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 POST OFFICE BOX P
 OAK RIDGE, TENNESSEE

 ORNL
 CENTRAL FILES NUMBER

CF-56-2-168

DATE: February 15, 1956

 SUBJECT: Pressure Measurement by Displacement
 of a Restrained Diaphragm

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COPY NO. 18

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PRESSURE MEASUREMENT BY DISPLACEMENT OF A RESTRAINED DIAPHRAGM

Introduction

The measurement of the pressure of systems at high temperature remains a difficult art. The use of mercury pistons is limited to cases where chemical reaction is negligible. Other fluid pressure transmitters, such as water, lead to uncertainties in volume and composition. By means of the balanced diaphragm, a closed system and high accuracy can be obtained. However, the equipment required to maintain pressure balance at high temperature and pressure is complicated and expensive.

A relatively simple method for determining the pressure of enclosed systems by the displacement of a restrained diaphragm has been developed. This method was used to measure unsaturated steam pressures in connection with volumetric water adsorption studies,⁽¹⁾ and should be applicable to other systems.

In principle, a flexible diaphragm is mounted between two restraining surfaces and can be moved freely from one surface to the other. A thermostated, enclosed system is connected through a small, central hole to the inner side of the diaphragm. The outer side of the diaphragm is connected through a similar small hole with an external system filled with mercury or other liquid of relatively low vapor pressure. The diaphragm is normally forced against the outer restraining surface by the pressure of the enclosed system. However, when the external volume is diminished by small increments, the external liquid pressure rises until it equals

(1) Richardson, D. M., "Adsorption of H_2O by ThO_2 at High Temperatures", ORNL CF-56-1-109.

the pressure of the enclosed system, and the diaphragm is then displaced at constant pressure toward the inner restraining surface. When the latter is reached, the external liquid pressure again rises. Thus, with an ideal diaphragm, the enclosed system pressure is indicated by the pressure plateau of the external system.

The spring constant of a practical diaphragm causes tilting and raising of the ideal plateau. However, a sharp rise in pressure does occur when the inner restraining surface has been reached, and the pressure difference required to displace the diaphragm completely can be calibrated.

A further consideration, in a practical case, is the decrease in volume of the enclosed system as the diaphragm moves inward. Since the diaphragm volume displacement may be made small relative to the system volume, and since the latter must be known only when the diaphragm is at its inner limit, no significant limitation to accuracy is imposed by this effect.

Experimental Details

The shallow, bell-shaped diaphragm was formed from .005-inch 18-8 SS sheet. The end cap of the pressure vessel (outer restraining surface) was used as the forming die, the diaphragm being struck under a 0.25-inch thick rubber block in a hydraulic press. The profile used, not including gasket V-grooves, is specified in Table 1 and is shown in Figure 1. The effective diaphragm diameter was one inch. At maximum movement the diaphragm traveled 1/32 of an inch and displaced a volume of 0.25 milliliters.

In early trials it was found that a simple bell-shaped diaphragm did not displace uniformly and, in fact, made a very good "cricket", snapping sharply from concave to convex. Such performance caused saw-toothed plateaus of poor precision. This difficulty was corrected by adding concentric corrugations, as specified in Table 1, and by changing the inner restraining surface (end of pressure vessel) to a flat. The corrugations reduced stress localization and made smooth, symmetrical displacement possible. Similar patterns are used in aneroid barometers.

The assembled diaphragm, end cap and pressure vessel were designed to be leak-tight against three fluids: mercury in the external system, water in the internal system, and molten salt in the thermostat bath. The matching V-groove and "O"-ring assembly used is shown in Figures 1 and 2. A soft gold "O"-ring was used for the inner, high-pressure seal, the end cap being tightened until the diaphragm margin was firmly pressed between the flat surfaces of the end cap and the pressure vessel. No leaks occurred in this assembly during the experiments.

The SS 347 pressure vessel was made from a single-end, heavy wall, commercial model by reborring, and by remachining the bottom end for the diaphragm assembly and standard screw cap. A special SS 347 end cap for the top end of the pressure vessel was used which contained a high pressure valve. This valve permitted evacuation and subsequent vacuum distillation of water into the pressure vessel, and final closure of the pressure vessel without loss of vacuum. This end cap is shown in Figure 3. A flat gold ring gasket in a flat retaining groove was used. The end

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cap was tightened until the steel surfaces touched each other in order to minimize the enclosed volume uncertainty.

The normal method of controlled volume displacement requires a calibrated piston. Since a quantitative measure of volume change is not necessary in the present case, a piston substitute was used. This consisted of a separate pressure vessel filled with mercury. When electrical heating was applied to this vessel, a smooth and, for practical purposes, linear rise in pressure with time occurred. This vessel was connected to the external system filled with mercury up to the diaphragm, and produced a very satisfactory piston action.

A third pressure vessel served as a mercury reservoir. This vessel and the other essential system components are shown schematically in Figure 4. By applying nitrogen gas under pressure to the reservoir, and opening the outlet valve, the mercury system could be initially pressurized without heating the "piston". In addition, when the outlet valve was operated as a needle, the resulting slow rise in pressure of the mercury system served as an additional piston device and was useful for finding the plateau. Once the plateau had been found in this way, the system pressure was adjusted about 50 psi lower, and the heated piston was then operated to obtain a more precise measurement.

The fluid pressure in the external system was measured with a Baldwin SR-4 (four element strain gage) pressure cell which had been specially selected for linearity and reproducibility within 0.1% full scale (2000 psi). Both input and output voltages of the pressure cell were

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measured with a Rubicon precision potentiometer. The pressure cell and input calibration shunt were thermostated at 35°C. A second pressure cell, not shown in Figure 4, was used. This cell was connected to a strip chart recorder which provided a graphic record of the pressure plateau. This record was particularly useful in the initial operation of finding the plateau and adjusting the mercury system pressure preparatory to slow scanning with the heated "piston".

The required precision in temperature was obtained by use of a well-stirred molten salt bath containing approximately 200 pounds of KNO_3 and NaNO_2 (60-40) with melting point of about 145°C.⁽²⁾ Temperature control within .01°C was obtained by means of an Electronotherm (a.c. resistance bridge, on-off controller) whose amplifier output was shunted to the galvanometer of a Pyrovane controller for greater precision. The precision of temperature selection of the standard Electronotherm unit was improved by means of an external bandspreading device. Temperatures were measured with a platinum resistance thermometer, N. B. S. certificated, and a Leeds and Northrup Muehler Bridge. Thus, readings were made to .001°C with an accuracy of $\pm .02^\circ\text{C}$.

Interpretation of Data

The first case which will be discussed is that in which each pressure plateau measurement must be evaluated separately as an absolute pressure. Later the simpler case will be presented, with actual data,

(2) Lyman, Taylor (Editor), Metals Handbook, 1948 Edition, The American Society for Metals, Cleveland.

in which unsaturated steam pressures at high temperatures are evaluated relative to a saturated steam pressure measured at a lower temperature.

The static heads of mercury in the external system are indicated in Figure 4; the reference pressure chosen for convenience was atmospheric. The following symbols will be used:

G = gage pressure reading

g = gage zero correction, to absolute zero pressure

P_{atm} = atmospheric pressure, absolute

$(hdg)_1$ = mercury head above gage, at temperature T_1

$(hdg)_2$ = mercury head from gage to thermostat, at temperature T_2

$(hdg)_3$ = mercury head within thermostat, at temperature T_3

K = pressure difference required to displace diaphragm from the outer to the inner restraining surface

P = absolute pressure of enclosed (internal) system.

It will be assumed that the gage zero error is significant and that its effect is additive on a consecutive gage pressure reading. Before each pressure plateau measurement, therefore, the gage zero correction should be determined. The gage pressure is read with the valve to the mercury leveling bulb open. Then

$$g = G' - \left[P_{atm} + (hdg)_1 \right] .$$

From the gage pressure reading at the pressure plateau, finally, the absolute pressure of the enclosed system is calculated as follows:

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$$P = [G - g] + [(hdg)_2 + (hdg)_3] - K.$$

The value of K must be determined separately. In these experiments K was found to be 5 psi, and remained constant. At extreme temperatures or with severe corrosion, however, K would be expected to change.

The absolute error, using this method of evaluation, arises primarily from the difficulty in assigning an effective elevation to the pressure gage, and from the difficulty in estimating the integral mercury head where there is a temperature gradient. The former problem can be eliminated by means of horizontal mounting of the pressure gage (however an internal venting tube would be required to allow all gas to escape). The latter problem is conventionally met by means of cooling jackets and thermal barriers so that the limits of the temperature gradient can be more accurately located.

An interesting run was made for calibration purposes in which only water, which had been deaerated by repeated freezing and thawing at less than 1 micron pressure, was present in the enclosed (internal) system. Pressure plateau measurements were made at 380.00°F (193.33°C) and at 60°F intervals up to 680.00°F (360.00°C). These temperatures were convenient since published values for the pressure and specific volume of unsaturated steam were available.⁽³⁾ Thus for a given temperature, a plot of

(3) Keenan, Joseph H. and Keyes, Frederick G., Thermodynamic Properties of Steam Including Data for the Liquid and Solid Phases, John Wiley and Sons, Inc., New York.

pressure against specific volume was made and from the resulting smooth curve a particular specific volume was obtained.

The pressure plateau obtained at 193.33°C was typical and is shown in Figure 5. The system was saturated at this temperature and should have a pressure of 195.8 psia.⁽⁴⁾ The gage "zero" in this case is the gage reading at an arbitrary fixed value of $[P_{\text{atm}} + (\text{hdg})_1]$. However, the value of K and the magnitude of the various mercury heads are not given. Thus the absolute pressure of 195.8 psi can not be calculated from the data of Figure 5. However, other plateau measurements with the same diaphragm assembly can be referred to this one at 195.8 psia, provided their "zero" is obtained at the same value of $[P_{\text{atm}} + (\text{hdg})_1]$.

The data of this run are shown in Table 2, and the measurements at temperatures above 193.33 are evaluated relative to that temperature. At the lower two temperatures the system was saturated and the calculated pressures are in error relative to saturated steam values by less than 1 psi. At the three higher temperatures the system was unsaturated and is compared in Table 2 with the published values for unsaturated steam.⁽⁵⁾ The agreement is better than 0.1%. Plateau measurements were made each day for five days at 360.00°C . The pressure remained constant within 1 psi.

The value of $(\text{hdg})_3$ changes with the thermostat temperature and should, strictly, be included as a correction at the higher tempera-

(4) and (5) Ibid.

tures. However, the magnitude of this correction was estimated and, at 360°C, would be minus 0.2 psi. Since this is not significant, it was not included in Table 2.

It is of interest to note that plateau measurements can be made at temperatures higher than the boiling point of mercury (or other fluid) provided the system pressure being measured is greater than that of saturated mercury vapor at the same temperature.

The final practical observation should be made that, with suitable valving, a large number of diaphragm and pressure vessel assemblies can be operated with the same external system. To avoid overstressing of the diaphragms displaced against the lower pressures, however, it would probably be desirable to operate the diaphragms one at a time.

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TABLE 1. DIAPHRAGM SUPPORT PROFILE

(Stepwise cuts to obtain smooth surface upon emery polishing)

<u>Radial Distance from Center Inches</u>	<u>Depth of Cut Inches</u>
0 to 1/6	.03125
1/16 to 1/8	.028
1/8 to 3/16	.024
3/16 to 1/4	.019
1/4 to 5/16	.012
5/16 to 3/8	.007
3/8 to 7/16	.003
7/16 to 1/2	0
1/2 to edge of flange	0

Diaphragm Support Corrugations

1/32-inch diameter grooves, 1/64-inch deep, concentric at radii of 3/16, 5/16, and 7/16 inch; radius edges and polish with emery cloth.

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TABLE 2. VAPOR PRESSURES OF SATURATED AND UNSATURATED STEAM, BY RESTRAINED DIAPHRAGM

(6.8274 grams H₂O in a SS347 vessel of volume 193.34 ml at 25°C)

Temperature (°C)	"Zero" (microvolts)	Plateau (microvolts)	Plateau Minus "Zero"	Column Minus Reference*	Column 5 (psi) ^a	Total Pressure (psia) ^b	Density of Dry Steam (g/ml) ^c	Volume at Temperature (ml) ^d	Calculated Grams of H ₂ O Vapor	Per Cent of Actual H ₂ O Added
193.33	72.7	546	473.3*	0	0	195.8 (saturated)		195.0		
226.67	73.8	1103	1029.2	555.9	185.2	381.0 (381.59 saturated)		195.3		
260.00	73.8	2005	1931.2	1457.9	485.7	681.5 (680.8 saturated)		195.7		
293.33	73.5	2995	2921.5	2448.2	815.5	1011.3	0.03486	196.0	6.8326	100.08
326.67	73.4	3335	3261.6	2788.3	928.8	1124.6	0.03479	196.3	6.8293	100.03
360.00	73.0	3648	3575.0	3101.7	1033.2	1229.0	0.03472	196.7	6.8294	100.03

^aColumn 5 divided by 3.00192 microvolts/psi. Gage factor = 1000 microvolts/volt input at full scale (2000 psi). Input voltage = 6.00385 volts.^bEvaluated relative to plateau obtained for saturated steam at 193.33°C. Saturated steam pressures from reference 3.^cObtained from smooth curves through values from reference 3.^dCubical coefficient of expansion of SS347 = $5.13 \times 10^{-5}/^{\circ}\text{C}$. Reference 2.

FIGURE 1
DIAPHRAGM MOUNTING

FIGURE 2
DIAPHRAGM - "O" RING
FLANGE DETAIL

FIGURE 3

CAP AND HEAD ASSEMBLY FOR VACUUM FILLING OF PRESSURE VESSEL

FIGURE 4

FIGURE 5
PRESSURE PLATEAU OF SATURATED STEAM
AT 193.33°C

Gage "Zero" Reading = 72.7 μ volts
Input Voltage = 6.00385 volts
Gage Factor = 1000 μ volts per
volt input, full scale (2000 psi)