gauges	10
3.	Float valve arrangement for storing gas in a McLeod gauge.	13
4.	Arrangement for freezing mercury in a McLeod gauge capillary.	17
5.	Electromagnetic mercury valve installation in a multiple compression McLeod gauge.	20
6.	Magnetic sphere valve arrangement for storing gas in a McLeod gauge capillary.	25
7.	Low pressure pumping system used to evaluate the operational characteristics of a multiple compression McLeod gauge	32
8.	Close-up view of the McLeod gauge used for multiple compression measurements	35
9.	Single sample pressure values obtained with a multiple compression McLeod gauge vs. the number of compressions	54

ABSTRACT

11328

A description is given of four methods of modifying an ordinary McLeod gauge to extend its range of operation to lower pressures. The "multiple compression" technique used is that of accumulating gas samples and storing them in the closed end capillary of the gauge. Experimental results are presented to show how pressures below 1×10^{-5} mm Hg are measured with greater precision and accuracy than can normally be obtained. Accumulated samples of nitrogen gas were stored in the closed capillary of the gauge for periods of time up to 42 hours with no apparent loss or gain of molecules.

AUTHOR

I. INTRODUCTION

McLeod gauges that are used as pressure standards to calibrate a variety of other pressure gauges and mass spectrometers and also to measure the pumping speeds of vacuum pumps should provide the highest possible accuracy. It is generally accepted that accuracies of the order of 1 or 2 per cent may be obtained for pressures greater than 1×10^{-4} mm Hg, providing that precautions are taken to eliminate such systematic errors as mercury vapor stream pumping[1,2] and thermal transpiration effects[3]. One particular commercial gauge claims an accuracy of ± 1.4 per cent at a pressure of 1×10^{-4} mm Hg based on a ± 0.25 mm precision in reading the position of the mercury meniscus*. This same gauge is said to measure pressures of 1×10^{-5}, 1×10^{-6}, and 1×10^{-7} mm Hg with accuracies of ± 4.4 per cent, ± 14 per cent, and ± 44 per cent, respectively. It has been the experience of the writer, and of others, that McLeod gauge accuracies quoted for pressures below 1×10^{-4} mm Hg are to be regarded with suspicion - especially when these pressures are near the lower measurement limit of the gauge.

It is generally not very well appreciated that the accuracy of McLeod gauge measurements depends on errors that can arise from two separate sources. The first source includes imperfections in the gauge itself, capillary effects, reading errors, etc. The second source consists of the presence of a non-representative sample of gas in the gauge. The latter category includes the effects of mercury vapor stream pumping, thermal transpiration, outgassing or adsorption in the gauge or

---

* Model GM-110 McLeod Gauge made by Consolidated Vacuum Corp., Rochester, New York.

mercury, and, finally, pressure fluctuations or non-equilibrium conditions in the system whose pressure is being measured.

In very general terms, one can say that the precision and accuracy of McLeod gauge readings vary directly with the pressure being measured. Low precisions are obtained at low gas pressures where the random variations of capillary depression, reading errors, etc., are large relative to the short length of the unfilled closed capillary and the small difference in height of the two capillary menisci. By effectively increasing the pressure that the gauge measures, one increases both the precision and accuracy of the readings.

The most obvious way of increasing accuracy is to construct a larger gauge. This is not impossible, but it becomes very costly to construct large reservoirs and fill them with hundreds of pounds of mercury. Also, as the reservoir and compression bulb sizes increase, the vacuum system required and the outgassing problems become greater.

It is possible to increase the precision and accuracy of McLeod gauges without changing their dimensions. The method of doing this is to use multiple compressions[4]. In the multiple compression technique, the McLeod gauge is equipped with a valve of some sort that will allow gas to be isolated and stored in the closed-end capillary. After a sample of the gas to be measured is admitted to the compression bulb and is compressed by raising the mercury until it is forced into the closed end capillary, a small valve located at the base of the capillary

is closed. At this point, the mercury is lowered below the cut-off level of the compression bulb and a second sample of gas is allowed to enter the compression bulb. The second sample is compressed by raising the mercury and the capillary valve is opened to permit the second sample to be combined with the first sample. Both samples of gas are then stored in the closed capillary and the cycle is repeated to obtain a third sample, etc. A measurement of the gas pressure may be made after any number of samples have been taken.

Multiple compression provides two major benefits in making McLeod gauge measurements. The first of these is obviously the transformation of a low pressure gas sample to a much higher pressure sample that can be measured with greater precision. The second major benefit is that one takes a series of samples of the gas being measured over an extended period of time. Because of this, one obtains a more representative sampling of the gas being measured. The effects of pressure fluctuations, for example, will be minimized.

In the sections that follow, one will find a discussion of the very simple theory that underlies multiple compression, a discussion of various ways of storing gas in the McLeod gauge, descriptions of different multiple compression gauges that have been built, descriptions of experiments that have been performed with multiple compression gauges, and, finally, a summary and discussion of various conclusions that have been reached.

II. THEORY OF MULTIPLE COMPRESSION

The McLeod gauge is essentially a combination pressure amplifier and liquid manometer. A known amount of gas (that contained in the compression bulb with or without the added volume of the closed end capillary) at an unknown pressure to be determined is compressed to a new, smaller, known volume such that its new, higher pressure is large enough to be measured by the liquid manometer portion of the instrument. The operation of the gauge is based on the principle of Boyle's Law, viz. the product of pressure and volume of a perfect gas remains constant at constant temperature. Thus, if one can rule out the presence of such disturbing phenomena such as condensation of the gas, deviations from ideal behavior (Van der Waal's gas, etc.), dissociation, association, chemical reaction, sorption and desorption processes, then the McLeod gauge has the capability of yielding accurate results.

For a continuous flow system, the usual derivation of the McLeod gauge equation is obtained by equating the product of the pressure and volume of the gas in the large compression bulb prior to compression with the product of the pressure and volume of gas in the closed end capillary after compression. If one includes corrections for the capillary distortion and varying mercury depression, the equation may be written as shown below:

$$PV = (P + h_d + \Delta h)(\sigma h_c' + V_D) \qquad (1)$$

where the symbols above have the following meaning:

P = sample gas pressure being measured (mm Hg)

V = volume of the compression bulb and closed capillary (cm^3)

h_d = vertical difference in height between the mercury menisci in the open and closed capillaries (mm)

Δh = the capillary depression difference between the open and closed capillaries (mm)

σ = cross sectional area of the closed end capillary expressed in units of cm^3/mm length

h'_c = the length of the unfilled portion of the closed capillary including a meniscus shape correction (mm)

V_D = distortion volume at the top of the closed end capillary (cm^3).

When one is measuring pressures of 10^{-2} mm Hg and below, the pressure P, expressed in mm Hg, is small compared with the difference in height h_d of the capillary mercury columns and can be safely neglected in the right hand member of equation (1). The volume correction V_D for the irregular shape of the top of the closed capillary may be determined by weight and length measurements made in the same fashion as the measurement of the cross section parameter σ. For carefully fabricated capillaries the volume V_D may be equivalent to the volume contained in about 0.1 mm of capillary length. Thus the correction V_D can be included in determining the "effective" length h_c of the unfilled closed capillary. The difference in capillary depression of the two columns of mercury is usually determined as a function of capillary position at a pressure below the low pressure limit of the gauge. This

procedure, however, will not yield accurate values of Δh because it has been shown that capillary depression in small bore tubes varies in an unknown way in the presence of a gas phase, such as that existing above the mercury in the closed end capillary[5]. Because of the variation of capillary depression with gas pressure above the mercury, the technique of eliminating the comparison capillary tube and using a large bore tube together with capillary depression measurements to determine the final pressure of the compressed gas can provide only a limited improvement[6]. In a McLeod gauge, the difference in capillary depression of the two capillaries Δh depends in part on such variables as the cleanliness of the glass surface, the presence of surface irregularities, the manner in which the mercury is raised to its final position, and the presence of mechanical vibration. About all that can be done at present is to include the term Δh in the theoretical equation and make some estimate of its value as required.

From the foregoing discussion, it is apparent that equation (1) may be simplified to the equation

$$P = \frac{\sigma}{V} h_c(h_d + \Delta h) = K_1 h_c(h_d + \Delta h) \qquad (2)$$

when pressures below 10^{-2} mm Hg are being measured in a continuous flow system. The term h_c is the <u>effective</u> length of the unfilled portion of the closed end capillary, corrected for the changes in volume caused by the curved surface of the mercury and the distortion of the capillary bore.

When a multiple compression McLeod gauge is used to gather many samples of the gas being measured, the effective volume V in equations (1) and (2) is increased. Equation (1) becomes:

$$P\,[V + (n-1)\,V'] = (P + h_d + \Delta h)(\sigma h_c' + V_D) \qquad (3)$$

where the symbols have the same meaning as before, with the understanding that V represents the volume of the compression bulb, closed capillary, and any volume associated with the gas storage value. The term V' represents the volume of the compression bulb below the gas storage value in cm^3. Similarly, for multiple compression, equation (2) becomes

$$P = \frac{\sigma h_c (h_d + \Delta h)}{V + (n - 1)\,V'} = K_n h_c (h_d + \Delta h) \qquad (4)$$

where K_n is the gauge constant for n compressions. For some methods of storing gas in the closed capillary, the volumes V and V' are identical within less than 0.1 per cent, and the gauge constant simplifies to

$$K_n = \sigma / nV' \quad . \qquad (5)$$

III. METHODS OF STORING GAS

A. SYSTEM AND EQUIPMENT

The tranformation of multiple compression McLeod gauge theory into practice required that certain techniques and devices be developed to permit storage of the gas being measured after each compression cycle. In order to develop and test different methods of gas storage, it was decided to build a separate vacuum system which would accommodate various McLeod gauges. A photograph of this system is shown in Figure 1. A close-up view of the McLeod gauges used is presented in Figure 2.

The diffusion pump that was used with this system was a Leybold model Hg 3 mercury diffusion pump having a nominal pumping speed of 3 liters/sec and a rated ultimate pressure better than 10^{-6} torr. This pump was backed up with a Welch model 1402B 140 liter/min vented-exhaust type mechanical pump. A dry ice cold trap was placed between the mechanical pump and the diffusion pump to minimize the flow of oil vapor from the mechanical pump into the rest of the system.

The McLeod gauges to be tested were connected to a two foot length of glass tubing having a diameter of about 4 cm. This tubing served as the high vacuum manifold. It was sealed off at one end and connected through a 20 mm bore high vacuum stopcock to the diffusion pump through two cold traps in series. As can be seen from the photograph, an ionization gauge (Veeco Model RG-75P) and a glass envelope

Figure 1. Photograph of the experimental McLeod Gauges and vacuum system used to test various methods of gas storage.

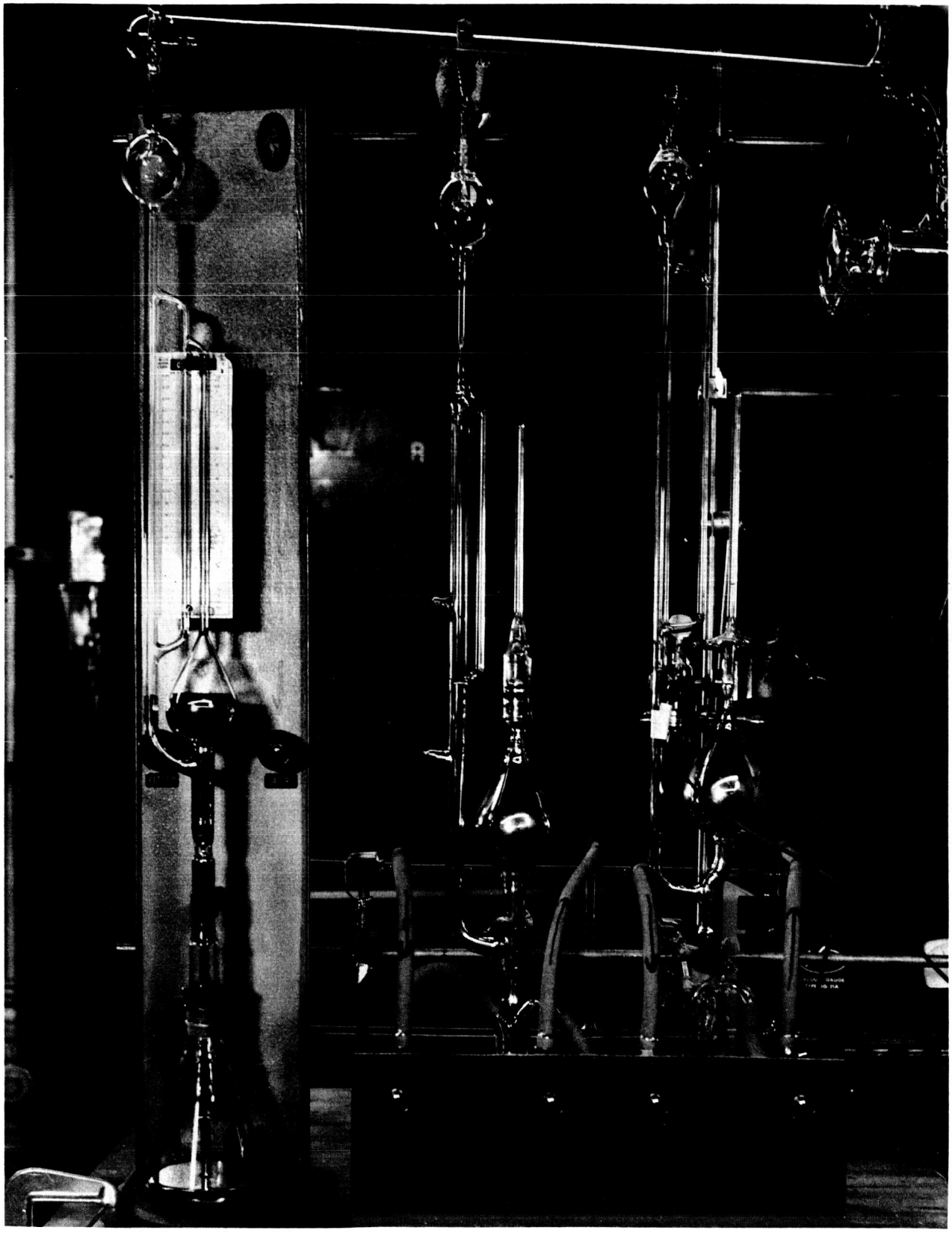

Figure 2. Close-up photograph of the experimental McLeod Gauges.

thermocouple gauge (Hastings Raydist model DV-17) were connected to the glass tubing that joined the two cold traps. This arrangement permitted the pressure in the system to be monitored continuously.

The McLeod gauge that was used to develop the magnetic sphere mercury valve arrangement was a Greiner Scientific Company model G10701B gauge, having a listed pressure range of .005 to 0.2 mm Hg. This gauge is seen on the left in the photograph. Notice that it uses metal needle valves to control the flow of air into and out of the mercury reservoir. The use of metal needle valves to control the movement of mercury in the gauge by controlling the pressure of air in the reservoir is a marked improvement over the use of ordinary stopcocks for this purpose. All of the McLeod gauges which have been used to date have been equipped with Hoke model DR297 brass needle valves to control the mercury movement.

The two McLeod gauges that were modified to test the feasibility of using a float valve and an electromagnetic valve for storing gas samples were Scientific Glass Apparatus Company model JV-2970 gauges. These particular gauges were selected because of their relatively simple construction and low cost. Four of the needle values mentioned above were mounted together on a panel as shown in the photographs and were used to control the mercury movement of these gauges.

A small separate mechanical pump (Welch model 1400B) with a free air capacity of 21 liters/min was used to furnish the vacuum required

to lower the mercury in the McLeod gauge. Atmospheric air that was used to raise the mercury was drawn from the laboratory through a glass wool filter and an indicating desiccant (Drierite, sold by Fisher Scientific Company). The high vacuum manifold and experimental McLeod gauges could be vented to atmospheric air via a stopcock, a fritted glass filter, and indicating desiccant. To control the pressure in the system, atmospheric air was leaked into the high vacuum side of the diffusion pump by means of a Hoke model 1197 metal valve.

B. FLOAT VALVE

The first device that was developed and used for the purpose of storing gas in a McLeod gauge was the float valve. A simple form of such a valve has been installed in the center gauge shown in the photographs of Figures 1 and 2. This valve is shown in detail in Figure 3. The close-fitting portion of the valve consists of a ground ball and socket that have been lapped together. The socket, which connects to the upper part of the compression bulb, is made of pyrex. The ball portion may be made of glass or metal. The float is given sufficient weight to seal properly by filling it with mercury. An iron core can be included within the float so that it can be moved with the aid of an external magnet. Of course, the mercury and iron must be sealed vacuum tight within the float. It should be noted that it is not actually the closeness of the ball and socket mating surfaces that form the gas storage seal, but rather the small amount of liquid mercury that is

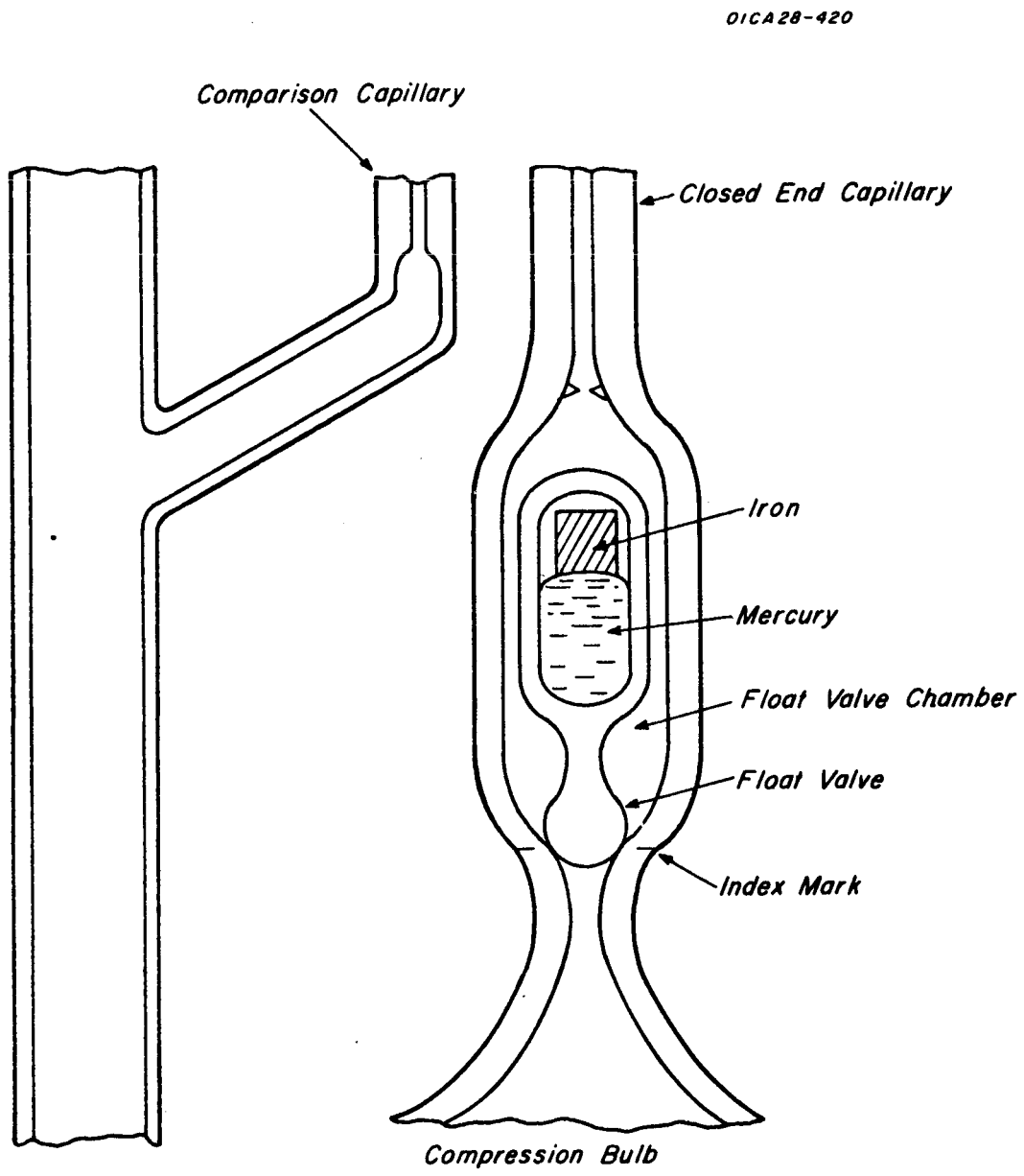

Figure Float valve arrangement for storing gas in a McLeod Gauge.

trapped above the mating surfaces of the ball and socket joint. The amount of mercury so trapped depends on the weight and displacement of the float. The heavier the float valve and the smaller its displacement (volume submerged in the mercury), the more mercury will be trapped above the ball and socket joint. The higher the column of mercury isolated in the valve, the greater the amount of gas that can be stored above the valve.

The float valve that was constructed for experimental purposes was made entirely of pyrex glass. The valve was made by modifying a commercially available float valve (Eck and Krebs type 4013 float valve).

The modification consisted of removing the top half of both the float chamber and the float. The upper part of the float chamber contained a lead-in wire and an inner guide or bearing surface for the 0.5 inch diameter float tubing. The upper part of the chamber was replaced with a piece of plain, cylindrical pyrex tubing approximately 1 inch in diameter. The upper part of the float, which had been about 0.5 inch in diameter, was replaced with a plain cylindrical pyrex tubing about 0.75 inch in diameter so that it could be heavily weighted with mercury. A short length of 0.5 inch magnetic Kovar tubing was also placed within the float tubing before its top end was sealed vacuum tight. The overall length of the float was about 2.5 inches while the overall length of the float chamber was about 3.0 inches.

In operation, it was found that a column of mercury approximately 20 mm high was trapped above the ground, mating surfaces of the valve. This column height could be increased or decreased by placing more or less mercury within the float. It was noticed that when the mercury was raised in the experimental gauge that contained the valve described above, the float portion of the valve would not rise until the mercury level in the gauge tubulation (leading to the vacuum system) was well above the position of the valve seat. Hence, when the float finally did move upwards from its closed position, it moved rather rapidly until it was stopped by the upper part of the float chamber. This sudden opening action constituted a mechanical shock to the entire gauge. In addition, the sudden opening was usually accompanied by a turbulence and jetting of mercury within the float chamber. If great care was taken in raising the mercury level, the turbulence and shock was minimized. If the mercury level is lowered rapidly, the float will strike the bottom of the float chamber with considerable momentum, again imparting a mechanical shock to the entire gauge. The large mechanical shock is primarily a result of the great weight of the float.

The float valve does effectively isolate the compressed gas that is being measured and stores it within the float chamber and the closed end capillary. However, in the process of combining gas samples, the presence of turbulence and jetting of the mercury during the valve opening makes it possible to isolate and trap small quantities of gas between the mercury and the glass walls of the float and float chamber.

The gas so trapped will be lost from the gas samples, and the pressure readings of the gauge will be erroneously low.

From the foregoing discussion, it is clear that a float valve method of storing gas in a McLeod gauge has a number of disadvantages. One of the disadvantages that has not been mentioned thus far is the need to build a special gauge which contains the float valve. In addition, the gauge must be calibrated volumetrically with the valve in position. For this purpose, it is almost essential that there be magnetic material present in the float valve so that the float may be moved externally. The principal advantage of a properly adjusted float valve is that is is automatic in operation, that is, it is self-opening and self-closing with the rise and fall of the mercury.

C. MERCURY FREEZING TECHNIQUE

A second rather obvious method of storing gas in the closed end capillary of a McLeod gauge is to freeze a small quantity of the mercury which is used to force the compressed gas into the capillary. The freezing point of mercury is -38.9^{o}C so that this temperature can be reached quite conveniently with dry ice.

As shown in Figure 4, the experimental arrangement for the frozen mercury storage valve is very simple. An ordinary McLeod gauge of suitable geometry can be fitted with a small cardboard or plastic container located near the base of the closed capillary. A few turns of nichrome wire enclosed in electrically insulating tubing are placed

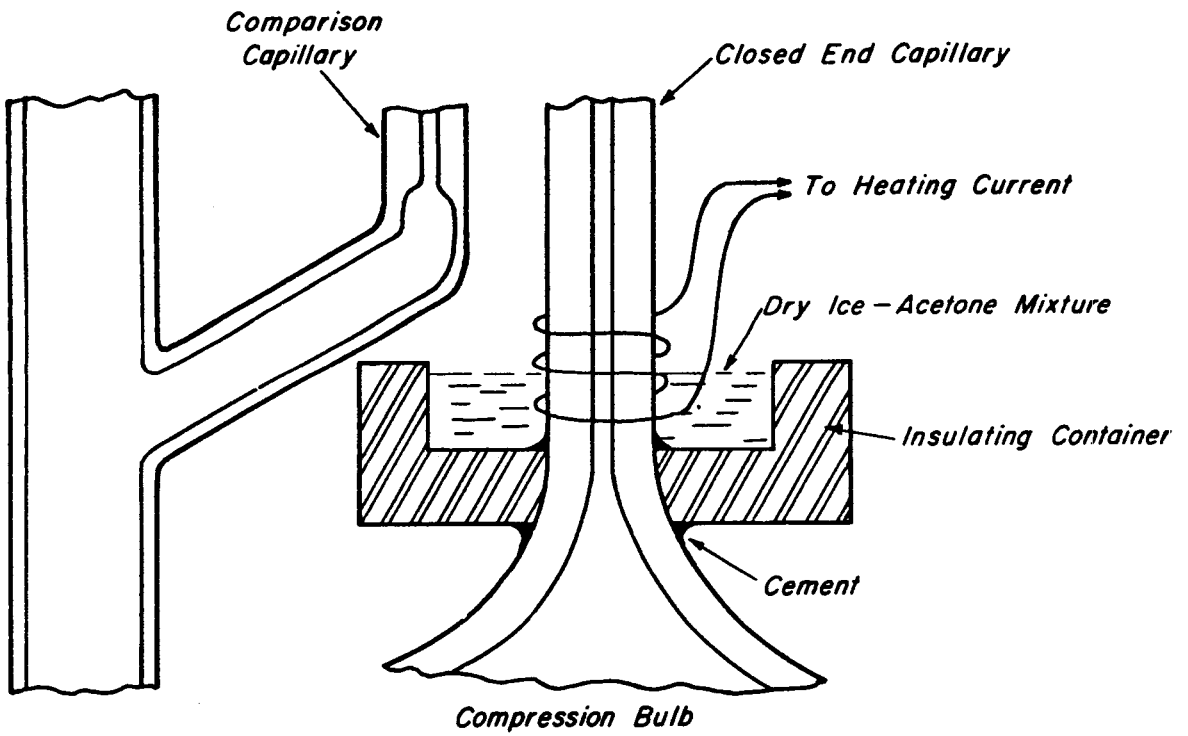

Figure 4. Arrangement for freezing mercury in a McLeod Gauge capillary.

01CA28-410

around the glass capillary tube and are connected to a variac. In our experiments, powdered dry ice was used to cool the capillary and freeze the mercury.

When it is desired to make multiple compression measurements, the base of the closed end capillary is first cooled down with dry ice. The gas sample is then compressed in the normal manner and the top of the mercury volumn in the closed end capillary is brought to the same level as the top of the dry ice. After a few minutes the mercury thread will freeze and the mercury level can be lowered in order to obtain a second sample. In order to combine samples, it is necessary to warm up the frozen pellet of mercury via the heating coil. When the mercury is molten, it will drop down into the main body of liquid mercury which has been positioned a few cm below the base of the closed capillary. The two gas samples are now combined above the surface of the mercury. To continue the process, the base of the closed capillary is cooled again by repacking it with dry ice and the cycle is repeated.

The method described above could possibly be refined by using refrigeration equipment, thermoelectric cooling, etc. to replace the use of a refrigerant such as dry ice. The value of doing this is questionable, however, since the general method has two serious inherent disadvantages. The first of the disadvantages is the relatively long time required to freeze and melt the mercury to obtain valving action; the second, more serious disadvantage is the liklihood that the repeated heating and cooling of the glass capillary will induce strains in the

glass. On previous occasions, glass capillaries have been easily broken after repeated heating and cooling as described above.

The method of freezing mercury in the capillaries has a number of advantages: There is no need to modify the construction of a conventional gauge. Even more important, there is no need to admit any foreign element to the gauge interior. Nevertheless, the frozen mercury storage valve cannot be recommended for use on glass McLeod gauges because of the danger of breakage. For certain type of research, however, where the cooling-heating cycle is repeated only once or twice, this technique can be very useful.

D. ELECTROMAGNETIC MERCURY VALVE

The third method of storing gas that was experimented with was a relatively new one, that of moving and holding the mercury in position by electromagnetic forces. As can be seen from the drawing of Figure 5 and the photographs of Figures 1 and 2, the use of such a valve requires a modification of the McLeod gauge.

Essentially, the electromagnetic storage valve consists of two, long, parallel current electrodes placed along the narrow edges of a rectangular-shaped glass tube. A magnetic field is then created normal to the plane defined by the current electrodes. When mercury is moved upwards into the region between the electrodes, a relatively strong current can be passed through the mercury from one electrode to the other via an external power supply. In our experimental work, an

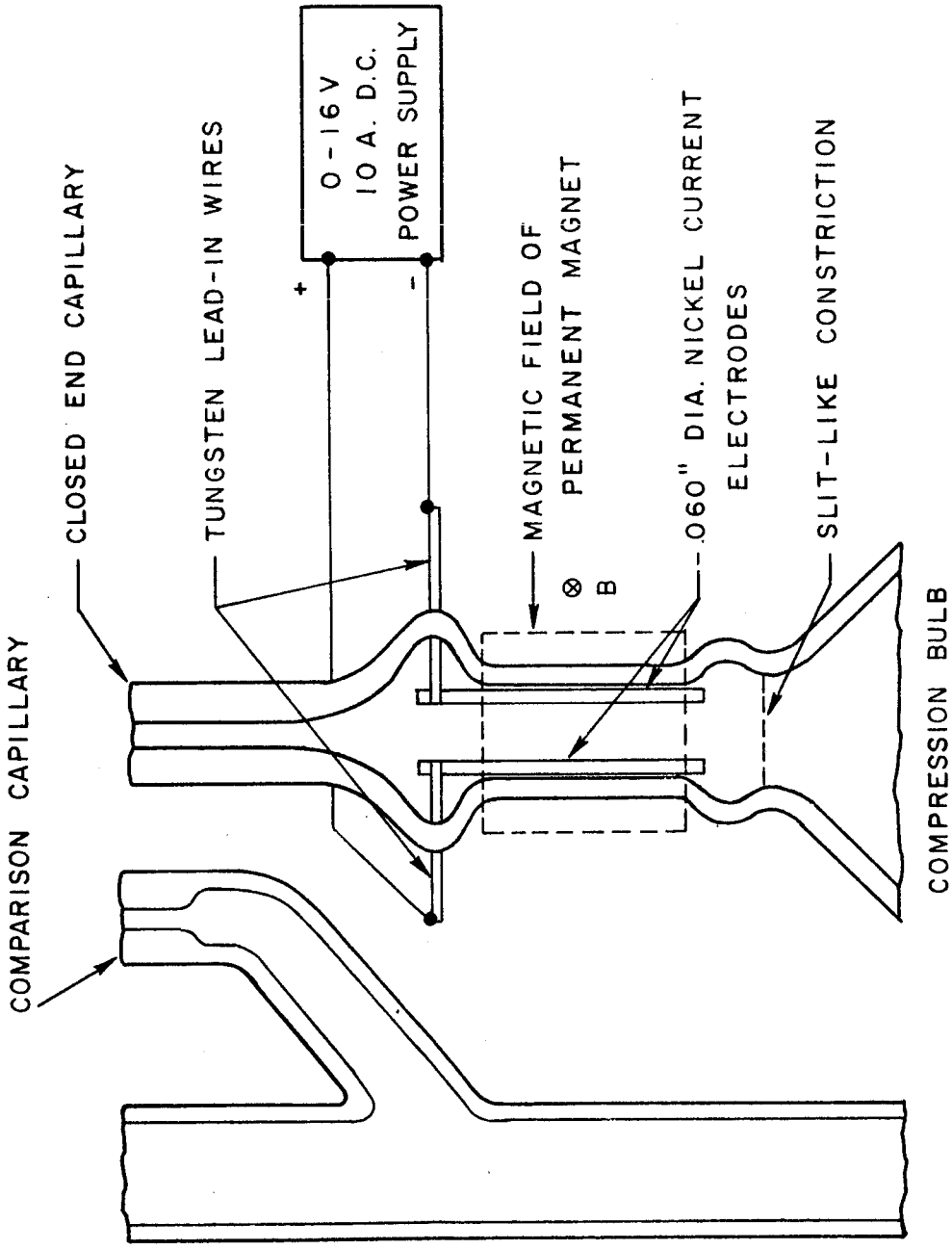

Figure 5. Electromagnetic mercury valve installation in a multiple compression McLeod gauge.

Electro model D612-T filtered D.C. power supply was used. This unit furnished up to 10 amperes of current. Since the situation is that of a conductor carrying current in a transverse magnetic field, there will be a force acting to move the mercury either upwards or downwards perpendicular to both the direction of current flow and the direction of the magnetic field.

Experimentation with the electromagnetic valve shown in Figure 5 showed at once that the electromagnetic forces developed were sufficient to raise or depress the mercury by as much as 2 cm. This appeared to be a sizeable force, and it was believed that there would be no difficulty in holding mercury between the electrodes as the general level of the mercury was lowered. Unfortunately, this belief was an illusion! It soon became very clear that the tensile strength of the clean mercury in the vacuum atmosphere of the gauge was so much greater than the electromagnetic forces that it was impossible to rupture the mercury and retain a layer of mercury between the electrodes, as desired.

In an attempt to increase the possibility of rupturing the mercury film at a point just below the bottom ends of the current electrodes, the mercury valve was first modified by placing a very narrow (about .005" wide) slit-like constriction in the rectangular-shaped tube at this location. The position of this constriction is indicated in Figure 5. The modified valve showed no improvement over the original design. Out of curiosity, it was decided to try a second modification in which the narrow slit-like constriction was replaced by a very small diameter

tubular constriction. Again, there was no change in the behavior of the valve: The mercury could be raised or lowered by the electromagnetic forces, but it could not be ruptured.

During the course of the experiments, one of the disadvantages of such a valve became evident. If care was not taken during the application of current to the valve electrodes, it was possible to create a considerable spark or arc between the mercury and the electrodes at the moment that the surface of the mercury just approached or receded from the bottom ends of the current electrodes.

Such arcing appeared to coat the electrodes and glass walls with an oxide film and undoubtedly caused outgassing of the electrodes. Sparking and arcing between mercury and electrodes must be prevented under all conditions.

An electromagnetic type of mercury valve such as that described above would have the advantage of having no moving parts. In addition, the forces that act on the mercury column are adjustable in magnitude and direction. The magnitude of the electromagnetic force can be expressed in terms of the well known equation F(dynes) = B(gauss) x ℓ(cm) x I(abamperes). The direction of the force F is that of the vector product I x B. In the case of the electromagnetic valve that was constructed for experimental purposes, a magnetic field of about 3000 gauss was used. The rectangular film of mercury between the current electrodes was approximately 0.5 cm long, 3.0 cm wide, and 0.15 cm thick. For a current of 10 amperes (1 abampere) passing through such a

film, the electromagnetic force would be 1,500 dynes. If we assume that this force acts over the entire vertical, cross sectional area of the rectangular mercury film, the upward or downward pressure exerted is 1,500 dynes/0.075cm^2 = 2.0 x 10^4 dynes/cm^2. This is a pressure of 15 mm of mercury. By varying the magnitude and polarity of the current, it is easy to control the height of the mercury column in a continuous fashion.

Summing up the results of experimentation with the electromagnetic valve, it can be said that the valve did not operate as desired. Although the force developed could support a column of mercury about 15 mm long, it was not possible to separate a short segment of mercury of this length from the main body of the liquid. Some additional technique is necessary to pinch-off or rupture the mercury column. This could probably be accomplished by heating the mercury strongly at a point where the column is constricted. However, strong localized heating can strain the glass and lead to eventual mechanical failure. In view of the general complexity of this method of gas storage and the availability of a much simpler method (to be described next), it was decided not to pursue this development.

E. MAGNETIC SPHERE VALVE

The fourth and last method of storing gas in a McLeod gauge makes use of magnetic forces. In this so-called magnetic sphere method, storage is accomplished by effectively sealing-off the bottom open end

of the closed end capillary with a small sphere of magnetic material. As can be seen from the drawing of Figure 6, the method is extremely simple. A small sphere having a diameter that is somewhat greater than the diameter of the capillary is placed within the compression bulb. The sphere is normally held in position on the inner wall of the compression bulb at a point near the entrance of the closed end capillary. A small permanent magnet or an electromagnet can be used to hold the magnetic sphere in position. If, now, the external magnet is moved upwards to the position shown in the illustration, the small sphere can be lodged tightly in the tapered portion of glass that leads to the cylindrical capillary bore. When the magnetic field is sufficiently strong and the taper of the glass is sufficiently uniform, such a sphere can support a column of mercury that may be 80 to 100 mm in length. In ordinary useage, the sphere supports a column of mercury that may vary from 10 to 40 mm in length. As has been mentioned earlier, it is the column of mercury which the sphere supports that actually seals off the base of the capillary and keeps the gas sample stored above this mercury column isolated from the rest of the gauge.

It is immediately evident that the magnetic sphere method of gas storage requires no modification of the McLeod gauge. Three different designs of McLeod gauge were fitted with the magnetic sphere with no difficulty. Generally, the sphere is introduced into the gauge via its tubulation to the vacuum system. External magnets are used to guide the sphere into the compression bulb. In some cases where the compression

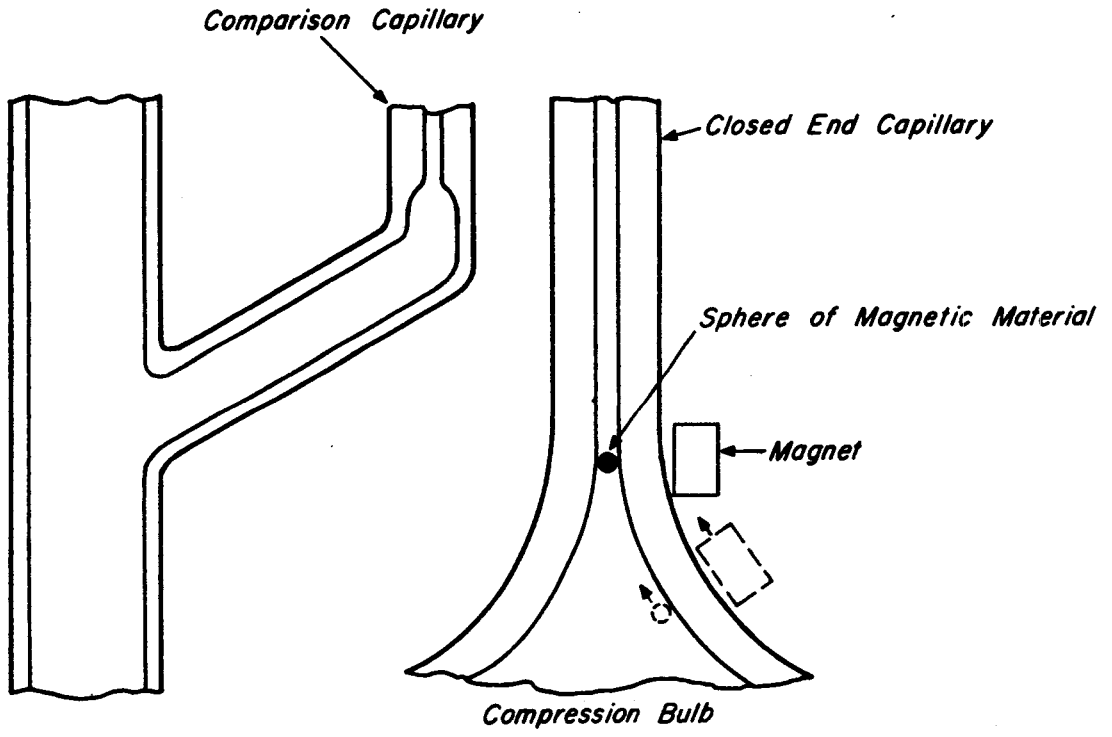

Figure 6. Magnetic sphere valve arrangement for storing gas in a McLeod Gauge capillary.

01CA28-430

bulb can only be entered via small diameter tubing that is ring sealed to the main tubing, it has been necessary to use the gauge mercury to help install the magnetic sphere.

There are a number of materials from which the small sphere can be made. Chrome alloy and type 440 stainless steel balls have been used successfully. Other materials such as pure nickel, kovar, and pure iron would also be suitable. Spheres of the order of 3/32" diameter have been used to date.

If a permanent magnet is used outside the gauge to move the magnetic sphere, the magnet is most conveniently mounted on some sort of pivot arm so that it can be moved quickly and reproducibly between its two fixed positions. The magnet may be moved manually or it may be moved electrically with the aid of an electromagnetic solenoid.

A second method of moving the magnetic sphere is to use a combination permanent magnet-electromagnet assembly in which there is no physical movement of the assembly. In this arrangement, the permanent magnet is used to keep the sphere in the "up" or "closed" position of the valve. When the electromagnet is energized, the sphere is pulled downward to open the valve. If the valve is to be kept open for any length of time, a second permanent magnet is used in place of the electromagnet to keep the magnetic sphere in its lower, open position.

The magnetic sphere mercury valve operates in the following way: The first sample of gas is admitted to the McLeod gauge and the mercury level is raised. With the valve sphere in the "down" or "open" position,

the gas sample is compressed and forced into the closed end capillary. When the mercury surface in the closed capillary has reached a position that is 10 to 40 mm above the base of the capillary, the magnetic sphere is moved to the "up" or "closed" position. The natural buoyancy of the sphere in the presence of the heavy liquid mercury aids in this upward movement. At this point, the mercury is lowered and will break away from beneath the magnetic sphere, leaving an isolated column of mercury above the sphere. The first sample of gas is now stored in the closed end capillary. The mercury is lowered below the compression bulb cut-off level and a second sample of gas is permitted to enter the compression bulb of the gauge and come to equilibrium. The mercury level in the gauge is raised again to compress the second gas sample. When the mercury level reaches a position just below the magnetic sphere, the sphere is quickly lowered to its open position, allowing the previously isolated column of mercury above the sphere to fall out of the capillary into the main body of mercury. In this way, the two samples of gas are combined. The mercury is now raised (with the valve sphere still open) until the mercury surface in the closed capillary has again reached a position that is 10 to 40 mm above the base of the capillary. The magnetic sphere is moved to the "up" or "closed" position. The mercury is then lowered again so that it breaks away from beneath the magnetic sphere, leaving the combined first and second gas samples stored in the closed capillary. This process is repeated as many times as desired. McLeod gauge readings may be taken as each new sample is added to the combined sample in the closed end capillary.

The magnetic sphere mercury valve arrangement has many advantages and few disadvantages as a method for storing gas in a multiple compression McLeod gauge. One of the most valuable features of this method is the absence of any need to modify the conventional McLeod gauge. Any McLeod gauge can be transformed into a multiple compression instrument very quickly and simply. And when a gauge has been so transformed, it may still be used to make single readings in the normal fashion. The tiny magnetic sphere which is introduced into the gauge has a negligible volume compared to the compression bulb volume, and thus the gauge calibration factors are essentially unchanged. The small volume and surface area of the sphere also minimizes the outgassing which it can contribute to the interior of the gauge. The light weight and small size of the sphere insure a minimum of mechanical shock and mercury turbulence. With the use of an electromagnet external to the gauge, there is no necessity for moving parts outside of the gauge. The gauge capillary will be neither heated nor cooled by this device. There is no danger of straining the glass with subsequent breakage. Nor is there any danger of electrical arcing or sparking within the gauge.

The only practical disadvantage of the magnetic sphere valve is that something must be put inside the gauge. Also the valve action is not automatic in the same way that the float valve action is automatic, and so some additional mechanism is required to open and close the valve.

F. COMPARISON OF STORAGE METHODS

In order to sum up the advantages and disadvantages of the different methods of storing gas in a multiple compression McLeod gauge,

one can organize the pertinent information as shown in Table I. This table lists ten important, desirable characteristics of a multiple compression McLeod gauge gas storage device or technique. The characteristics listed are not all inclusive, but they are features which are clearly present or absent in the four gas storage techniques under consideration. The very important matter of gas trapping between the mercury and the glass wall or valve mechanism has not been included in the Table because practically no data concerning this characteristic is available. The mercury freezing technique of gas storage, however, should provide a minimum of gas trapping because there is nothing placed within the gauge.

When the relative merits of the four gas storage devices described above are compared, it becomes apparent that the magnetic sphere mercury valve has the greatest number of advantages and the fewest disadvantages. For this reason, it was decided to employ the magnetic sphere arrangement with a commercially available low pressure McLeod gauge and use this gauge to test the usefulness of the multiple compression technique.

TABLE I

RELATIVE MERITS OF FOUR METHODS OF

STORING GAS IN A MULTIPLE COMPRESSION MCLEOD GAUGE

Desirable Characteristics	Float Valve	Freezing Technique	Electro-magnetic Valve	Magnetic Sphere Valve
Gauge Modification not required		X		X
No material placed within gauge		X		
No change in gauge calibration		X		X
Rapid valving action	X		X	X
No danger of mechanical shock		X	X	X
No danger of thermal shock	X			X
No significant outgassing		X		X
No danger of electric arcing	X	X		X
Inexpensive gauge accessories	X	X		X
Automatic operation	X			

IV. LOW PRESSURE MULTIPLE COMPRESSION MCLEOD GAUGE SYSTEM

In order to evaluate the usefulness of a multiple compression McLeod gauge, it was necessary to construct a complete low pressure pumping system. This system is shown in the photograph of Figure 7.

Basically, the vacuum system was an all glass, mercury diffusion pump system. An H. S. Martin Company model M-40112 three stage mercury-vapor diffusion pump was used to create background or residual gas pressure as low as 6×10^{-9} mm Hg. The nominal speed of the pump is listed as 80 liters/sec. A Welch model 1402B 140 liter/min mechanical pump was used to obtain the forevacuum pressure required by the diffusion pump. A dry ice cold trap separated the mechanical and diffusion pumps. This trap minimized the flow of mercury vapor into the mechanical pump and also minimized the flow of oil vapor into the diffusion pump.

As can be seen in the photograph, there were a number of cold traps in the system. The cold trap, which was adjacent to the diffusion pump, was a standard cylindrical liquid nitrogen trap that was furnished with the diffusion pump. A second liquid nitrogen cold trap of the spherical variety followed the cylindrical trap. A hot filament ionization gauge (Veeco model RG75P) and a valve controlled tubulation to a 1 liter reagent grade gas bottle of pure nitrogen were connected to the system between the cold traps just described. A third, symmetrical H-Type liquid nitrogen cold trap was used to separate the McLeod gauge from the rest of the

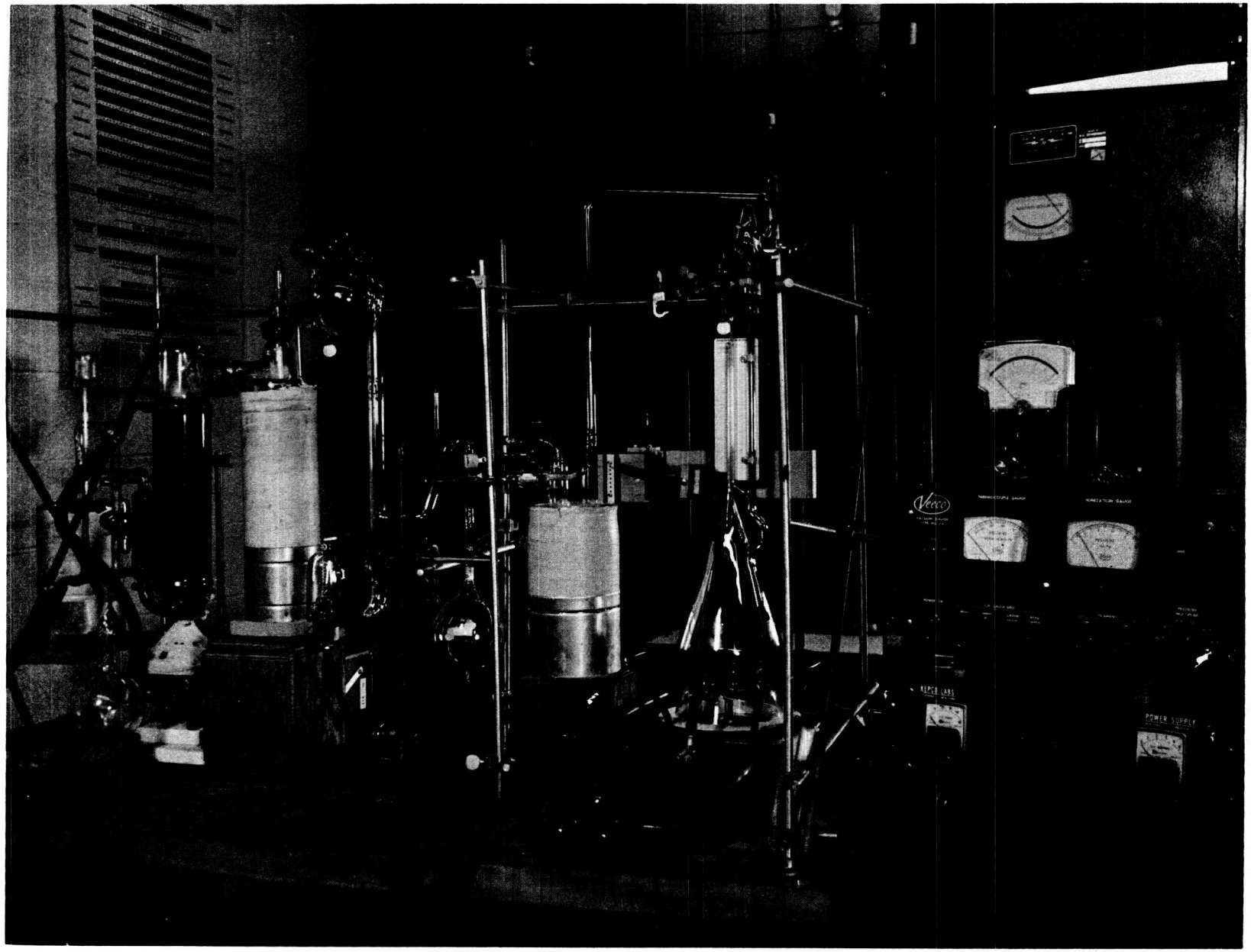

Figure 7. Low pressure pumping system used to evaluate the operational characteristics of a multiple compression McLeod gauge.

system. A second hot filament ionization gauge was located between the spherical cold trap and the symmetrical H-type cold trap. The reading of the latter ion gauge was taken to be representative of the gas pressure in the system, that is, of the gas pressure being measured by the McLeod gauge. The McLeod gauge was a Consolidated Vacuum Corp. model GM-110 which has a smallest scale division of 1×10^{-7} mm Hg and a gauge constant of approximately 1×10^{-7} mm Hg/mm^2. The gauge was mounted within a large plastic container for two reasons: First, the container is a safety device which would catch most of the gauge mercury in the event of breakage. Second, the container could be filled with a cooling bath of ice water in order to reduce the mercury vapor streaming from the gauge to the symmetrical H-Type cold trap. The mercury reservoir was connected through a metal Y connector to two Hoke model DR297 needle valves. One of these valves was connected to a Welch model 1400B mechanical pump while the other was connected to the atmosphere through a drying tube that contained Drierite indicating desiccant and glass wool filters. It should be noted that there was no valve separating the McLeod gauge from the rest of the vacuum system.

The magnetic sphere valve arrangement was used with this McLeod gauge to permit multiple compression measurements to be made. A small 3/32" diameter sphere made of No. 440 stainless steel was introduced into the McLeod gauge with the aid of permanent magnets. The sphere was held in either the open or closed positions by means of a small external C-Type magnet (Arnold Engineering Company Type No 8756) that was mounted

on a pivoted arm. The magnet holder was a bi-stable device in which the pivot arm could assume either of two stable positions.

The stable positions of the pivot arm were adjustable and reproducible. The magnet holder was mounted to the two upright rods that were a part of the gauge support structure. Provision was made to adjust the position of the magnet along three mutually perpendicular axes. The magnet holder was made almost entirely of aluminum. The photograph of Figure 8 shows both the McLeod gauge and the magnet holder. The small magnetic sphere can be seen within the gauge adjacent to the magnet.

In normal operation, the system would attain a background pressure of about 6×10^{-8} mm Hg after about 24 hours of pumping, with no liquid nitrogen present in the spherical cold trap. After an outgassing period of about 1 hour (heating the glass to 300 to 400°C with a heat gun) and filling the spherical cold trap with liquid nitrogen, the pressure in the system would drop into the 10^{-9} mm Hg region in a few hours. Usually, when multiple compression measurements were to be made, the system would first be pumped down to the lowest background pressure attainable and then the metal Hoke valve would be opened slowly to start a flow of pure nitrogen into the diffusion pump. The gas pressure in the system would be monitored with the system ionization gauge, which was operated at a reduced emission of 100 microamperes to minimize its pumping effect. When possible, the gas flow was left undisturbed for at least 3 or 4 hours before any measurements were started.

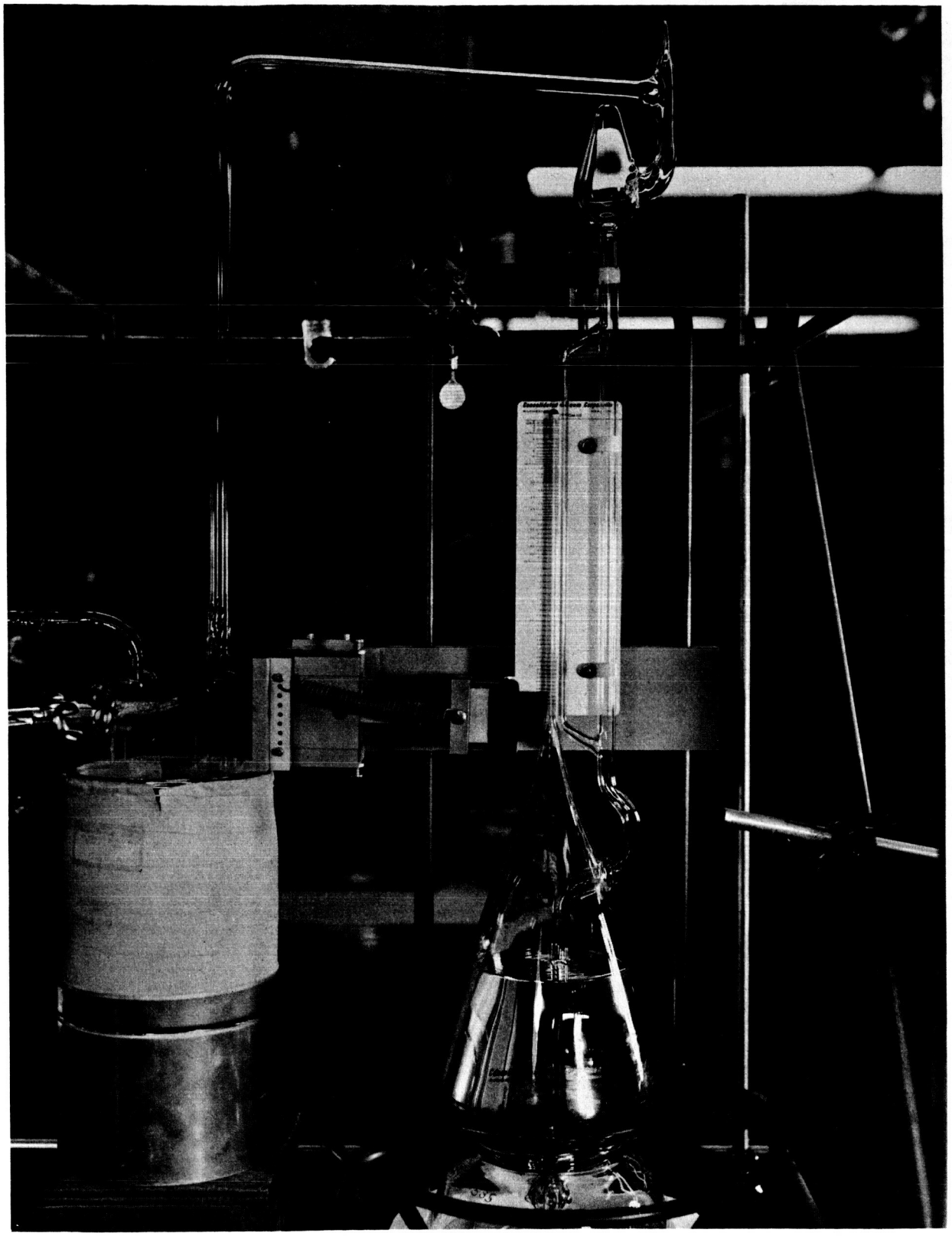

Figure 8. Close-up view of the McLeod gauge used for multiple compression measurements.

The positions of the mercury columns of the McLeod gauge were measured with an Ealing Cathetometer model 11-550 which would measure vertical positions with an accuracy of 0.05 mm. The cathetometer was leveled, and the telescope cross hairs were adjusted to the vertical with the aid of a plumb bob. A mercury-in-glass laboratory thermometer was used to measure the ambient temperature.

The mercury in the McLeod gauge was triple distilled, instrument grade mercury. It was admitted to the gauge reservoir after vacuum filtration through a coarse fritted glass filter in order to remove particles of dirt or dust from the surface. The mercury was outgassed by raising it frequently into the low pressure region of the compression bulb over a period of several weeks. The degree to which the mercury had been outgassed could be determined in many ways. The outgassing rate of the mercury was obviously low enough to permit background pressures of 6×10^{-9} mm Hg to be attained. This imposed a rough upper limit for outgassing of about 7×10^{-8} mm Hg liters/cm^2/sec. The outgassing of the mercury could be measured by raising the mercury above the cutoff level of the compression bulb and exposing a known surface area of mercury to the high vacuum (of the order of 6×10^{-9} mm Hg) of the compression bulb. The mercury was thus permitted to outgass into the known volume of the compression bulb for a measured interval of time. After this time interval, the McLeod gauge was used to measure the gas that had accumulated in the bulb during this period. In one recent test, for example, a final pressure of 5×10^{-7} mm Hg was measured with a single compression of the gauge. The compression

bulb volume of the gauge was approximately 2 liters and the outgassing mercury surface of 100 cm^2 was exposed for 10^3 sec. The mercury outgassing rate corresponding to this measurement was thus 1×10^{-11} mm Hg liters/cm^2/sec. Outgassing rates much lower than this were usually obtained prior to experimentation.

In all the important experiments that were performed to determine the operational characteristics of a multiple compression McLeod gauge, the lowest possible background pressure was first attained by long term pumping and outgassing of the system. After this, a flow of pure nitrogen gas was initiated to raise the system pressure to the desired level, and this flow was maintained for many hours prior to experimentation. In this way, impurities in the nitrogen gas being measured were kept to a minimum.

V. EXPERIMENTAL RESULTS

There are at least three fundamental experiments that can be performed with a multiple compression McLeod gauge. The first experiment is to measure a pressure that can be measured well within the normal pressure range of the gauge with some degree of confidence. This pressure is measured using the ordinary, single compression technique and also the multiple compression technique. The results of these two types of measurements can then be compared.

A second fundamental experiment is to measure a pressure which is within the range of the gauge but is low enough so that it cannot be measured with confidence using the ordinary single compression technique. Again, the results of single compression and multiple compression measurements are to be compared.

Finally, a third fundamental experiment is to measure a gas pressure which is so low that it is below the normal range of the McLeod gauge when used in the ordinary fashion. In addition to the experiments listed above, it should be determined whether or not there is any adsorption or desorption of gas in the McLeod gauge during the relatively long measuring period. All of the experiments mentioned above have been performed and are described below.

A. IMPROVED PRECISION IN MEASURING PRESSURES WITHIN THE NORMAL RANGE OF THE GAUGE.

The first experiment to be carried out with the multiple compression McLeod gauge was to compare the readings taken with the

gauge in the normal fashion (a single compression) with those obtained after several compressions. A gas pressure of approximately 9×10^{-6} mm Hg was chosen to be measured for several reasons. First of all, this pressure could be measured with the gauge with an error that was just greater than ± 4.4 per cent, according to the manufacturer. The error determination was based on a ± 0.25 mm Hg precision in reading the position of the mercury meniscus. A pressure of 9×10^{-6} mm Hg could also be measured with the system hot filament ionization gauge with good precision. Small changes in the system pressure could be easily detected during the period of time that McLeod gauge measurements were made.

The experimental procedure used was to first obtain a background pressure of about 1×10^{-7} mm Hg in the system, after which pure nitrogen gas was permitted to flow into the diffusion pump to raise the system pressure to approximately 9×10^{-6} mm Hg. The system ionization gauge was operated at a reduced emission of 100 μA to measure and monitor this pressure. The system was permitted to pump nitrogen gas at this pressure for about 16 hours before the experiment was started.

The experiment was performed in the following way: A measurement of the nominal gas pressure of 9×10^{-6} mm Hg would first be made by collecting a single sample of gas in the McLeod gauge in the usual way and then making five separate measurements of the pressure of this sample. Each measurement was made by lowering the mercury to the base of the capillary tubes and then raising it again so that the meniscus

of the mercury in the open capillary was at the same level as the closed end of the closed end capillary. In other words, square law operation was used. After these first five measurements, the first sample was stored in the closed end capillary by means of a magnetic sphere valve, and a second sample of gas was collected in the McLeod gauge. The first and second samples were combined and five measurements were made of the combined pressures of these two samples. The same procedure was repeated for the third, fourth, and fifth samples. Mercury menisci positions and the position of the top of the closed end capillary were measured with a cathetometer. A period of 10 minutes was allowed for the collection of each nitrogen gas sample.

The results of these measurements are shown in Table II. The first column of this table lists the number of compressions (that is, the number of gas samples collected). The second column shows the reading of the Veeco hot filament ionization gauge which was monitoring the system pressure in a relative way. The data was taken without interruption for a period of 12 hours. Notice that the pressure in the system apparently decreased by about 2.2 per cent during this period. There is a hint of this decreasing pressure in the McLeod gauge measurements, as will be seen shortly. The third column lists the average values of the five measurements obtained for each of the mercury column lengths that represent the final pressures of the combined gas samples in the closed end capillary. In other words, this is the value of the quantity h_n in the expression for the McLeod gauge pressure $P_n = Kh_n^2$ where

TABLE II

COMPARISON OF PRESSURE READINGS AND THEIR DEVIATIONS FOR MULTIPLE COMPRESSION MCLEOD GAUGE MEASUREMENT.

(Each average is an average of five readings.)

1	2	3	4	5	6	7	8	9
Number of Compressions	Ionization Gauge System Pressure Reading	Combined Sample Average Hg Column Length	Combined Sample Standard Deviation of Hg Column Length	Combined Sample Average Pressure	Fractional Standard Deviation of Combined Sample Pressure	Differences of Combined Sample Average Pressures	*Average Pressure of a Single Sample	Fractional Standard Deviation of Average Sample Pressure
n	P_s (mm Hg)	h_n (mm)	S_{h_n} (mm)	$P_n(h_n)$ (mm Hg)	S_{p_n}/P_n (%)	$P_n - P_{n-1}$ (mm Hg)	$P_{n1}=P_n/n$ (mm Hg)	$S_{p_{n1}}/P_{n1}$ (%)
1	9.10×10^{-6}	8.94	0.134	8.185×10^{-6}	2.99	8.18×10^{-6}	8.18×10^{-6}	2.99
2	9.10×10^{-6}	13.28	0.144	1.806×10^{-5}	2.17	9.88×10^{-6}	9.03×10^{-6}	2.17
3	9.00×10^{-6}	16.06	0.167	2.641×10^{-5}	2.08	8.35×10^{-6}	8.80×10^{-6}	2.08
4	8.95×10^{-6}	18.61	0.108	3.546×10^{-5}	1.16	9.06×10^{-6}	8.87×10^{-6}	1.16
5	8.90×10^{-6}	20.79	0.065	4.426×10^{-5}	0.63	8.79×10^{-6}	8.85×10^{-6}	0.63

* These values are based on the assumption that the individual contributions of each sample or compression are equal. According to Column 7, these contributions are quite equal.

K is the gauge constant. The next column lists the standard deviations S_{h_n} associated with each of the average values h_n and is a measure of the precision or reproducibility with which the column length h_n could be determined. The absolute deviations for measurements taken with 1, 2, and 3 compressions increased in that order, while the absolute deviations decreased for measurements taken with 3, 4, and 5 compressions. The interpretation of this behavior is not clear, since one would expect the absolute deviations to either stay constant or continually decrease as the gas pressure builds up within the closed end capillary. Of course the relative deviations S_{h_n}/h_n continuously decrease with the increasing amount of gas being measured, and this is reflected in the decreasing fractional standard deviation of the combined sample pressure as listed in Column 6. Column 5 of the table shows the computed values of the combined sample average pressures. The gauge constant was $K = 1.0241 \times 10^{-7}$ mm Hg/(mm)^2 corresponding to a compression bulb volume of 2195 cm^3 and a closed end capillary diameter of 0.535 mm. The sixth column of the Table shows clearly how the precision of the gas pressure measurement increases as the combined or accumulated gas sample pressure increases. An accumulation of five gas samples (n = 5), for example, was measured with a relative deviation of less than 1 per cent.

At this point, the crucial part of the entire experiment is approached. It is evident from what has just been said that an accumulation of n gas samples can be measured with a greater precision than a single sample. The question to be asked next is this: What precision can be

attributed to the measurement of a single sample component of the total accumulated sample? As shown in Column 7, one can determine the single sample component pressures by subtracting one combined sample average pressure from the next. The average value of the single sample components listed in Column 7 is the pressure $\overline{P}_{51} = 8.85 \times 10^{-6}$ mm Hg listed at the bottom of Column 8. Notice that the first sample measurement of 8.18×10^{-6} mm Hg is about 7.6% below the average value. The second single sample component measurement of 9.88×10^{-6} mm Hg is about 11.6% above the average value. The third single sample component is 5.6% below the average value, the fourth single sample component measurement is about 2.4% above the average value, while the fifth and last single sample component measurement is only about 0.68% below the average value. Thus it is clearly seen how the measurements of an individual gas sample (results of a single compression) become less scattered and begin to cluster about an average value. The same trend is evident if one assumes that the individual single sample components are equal and can be found by dividing the accumulated or combined sample average pressures by the respective number of samples or compressions. The results of this operation are shown in Column 8. Notice that the values for 3 and 4 compressions closely approach the "best" value of 8.85×10^{-6} mm Hg, which is the average of the five compressions. The figures of Column 7 and 8 show how the measurements of a single sample component approach a sort of limiting value.

Having found that one can determine the pressure of a single sample component by dividing the total accumulated pressure by the number of

compressions, and having further discovered that the values so obtained approach a limiting value as the number of compressions increases, it remains to assign some measure of precision to the single sample component pressures. One way to do this is to make the fundamental assumption that the errors of the individual samples are independent so that the square of the deviation of the combined or accumulated sample is equal to the sum of the squares of the deviations of the individual single sample components. If, for example, the standard deviation of the accumulated pressure after n compressions is Sp_n, then the deviations Sp_{n1} to be assigned to the n equal, single sample components $Pn_1 = P_n/n$ are found from the relationship: $Sp_n^2 = nSp_{n1}^2$ or $Spn_1 = (1/\sqrt{n})\ Sp_n$. The standard deviations Sp_n, and hence Sp_{n1}, are deviations for a <u>single measurement or reading</u>. The corresponding standard deviations for the <u>average values</u> of series of measurements or readings are reduced by multiplying the single measurement deviations by the factor $1/\sqrt{n}$. Thus the values of Sp_{n1} for the deviations of the <u>average</u> pressure $P_{n1} = P_n/n$ are exactly the values Sp_n/n, so that, finally, the fractional standard deviations of an <u>average</u> single sample pressure is identical with the fractional standard deviation of a single measurement of a combined or accumulated sample pressure. This accounts for the equality of the deviations in Columns 6 and 9. Column 9 illustrates the improved precision that is obtained by using more compressions. The "best" or most representative value of the system pressure measured in this experiment, excluding systematic errors, was the value $p = 8.85 \times 10^{-6}$ mm Hg. The fractional standard deviation corresponding to this pressure was 0.63%.

Returning to the objective of the experiment, it can be seen that the random error obtained for the single compression measurements was about 3 per cent and that this error steadily decreased as more compressions were made. It is interesting to notice that the presence of a rather large systematic error in the measurement of the system pressure using a single compression was indicated by the rather large discrepancy between the pressure value $P_{11} = 8.18 \times 10^{-6}$ mm Hg and the final pressure $P_{51} = 8.85 \times 10^{-6}$ mm Hg, at the same time that the random error in P_{11} was about 3 per cent. This particular systematic error was probably due in part to the smallness of the gas pressure after one compression when compared with surface forces acting in the capillary. The point to be made here is that the multiple compression technique not only reduces the random or experimental errors of measurement but also reduces those systematic errors that arise because of the smallness of the gas sample when it is confined near the top of the closed end capillary. The reduction of certain systematic errors through the use of the multiple compression technique will be further illustrated in the description of the next experiment.

B. IMPROVED ACCURACY IN MEASURING PRESSURES AT THE LOWER LIMIT OF A MCLEOD GAUGE'S RANGE.

The second experiment that was performed with the multiple compression McLeod gauge was to take a series of 20 compressions or samples of a nominal system pressure of about 9×10^{-7} mm Hg of nitrogen gas. Except for the first and eleventh compressions, readings were taken after every other compression, beginning with the second compression.

The objective of this experiment was to determine the degree of confidence that could be placed in a measurement made near the lower limit of a McLeod gauge's range.

According to the manufacturer of the McLeod gauge that was used, a pressure of 1×10^{-6} (somewhat greater than the pressure under consideration) could be measured with an error of ± 14 per cent, based on a ± 0.25 mm Hg precision in reading the position of the mercury meniscus. If this error computation is correct, even a single compression measurement of a pressure of 9×10^{-7} mm Hg would yield a reasonably accurate and useful reading.

As in the first experiment, the nominal system pressure was measured with a hot filament ionization gauge to determine the relative change in pressure during the experimental period. The gauge was operated at a reduced emission of 100 microamperes to minimize its pumping effect.

The vacuum system was prepared for the experiment by first attaining a background pressure of about 3×10^{-8} mm Hg. After this, the flow of pure nitrogen gas into the diffusion pump was adjusted until the hot filament Bayard Alpert gauge indicated a pressure of 9.4×10^{-7} mm Hg. The flow of nitrogen continued for a period of about 21 hours before the experiment began. Slightly less than 4 hours before the experiment began, the spherical cold trap that separated the ionization and McLeod gauges from the point at which the nitrogen gas entered the system was filled with liquid nitrogen. The system pressure, as read by the ionization gauge, decreased to 8.8×10^{-7} mm Hg, thereby indicating that approxi-

mately 3 to 4 per cent of the incoming gas consisted of condensables.

The taking of data during this experiment was divided into two sessions. The first 10 compressions or gas samples were taken during a 5 hour period. The combined or accumulated 10 samples were then stored in the closed end capillary overnight for a period of about 14 hours. After this, the next ten compressions were made during a 6.5 hour period. The pressure of the accumulated 10 gas samples before the 14 hour storage period when compared with the pressure of 11 gas samples just after the storage period, gave a measure of the adsorption of nitrogen in the capillary or compression bulb. The accumulated 20 samples were then stored for 42.5 hours in the closed capillary, a 21st sample was added, and the final pressure was measured.

Only a single pressure reading was taken for each of the accumulated samples that was measured. The measurements were made using square law operation of the gauge, as in the first experiment. The same cathetometer was used to measure the positions of the mercury menisci and the top of the closed end capillary. A 10 minute sampling period was used, except for the 19th and 20th compressions in which a 5 minute sampling period was used. The ambient temperature varied between 24.5^{o}C and 25.5^{o}C during the experiment.

The results of the measurements made during this second experiment are shown in Table III and the graph of Figure 9.

TABLE III

MEASUREMENT OF A LOW GAS PRESSURE WITH A MULTIPLE COMPRESSION MCLEOD GAUGE

1	2	3	4	5	6	7
Number of Compressions	Ionization Gauge System Pressure Reading	Combined Sample Mercury Column Length	Combined Sample Pressure	Differences of Combined Sample Pressures	Average Pressure of Single Sample	†Adjusted Average Pressure of Single Sample
n	P_s	h_n	$*P_n = Kh_n^2$	$P_n - P_{n'-2}$	$P = \frac{P_n}{n}$	P_A
	(mm Hg)	(mm)	(mm Hg)	(mm Hg)	(mm Hg)	(mm Hg)
1	8.80×10^{-7}	1.10	1.24×10^{-7}	---	1.24×10^{-7}	1.24×10^{-7}
2	8.80×10^{-7}	1.95	3.89×10^{-7}	3.89×10^{-7}	1.95×10^{-7}	1.95×10^{-7}
4	8.75×10^{-7}	3.60	1.327×10^{-6}	9.38×10^{-7}	3.32×10^{-7}	3.32×10^{-7}
6	8.75×10^{-7}	5.00	2.560×10^{-6}	1.23×10^{-6}	4.27×10^{-7}	4.27×10^{-7}
8	8.70×10^{-7}	7.90	6.391×10^{-6}	3.83×10^{-6}	7.99×10^{-7}	7.99×10^{-7}
10	8.70×10^{-7}	9.65	9.536×10^{-6}	3.14×10^{-6}	9.54×10^{-7}	9.54×10^{-7}
**11	8.60×10^{-7}	9.65	9.536×10^{-6}	---	8.67×10^{-7}	9.54×10^{-7}
12	8.60×10^{-7}	9.80	9.835×10^{-6}	2.99×10^{-7}	8.20×10^{-7}	9.84×10^{-7}
14	8.55×10^{-7}	10.25	1.076×10^{-5}	9.20×10^{-7}	7.71×10^{-7}	9.78×10^{-7}
16	8.55×10^{-7}	11.35	1.319×10^{-5}	2.43×10^{-6}	8.25×10^{-7}	1.01×10^{-6}
18	8.55×10^{-7}	11.80	1.426×10^{-5}	1.07×10^{-6}	7.94×10^{-7}	9.51×10^{-7}
20	8.55×10^{-7}	12.70	1.652×10^{-5}	2.26×10^{-6}	8.25×10^{-7}	9.72×10^{-7}
**21	8.55×10^{-7}	12.70	1.652×10^{-5}	---	7.86×10^{-7}	---

* The gauge constant was $K = 1.024 \times 10^{-7}$ mm Hg $(mm)^2$.

** These compressions and measurements were made after the gas was stored for long periods of time in the closed end capillary.

† These computations were made on the assumption that no gas was added during the 11th, 12th, and 13th compressions.

As in Table II, the first two columns of Table III list the number of compressions and the nominal system pressure as measured with the Bayard Alpert Type ionization gauge. It can be seen from the figures in Column 2 that the system pressure slowly decreased during the period of time that measurements were being made. The overall decrease in pressure was something less than 3 per cent. Column 3 of the table lists the lengths of the mercury columns corresponding to the final pressures of the combined gas samples in the closed end capillary. This, in a sense, is the raw data from which the combined sample pressures of Column 4 are computed. Notice that in Column 4 the combined sample pressure increased for the first 10 compressions. After these 10 compressions were made, the combined sample was stored in the closed end capillary for a period of 14 hours. After the tenth compression, the magnetic sphere valve being used was closed, and it supported a column of mercury about 1 cm long at the base of the capillary to form a positive gas seal. The mercury in the compression bulb was then lowered to a level just above the cut-off position of the compression bulb and was kept in this position for the 14 hour storage period. During this period of time, the wall of the compression bulb was exposed to a very low pressure since the only gas within the bulb was that due to outgassing of the mercury and desorption from the glass. When data taking was resumed, the first step was to lower the mercury below the compression bulb cut-off level in order to obtain a new (the 11th) gas sample. As the mercury was so lowered, it was noticed that the system hot filament gauge registered a <u>decrease</u> in pressure, indicating that there had been

no pressure build-up in the compression bulb to a level comparable with the system pressure of 8.60×10^{-7} mm Hg. It was also noticed that there were very fine droplets of mercury condensed on the walls of the compression bulb. The mercury was allowed to rise into the compression bulb to collect these droplets and was then lowered once more below the cut-off level. Approximately 23 minutes after the mercury was lowered below the cut-off level, the 11th compression was started.

As shown in Column 4, there was no apparent increase in the pressure of the combined gas sample between the 10th and 11th compressions. Further, there was only a very small increase in pressure after the 12th compression. This sort of behavior suggests that the 11th, 12th, and even 13th gas samples were mostly adsorbed on the wall of the compression bulb and never reached the closed end capillary. An alternative explanation would be that an amount of gas just equal to that contained in a single sample was temporarily adsorbed in the closed end capillary during the 14 hour storage period. However, this explanation does not account for the small amount of gas added in the 12th and 13th compressions. And to further discredit this explanation, one can see from the data that after the 20th compression when the combined sample was stored for 42.5 hours in the closed capillary in the same way as before, the 21st compression again left the total pressure unchanged. The most reasonable explanation in these cases is that there was no adsorption of the stored gas in the closed end capillary, but because of the manner in which the

compression bulb was isolated at a low pressure during the storage period, the glass walls of the bulb became relatively gas free and thus constituted a good pumping surface. When new samples of gas were admitted to the gauge, the majority of the molecules were "pumped" by the wall until a new equilibrium between adsorption and desorption was established.

The figures listed in Column 5 of Table III show the differences in pressure between successive readings. Each of these numbers represents the increase in pressure due to two compression samples. At a nominal system pressure of 9×10^{-7} mm Hg, each of these figures in Column 5 would be 1.8×10^{-6} mm Hg for a fictitious "ideal" McLeod gauge. It can be seen that the first six compressions gave pressure increases well below this value, indicating large systematic errors for gauge operation in the region near the low pressure limit of the gauge. On the other hand, the data for the 8th and 10th compressions show clearly that although lower than normal pressure readings were obtained for the first six compressions, none of the gas collected during these compressions was lost, and when the gauge operation moved to a region of smaller systematic error, the presence of the accumulated gas samples was manifested by higher than normal pressure increases. The small pressure increases after the 12th and 14th compressions were due to adsorption in the compression bulb, as already indicated. The apparent oscillation of the pressure increases about the "ideal" value of 1.8×10^{-6} mm Hg for the 16th, 18th, and 20th compressions could be due to the "overshoot" occasioned by the abnormally low pressure increases that were caused by compression bulb adsorption.

Column 6 of Table III lists the average pressure of a single gas sample as obtained by dividing the total accumulated pressure by the number of compressions made. This column shows the systematic nature of the errors involved in making measurements near the low pressure limit of the gauge. After a sufficient number of samples have been collected, the average pressure of a single sample begins to fall within a limited range. That is, the systematic change in the pressure of a single sample is eliminated and only random variations about some "average" value remain.

Because of the unusual influence of the method used to store the accumulated gas samples for 14 hours after 10 compressions had been made, an attempt was made to remove the effects of the storage period by adjusting the data as follows: It was assumed that no gas was added to the combined or accumulated gas sample during the 11th, 12th, and 13th compressions. Thus the average pressures computed for single samples obtained from the 11th through 20th compressions would be increased over the corresponding values listed in Column 6. Column 7 lists the new, adjusted average pressures of single gas samples.

One can obtain a good estimate of the system pressure by taking a weighted average of the last five meaningful readings. The last five meaningful readings in this case are those values in Column 7 of Table III that correspond to the numbers n = 10, 14, 16, 18, and 20 of Column 1. Based on the assumption that no gas was effectively added to the combined

sample during the 11th, 12th, and 13th compressions, these values correspond to the following number of "effective" samples: 10, 11, 13, 15, and 17, respectively. A weighted average of the five single sample pressures mentioned above is obtained by multiplying each pressure value by the "effective" number of samples it represents, adding these products, and dividing by the total number of "effective" samples. When this is done, one obtains a weighted average single sample pressure of 9.73×10^{-7} mm Hg. The fractional standard deviation of the five single sample pressure values from the weighted mean is 2.44%.

Another method of finding a good estimate of the system pressure is to fit a straight line to the last four or five values of the average single sample pressure as a function of the number of compressions. The method of least squares can be used for this purpose. The slope of the straight line will be a measure of systematic errors that depend on the position of the mercury in the capillaries.

The adjusted average pressure values of a single gas sample, as taken from Column 7 of Table III, are plotted as a function of the effective number of compressions in Figure 9. One should note how the apparent pressure rises from a very small value for one compression, the result of an extreme systematic capillary error, to a much larger value that varies within rather narrow limits. The relative constancy of the pressure values beyond 10 compressions suggests that the systematic errors associated with the position of the mercury in the capillaries have been eliminated. The random errors that remain can be de-

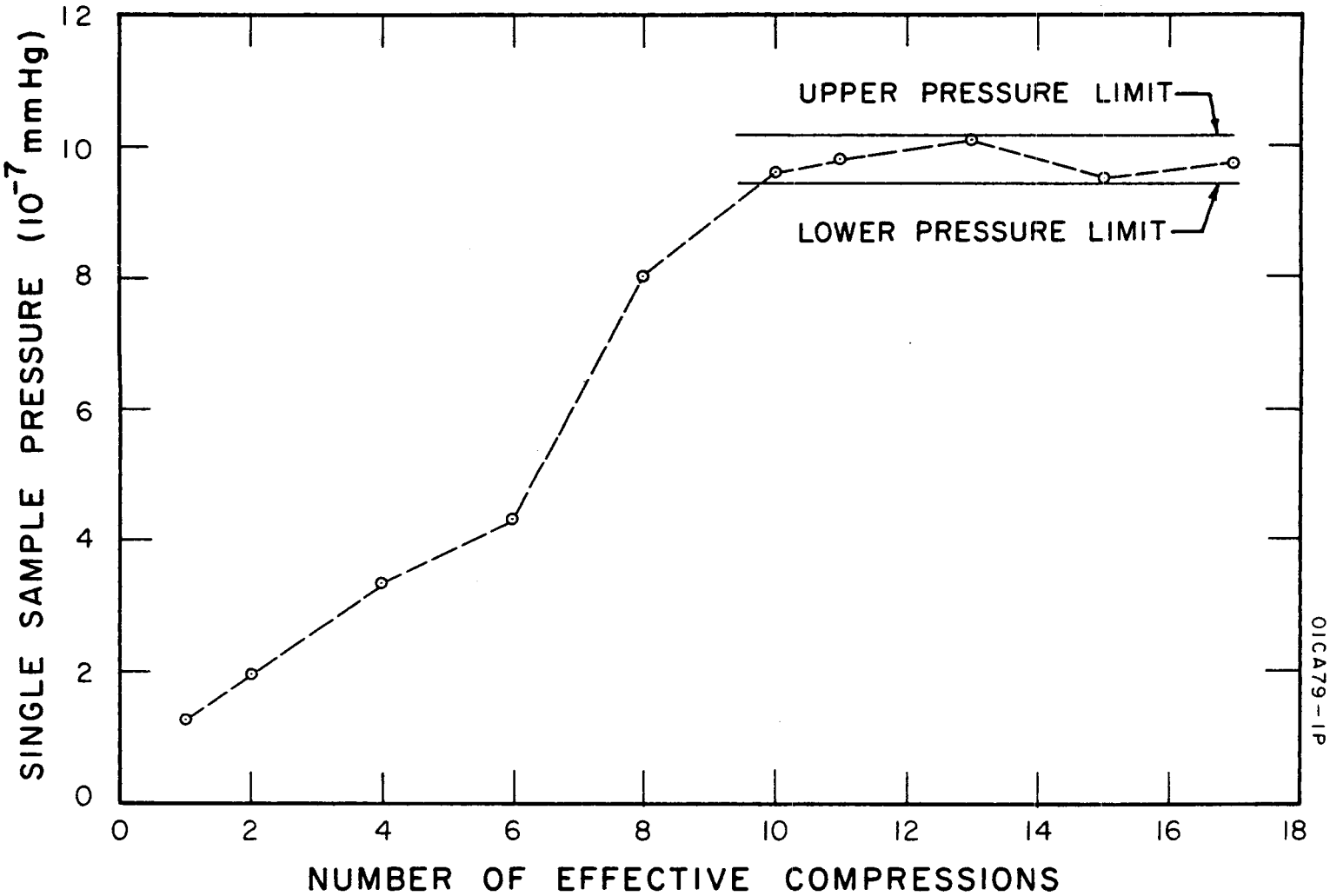

Figure 9. Single sample pressure values obtained with a multiple compressions McLeod gauge vs the number of compression.

termined and will furnish a measure of the precision of the average pressure.

If one studies Figure 9 rather closely, several interesting McLeod gauge characteristics emerge. For example, the full value of the pressure was not indicated by the gauge until about 10 compressions had been made. If we accept the correct value of the nitrogen pressure in the gauge to be a pressure of 9.73×10^{-7} mm Hg, the combined or accumulated pressure in the gauge after 10 compressions was 9.73×10^{-6} mm Hg. This value of pressure is evidently sufficiently large enough to be measured properly (with no large systematic error) with a single compression of the gauge being used. Further evidence of this fact derives from the results of the first experiment where the pressure of 8.85×10^{-6} mm Hg was measured with an error of only 7.5 per cent with a single compression. The data of Figure 9 lend substance to the assertion that any normal, "one compresssion" measurement made with a particular McLeod gauge should be viewed with suspicion if the gas pressure being measured is below a certain critical level. In the case of the gauge used in the experiments, this critical level corresponds to a pressure of about 1×10^{-5} mm Hg.

Another characteristic that is evident from the graph of Figure 9 is the rather sharp change in the slope of the curve after 6 compressions, at a combined sample pressure of about 5.84×10^{-6} mm Hg. This sharp change in behavior can be correlated with the sudden rise of the mercury in the closed capillary when it reaches a position at which the gauge would indicate this pressure in square law operation.

C. MEASUREMENT OF A GAS PRESSURE BELOW THE LOWER LIMIT OF A MCLEOD GAUGE.

The third experiment that was performed with the multiple compression McLeod gauge was the measurement of a pressure that was below the normal range of the gauge. The objective of this experiment was to determine the feasibility of making such a measurement and to determine those factors which could limit the usefulness of the multiple compression process at very low pressures.

The system was prepared for the measurement of a very low pressure by keeping it evacuated for about 2 weeks at the lowest pressure attainable without having liquid nitrogen in the spherical cold trap. The background pressure under these conditions was in the vicinity of 1.5 to 2×10^{-8} mm Hg as measured with the system Bayard Alpert type gauge at 10 ma. emission. At periodic intervals of about 2 hours, the mercury was raised rapidly into the gauge to create a turbulent flow within the compression bulb. The purpose of this operation was to outgas the mercury. In the light of later results, it is possible that some of the dry air used to raise the mercury may have been entrained by the mercury during this turbulent mercury motion.

Before the low pressure measurements were undertaken, the outgassing rate of the mercury was measured by filling the compression bulb about 2/3 full and leaving the mercury in this position to outgas overnight into the unfilled 700 cm^3 volume of the bulb. After a 19 hour

outgassing period, the mercury in the compression bulb was raised and a McLeod gauge reading was taken in the normal fashion. The gauge yielded a pressure reading of 3.5×10^{-6} mm Hg. This reading was based on a compression bulb volume of 2195 cm^3, whereas the actual volume of confined gas was only 700 cm^3. Thus, the actual pressure of the outgassing products in the 700 cm^3 unfilled bulb volume was $P = 2195 \times 3.5 \times 10^{-6} / 700 = 1.10 \times 10^{-5}$ mm Hg. Since the surface area of the outgassing mercury was 144 cm^2, the measured mercury outgassing rate was 7.81×10^{-13} mm Hg liters/cm^2/sec.

It had been planned to measure a pressure of approximately 9×10^{-8} mm Hg of nitrogen gas using a total of 80 compression cycles to yield a final accumulated gas sample pressure of 7.2×10^{-6} mm Hg. If the 80 compression cycles were to be made in a reasonable length of time, the time allotted for each compression cycle would have to be limited. Although approximately 30 minutes had been allowed per compression cycle in the first two experiments, it was decided to allow only 15 minutes for each compression cycle in the low pressure experiment. The 15 minute cycle consisted of a 5 minute sampling period, a 4 minute period for raising the mercury, a 2 minute period for combining and storing the gas samples, and a 4 minute period for lowering the mercury. In this way, 80 compressions could be made in two 10 hour measurement periods.

It was recognized rather early in the experiment that outgassing of the mercury during the 4 minute interval that the mercury was raised could introduce a large amount of extraneous gas into the nitrogen gas being sampled. A measure of this extraneous gas should be given by the

product of the mercury outgassing rate, the average mercury surface area during the compression (taken to be 150 cm^3), and the time required to raise the mercury. The result of this calculation showed that about 2.8×10^{-8} mm Hg liters of gas would be liberated from the mercury during each compression. This amount of extraneous gas should be compared with the amount of nitrogen gas in each sample, viz., 9×10^{-8} mm Hg x 2.2 liter = 2.0×10^{-7} mm Hg liter. The increase in the single sample pressure because of mercury outgassing could be expected to be approximately 14 per cent. Despite this relatively large error, it was believed that the measurements would be worthwhile, and so the experiment was started.

As with the other experiments, one of the first steps in making the measurements was to fill the spherical cold trap with liquid nitrogen and obtain the lowest possible background pressure before admitting the pure nitrogen gas to the system. A system background pressure value of 6.3×10^{-9} mm Hg was obtained as measured with the hot filament ionization gauge operated at 10 ma. emission. The nitrogen gas bottle valve was then opened until the system pressure rose to about 9×10^{-8} mm Hg (at 10 ma. emission). When the ionization gauge emission was reduced to 100 microamps, the indicated system pressure rose to a value of 1.2×10^{-7} mm Hg showing that the ionization gauge had been pumping very strongly. A Keithley model 600 electrometer was used to measure the small ion current output of the ionization gauge.

About 1 hour after the nitrogen leak was opened, the system pressure was 9.0×10^{-8} mm Hg and decreasing very slowly. It was decided to begin

the 80 compression measurements at this point and take a pressure reading after every 10 compressions. The first compression gave a pressure reading that was identically zero and bolstered the belief that the gauge was measuring a pressure below its normal limit. After the 10th compression had been made, a second reading of the accumulated gas sample pressure was made. The pressure measured at this time was 1.48 x 10^{-6} mm Hg. Such a pressure was obviously much higher than the expected pressure of 9 x 10^{-7} mm Hg or less. Another 10 compressions were then made, and the pressure of the 20 gas samples collected was found to be 5.05 x 10^{-6} mm Hg, corresponding to an average single sample pressure of 2.52 x 10^{-7} mm Hg instead of the 9 x 10^{-8} mm Hg nitrogen pressure expected. After 10 more compressions the accumulated pressure for 30 gas samples was found to be 7.27 x 10^{-6} mm Hg corresponding to 2.42 x 10^{-7} mm Hg for an average single sample pressure. At this point, it was decided to end the experiment because the readings for the 20 and the 30 accumulated gas samples indicated that the single sample pressure of 2.42 x 10^{-7} mm Hg would not change very much, although the trend appeared to be to lower values.

There are at least two plausible explanations for the unusually high pressures found in this experiment. The first explanation is that an insufficient period of time was allowed for "conditioning" the McLeod gauge at the nitrogen gas pressure of 9 x 10^{-8} mm Hg. Actually, only about 1 hour was allowed for this process, whereas past experience had indicated that at least two or three hours were necessary to obtain adsorption-desorption equilibrium at this pressure. Also, the gauge had

been exposed to a pressure of 1.10×10^{-5} mm Hg of outgassing products for a long period of time less than 2 hours before the multiple compressions were started. It is possible that both the closed end capillary and the compression bulb were still desorbing outgassing products during the 30 compressions made.

A second explanation for the large amount of gas obtained in the 30 compressions is that the mercury outgassed much more than was computed on the basis of a static outgassing test. When the mercury was raised in the McLeod gauge at the speed required to fill the gauge in 4 minutes, it was observed that the mercury motion in the gauge was turbulent. This very turbulent motion, in a more extreme form, had been used earlier to outgas the mercury. It is reasonable to assume that the outgassing rate for the moving mercury is much greater than that measured for the quiescent mercury.

The results of the low pressure experiment outlined above begin to show some of the limitations of the multiple compression McLeod gauge technique for measuring pressures in an absolute fashion. Adsorption-desorption equilibria and gas free mercury are required when making pressure measurements below the normal limit of the gauge.

D. ADSORPTION OF NITROGEN GAS IN THE CLOSED END CAPILLARY OF A MCLEOD GAUGE.

After the 30 compressions of nitrogen gas had been made in the third experiment just described, the accumulated sample pressure of 7.27×10^{-6} mm Hg was stored in the closed end capillary for 35 hours.

At the end of this storage period, and with the system pressure constant at 8.40×10^{-8} mm Hg of nitrogen as measured with the ionization gauge operating at 100 microamps, a 31st sample was added to the previously stored gas. The total pressure of the 31 samples was found to be identical with that of the 30 samples measured earlier, within the experimental error involved in reading the mercury positions. It will be recalled that results of this kind had been obtained earlier. It appears that the pressure of the new sample of gas was not manifested while the old pressure remained unchanged.

Based on the experimental results obtained, one can evidently store samples of gas in a small bore capillary of the type used in McLeod gauges (the capillary used in these experiments had a diameter of 0.535 mm and a length of about 21 cm) for long periods of time without any apparent change in the gas pressure. Thus it can be said that there is no permanent, long term adsorption or absorption in the sense of a chemical gettering action or diffusion of the gas into the glass wall or mercury. Such behavior, of course, is essential for the proper operation of a multiple compression McLeod gauge.

The inability to measure the pressure of a new gas sample that is added to a sample that has been stored for a long period of time is something of a mystery. If we may be permitted to speculate, it seems as though the new gas sample is temporarily adsorbed on the walls of the glass or mercury.

To continue with our speculation, temporary adsorption could also account for the results of the second experiment where it was shown that the full pressure of the accumulated gas samples only manifested itself slowly, after 10 compressions and an interval of time of about 5 hours. However, there are other phenomena that can account for the abnormally low pressure values that are usually obtained when a McLeod gauge is used near its lower limit of operation. For example, a decreased capillary depression due to the presence of a gas phase above the mercury meniscus, as found by Nottingham , would also account for the apparent lowering of the pressure.

It would be best to end this discussion on a note of caution. Adsorption-desorption effects are probably important in the operation of McLeod gauges at low pressures. The need for "conditioning" a gauge at the pressure to be measured to insure an adsorption-desorption equilibrium is an example of this. Sudden changes in the gas pressure (at low pressures) would tend to upset the adsorption-desorption equilibrium and could cause temporary adsorption or desorption of gas.

VI. SUMMARY AND CONCLUSIONS

The concept of using a McLeod gauge to accumulate a series of samples of the gas being measured is introduced. The idea is first examined from a theoretical standpoint, and it is shown that the equations describing the operation of the gauge are changed in only one respect: The volume of the compression bulb and closed capillary V is replaced by a term $V + (n - 1)V'$, where n is the number of gas samples collected and V' represents the volume of the compression bulb below the gas storage valve. In practical designs of a multiple compression gauge, the volumes V' and V differ by only a few tenths of a per cent, and so one can say that the "effective" compression bulb volume of a well-designed gauge used to accumulate n samples of gas is just n times greater than the actual bulb volume. In other words, the gauge becomes n times more sensitive. Thus, if a McLeod gauge used in the ordinary, single compression fashion can be expected to yield 1 per cent accuracy at a pressure P, the same gauge used in multiple compression fashion should yield a measurement of this accuracy at a lower pressure p/n.

In order to construct a practical multiple compression McLeod gauge, it is necessary to install a valve in the gauge between the top of the compression bulb and the bottom of the closed end capillary. Four types of valve mechanisms or gas storage techniques were studied experimentally. These were the float valve, the electromagnetic mercury valve, the magnetic sphere valve, and the mercury freezing technique. The relative

merits of the various devices were compared, and it was decided that the simplest, fastest, and most reliable device was the magnetic sphere valve. The latter valve consists of a small sphere of magnetic material (a 3/32 inch diameter ball bearing made of type 440 stainless steel, for example) that is placed inside the compression bulb and is moved by means of external permanent magnets or electromagnets. When gas samples are to be stored in the closed end capillary, a short column or pellet of mercury about 10 mm long is isolated and suspended at the base of the capillary by closing off the entrance to the capillary with the magnetic sphere. The short column of mercury actually provides the gas-tight seal. When a magnetic sphere is used in this fashion, there is no need to modify the McLeod gauge, and, further, the gauge can be used in its normal, single compression fashion.

A low pressure McLeod gauge can be used properly only if it is a part of a well-designed low pressure pumping system. A description is given of the low pressure system that was constructed for the purpose of testing the characteristics and utility of a low pressure multiple compression McLeod gauge. The system was basically an all glass, mercury diffusion pump system that was capable of attaining base pressures as low as 6×10^{-9} mm Hg with a minimum of bakeout. No waxed or greased joints were used in the construction, and no valve was interposed between the McLeod gauge and the rest of the system. A symmetrical H-Type liquid nitrogen cooled trap was placed between the McLeod gauge and the system ionization gauge to permit ionization gauge readings to be made. The McLeod gauge used was a Consolidated Vacuum Corporation

model GM-110 which has a nominal gauge constant of 1×10^{-7} mm Hg/mm^2 and a smallest scale division of 1×10^{-7} mm Hg. A magnetic sphere valve was used to permit multiple compressions to be made. Pure reagent grade nitrogen gas was flowed into the diffusion pump to establish various pressures of nitrogen in the system.

The last section of this report discusses some experiments that were perfomed with the low pressure multiple compression McLeod gauge. In general, it is shown how the multiple compression technique leads to more precise measurements and a minimization of systematic errors. The first careful experiment performed with a multiple compression McLeod gauge showed that a nominal pressure of 9×10^{-6} mm Hg, which is well within the normal range of the gauge used, could be measured much more precisely by using five compressions than by just taking five readings of a single compression measurement. When multiple compressions are made, a large sample of gas is observed and the relative errors become smaller. In the experiment just mentioned, the average pressure value obtained when five compressions were made was a pressure of 8.85×10^{-6} mm Hg with a fractional standard deviation of 0.63 per cent. The average pressure value obtained for a single compression was a pressure of 8.18×10^{-6} mm Hg with a fractional standard deviation of 2.99 per cent. The difference in the two pressure readings is believed to be due to the presence of a systematic error which is associated with the position of the mercury in the capillaries. The systematic error gets smaller as the amount of gas in the closed end capillary gets larger.

A second experiment was performed to measure a gas pressure near the lower limit of the gauge measuring capability. Twenty compressions were made of nitrogen gas at a nominal pressure of 9×10^{-7} mm Hg. Pressure readings were taken after every other compression. The results of these measurements showed very graphically that a very large systematic error was present in the gauge at this low pressure level. The pressure readings obtained were far below the correct value until 1[illegible] gas samples were accumulated to give a total combined sample pressure of about 9.5×10^{-6} mm Hg. As additional samples of gas were added to the total, the systematic change in the pressure of a single sample vanished, and the variations in the single sample pressur[illegible] became purely random and decreasing. A weighte[illegible] average value of the system pressure was found to be 9.73 [illegible] 10^{-7} mm Hg with a fractional standard deviation (coefficient of variation) of 2.44 per cent. It is possible that this is the most precise McLeod gauge measurement made to date of a pressure of this magnitude.

A statement concerning the <u>absolute</u> accuracy of multiple compression McLeod gauge measurements must await a closer study of many potential sources of systematic error as well as the interaction of the gauge and the system in which it is being used. The presence of condensable gases, the absorption of certain gases in the mercury [illegible] thermal transpiration effects, and mercury vapor stream pumping effects must be given careful consideration. The use of a multiple compression gauge, however, with its smaller random error and extended range of operation, should make it easier to detect most of these systematic errors.

A third experiment that was carried out with the multiple compression technique was the measurement of a gas pressure below the normal range of the gauge. This sort of measurement, of course, is completely impossible with a normal McLeod gauge. A gas pressure of about 9×10^{-8} mm Hg of nitrogen was established in the system and 30 compression samples of this gas were accumulated in the McLeod gauge. It was found that at this low a pressure level, outgassing from the mercury and possible desorption of gas from the walls of the gauge contributed approximately twice as much gas to the total pressure as the nitrogen. Because this experiment was not performed under the best possible conditions, it is believed that additional experiments of this type will show that pressures below the normal range of a McLeod gauge can be measured with the multiple compression technique. Although the primary objective of the experiment, measuring a nitrogen pressure of 9×10^{-8} mm Hg, was not achieved, the experiment did show the importance of using gas-free mercury and allowing sufficient time for adsorption-desorption equilibrium to be established.

In the course of the experimental work already discussed, a number of tests were made to determine whether or not accumulated samples of gas could be stored unchanged in the closed end capillary of a McLeod gauge. The length of the storage periods varied from 14 hours to 42.5 hours. It was found in every case that the stored gas remained unchanged. However, it was observed that when small, new samples of gas were added to the stored samples, there was no measurable increase in the total gas pressure immediately after the addition. It is suggested that temporary adsorption of the new gas sample was responsible for this result.

ACKNOWLEDGEMENT

I would like to express my thanks to Mr. Paul R. Yeager and Mr. Richard Stell of the Instruments Research Division of NASA Langley Research Center whose support made this work possible. It is a pleasure to acknowledge the contributions of Mr. Arthur Suprenard who constructed and modified the McLeod gauges and vacuum systems and Mr. Frank Robert who built the magnetic sphere positioning devices.

REFERENCES

1. Final Report for the Period 11 February 1959 to 11 February 1960, Contract NASW-25(Task I), pp. 43-49. Research performed for Goddard Space Flight Center, NASA, by the Geophysics Corporation of America, Bedford, Massachusetts.

2. Ishii, H. and Nakayama, K., Transactions of the Eighth Vacuum Symposium and Second International Congress, Pergamon Press, New York, 519 (1961).

3. Rusch, M. and Bunge, O., Z. Tech. Phys. 13, 77 (1932).

4. Groszkowski, J., Le Vide 4, 668 (1949).

5. Nottingham, W. B. and Blackford, B., Report on the Twenty-Second Annual Conference on Physical Electronics, Massachusetts Institute of Technology, Cambridge, Massachusetts, 351 (1962).

6. Podgurski, H. H. and Davis, F. N., Vacuum 10, 377 (1960).