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TO Dr. F. W. Hurd
LOCATION Laboratory Division

DATE December 16, 1953

ANSWERING LETTER DATE

ATTENTION
COPY TO Barton, J. C.

SUBJECT A McLEOD PRIMARY PRESSURE
STANDARD

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Plans have been made to study some aspects of flow through porous media at low pressures. Such pressures must be measured with devices that require calibration with a direct pressure measuring standard. The construction of such a standard has been completed and is described in this report.

Description of Calibration Vacuum System

A McLeod gauge has been designed to measure, within an accuracy of better than one per cent, pressures from about five to several hundred microns of mercury (5 to ca. 300×10^{-4} cm. Hg). The total system (see figure 1) consists of the McLeod gauge (MG) and a five liter buffer volume (BV) connected through a stop cock (S_{21}) and liquid nitrogen trap (T_1) to a pressure manifold (M) to which one may connect other pressure measuring devices. This manifold, by way of another liquid nitrogen trap (T_2) and a mercury cut-off valve (CO) leads to an oil diffusion pump (DP) and a Welsh fore pump (WP_1). Air or other gas may be admitted through a stop cock (S_{22}) and a 50 micron capillary (C) between the cut-off (CO) and the last liquid nitrogen trap (T_2).

Calibration of the McLeod Gauge

An 84 cm. length of 1 mm. I.D. precision bore capillary tubing was cleaned with hot tri-sodium phosphate cleaning solution and marked off in 5 cm. sections.

A thread of mercury, approximately 5 cm. long, was positioned in each of the seventeen sections. At each position, its length was carefully measured with a travelling microscope. On the basis of this uniformity survey, a 20 cm. and a 40 cm. piece were cut for eventual use as the closed and side arm capillaries, respectively, of a McLeod Gauge.

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MG McLeod Gauge
 BV Buffer Volume
 T Liquid Nitrogen Traps
 TG Thermocouple Gauge
 IG Ion Gauge
 DP Oil Diffusion Pump
 WP Welsh Fore Pump
 S2 Two-way Stop Cocks
 S3 Three-way Stop Cocks

N Nitrogen Tank
 R Mercury Tube Pressure Regulator
 C Capillary Tube Controlled Leak
 F Flasks for Pressure - Vacuum Control of Mercury Levels
 K Kerotest Valve
 M Manifold
 CO Cut-off

FIGURE 1

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The 20 centimeter capillary was further calibrated by measuring the length (l) of five different threads of mercury each at various positions along the capillary. This procedure was used to obtain a mean cross-sectional area corrected for the effects of a mercury meniscus at each end of the thread. That the technique accomplishes this can be seen by the following short analysis.

If v is the volume of a mercury thread (determined by weighing) and Δv is the difference between the total capillary volume included between the measured ends of the mercury thread and the volume v , then:

$$v + \Delta v = \bar{a} l$$

where $\bar{a}$ is the mean cross-sectional area of the capillary and l is the measured length of the thread.

Solving the expression for v/l , one obtains:

$$\frac{v}{l} = \bar{a} - \frac{\Delta v}{l}$$

Thus a plot of v/l versus $1/l$ gives a straight line whose intercept is $\bar{a}$ and whose slope gives $-\Delta v$. Note that Δv may be useful as an estimate of twice the meniscus correction for a 1 mm. capillary.

Thus, the mean cross-section of the 20 centimeter capillary tube was found to be as follows.

$$\begin{aligned} \bar{a} &= 0.007692 \text{ cm.}^2 \\ \sigma_{\bar{a}} &\pm 0.000004 \\ 95\% \text{ C.I. } &\pm 0.000008 \end{aligned}$$

The laboratory glass shop then constructed a McLeod gauge (see figure 2) with a trapped volume (V_1) of about 150 cm.³ and using a 3 mm. I.D. re-entrant tube (R) with a square ground edge for a very sharply defined cut-off. Microscopic reference marks (B) were cut with a diamond point around the capillaries at the positions indicated in figure 2. These marks, and especially the one near the end of the closed capillary, were for use as base positions for careful cathetometer measurements.

Before sealing off the ends of the measuring capillary (CC), the volume enclosed between the bottom end of the re-entrant tube (R) and the mark (B) on (CC) was determined by filling with mercury and weighing. Five such measurements were made.

Thus it was found that the trapped volume (V_1) at 25° C. can be represented by:

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- SC Side Arm Capillary
 CC Closed Capillary or Measured Capillary
 R Re-entrant Tube about 3 mm. I.D. and 1 cm. long
 S₃ Three Way Stop Cock
 F Flask For Pressure - Vacuum Control of Mercury Level
 B Reference Marks

FIGURE 2

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$$V_1 = 151.030 \text{ cm.}^3 + \delta v$$

$$\sigma_{V_1} \pm 0.0015$$

$$95\% \text{ C.I. } \pm 0.0042$$

where δv is the eventual volume contained from the mark (B) to the sealed end together with the meniscus correction.

An additional uncertainty involving the volume of the mercury meniscus hanging from the re-entrant tube (R) is conservatively estimated at less than $\pm 0.010 \text{ cm.}^3$

The end of the measuring capillary was now sealed off and the gauge as a whole incorporated in the vacuum system. Mercury was introduced into the gauge by distillation under a pressure of less than five microns of mercury. Nitrogen pressure is used to raise the mercury level in the gauge, and further, it is expected that oxygen at pressures of greater than 0.05 cm. Hg will never come in contact with mercury in the McLeod gauge.

The volume, (δv), above the mark (B) on the closed capillary (CC) and including the meniscus was evaluated by measuring a series of pressure-volume products in the McLeod gauge for each of four system pressure levels.*

System pressure levels (P_1) were estimated by using a National Research Corporation thermocouple gauge. Pressure-volume products were determined by measuring with a cathetometer h_1 (see figure 2) and Δh , the difference between the mercury levels in the closed (CC) and side arm (SC) capillaries.

If all lengths and pressures are expressed in centimeters and centimeters of mercury, respectively, and if

Δh = difference between mercury levels in the closed (CC) and side arm (SC) capillaries

$$P_2 = P_1 + \Delta h$$

V = volume enclosed between the reference mark (B) on the closed capillary (CC) and the top of the meniscus of the column of mercury in the closed capillary

V_2 = volume enclosed between the top of the closed capillary and the top surface of the mercury column in (CC)

V_1 = total volume trapped between the end of the re-entrant tube (R) to the top of the capillary (CC).

* This technique followed the suggestions of Clark, R. J., "A Method of Calibration of a McLeod Gauge", J. Scientific Instruments, 5, 126, (1928).

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Then:

$$V_2 = V + \delta v$$

and hence

$$P_2 V = P_2 V_2 - P_2 \delta v$$

where $P_2 V_2$ is a constant for a given system pressure level assuming the ideal gas law.

A plot of $P_2 V$ versus P_2 gives a straight line with intercept $P_2 V_2$ and slope $-\delta v$. This scheme was used at system pressure levels of approximately 2, 7, 20, and 100 microns of mercury. The resulting values of δv were weighted inversely according to their variances and the results were,

$$\text{weighted mean } \delta v = 0.003193 \text{ cm.}^3$$

$$\sigma_v \pm 0.000034$$

$$95\% \text{ C.I. } \delta v \pm 0.000068$$

where the uncertainties were based on the minimum variance.

It is convenient to convert this δv into an effective length, K , which must be added to the distance, h_1 , such that the uniform cross-section, $\bar{a}$, multiplied by $(h_1 + K)$ gives the true volume, V_2 , of gas contained above the mercury column in capillary, CC.

Thus

$$K = \delta v / \bar{a} = 0.415 \text{ centimeters}$$

$$\left. \begin{array}{l} \sigma_K \pm 0.004 \\ 95\% \text{ C.I. } K \pm 0.009 \end{array} \right\} \text{ assuming } \bar{a} \text{ is known exactly}$$

The final calibration may be expressed in terms of system pressure, P_1 , as

$$P_1 = \frac{P_2 V_2}{V_1} = \frac{P_2 (h_1 + K) \bar{a}}{V_1} = P_2 (h_1 + 0.415) \frac{0.007692}{151.033} \text{ cm. Hg}$$

The original thermocouple gauge readings used in the estimates of system pressure, P_1 , have been compared to the McLeod pressures (as determined by the final calibration) and the results are given in table I and figure 3. Figure 3 also includes the manufacturer's calibration for the thermocouple gauge.

Table I also indicates the precision obtainable in a measurement of P_1 and the number of readings required for a precision of $\pm 1.0\%$.

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TABLE I
CALIBRATION AND PRECISION SUMMARY FOR DRY AIR

Thermocouple Reading micro amps.	$\bar{P}_1$ microns, Hg	$\sigma_{\bar{P}_1}^2$	$\sigma_{\bar{P}_1}$	Number of Readings	Readings Required S_o $\sigma_{\bar{P}_1} = 1.0\% \text{ of } \bar{P}_1$
198	2.293	0.001922	0.044	14	51.2
174	7.322	0.000711	0.027	14	1.85
136	20.95	0.01360	0.12	14	4.34
64	101.01	0.2023	0.45	17	3.37

Recommended Procedure

It was found that the absolute accuracy of measurements with the McLeod gauge was primarily limited by the precision of reproducing a reading after trapping a sample of gas. Thus we can expect to know the P_1 values above five microns of mercury within a standard deviation of less than 1% of the P_1 value when at least five readings are taken at each pressure level. If greater scatter is observed the measurement should be repeated.

Experience showed that maximum precision and accuracy may be expected when the mercury level in the side arm capillary (SC) was set five centimeters or more above the closed capillary mark.

Experience suggested that the following procedure should be followed for the purpose of standardizing other instruments by means of the McLeod gauge.

1. Bring the mercury level in the side arm capillary (SC) to about 6 centimeters above the closed capillary mark (B).
2. Tap the capillaries near the mercury levels gently with a rubber tube.
3. Set cathetometer on side arm capillary mercury level.
4. Tap the capillaries as before and then read the cathetometer within ten seconds after tapping. Record the result.
5. Read and record the level of mercury in the closed capillary (CC).
6. Repeat steps 1 through 5 with the mercury level in the side arm capillary (SC) successively at 8, 10, 8, and 6 centimeters above the closed capillary mark (B).
7. Take five readings on the capillary (CC) reference mark (B) sometime during the course of any given work day or shift.

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Micro Amperes on Thermocouple Gauge
 FIGURE 3

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Summary

A McLeod gauge pressure standard, for the approximate range of 5 - 300 microns of mercury, has been constructed and calibrated. With proper precautions, pressure measurements in this range can be made with a precision of better than $\pm 1\%$.

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