STUDY OF DENSITY CALIBRATION IN SPACE

by P. Fowler and F. J. Brock

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for Goddard Space Flight Center

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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The research covered in this report was carried out by Peter Fowler (Principal Investigator) and Frank J. Brock.
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STUDY OF DENSITY CALIBRATION IN SPACE

By P. Fowler and F. J. Brock
National Research Corporation

1. SUMMARY

Methods of calibrating ion gauges and mass spectrometers were studied, under NASA Contract NAS5-3959, to determine a method which would be applicable to in-flight calibration of detectors installed on spacecraft. The method developed in the first phase of the program, consists of diffusing a known hydrogen flux through the wall of a small diffuser tube into the detector enclosure which produces a pressure increment equal to the ratio of hydrogen flux to exhaust conductance, by raising the temperature of the tube with an internal heater which dissipates a prescribed power for a prescribed time interval. In the first phase of the work, three successive generations of prototype in-flight calibrators were designed and constructed and the performance of the Mark I and Mark II were experimentally evaluated. The performance of Mark III has been evaluated in the second phase of the program.

The results indicate that the diffuser generates a hydrogen flux which is linear over a dynamic range of $3 \times 10^5$, that calibration pulse heights are repeatable within $\pm 2.0\%$ and that pulse height variation can be maintained less than $1.0\%$ for 30 seconds. The data indicate that the pressure decay rate, associated with the diffuser, following a calibration pulse is such that after 15 minutes the pressure is no more than several percent of the external pressure if the calibration pulse is no more than an order of magnitude above the external pressure, except for pressures less than $10^{-9}$ Torr, which require a longer recovery time. Recommendations are made for shortening the low pressure recovery time.

2. INTRODUCTION

During the first phase of this program a method was developed which is applicable to the calibration of ion gauges and mass
spectrometers during space flight. The method selected is based on the production of a known pressure in the detector envelope by controlling the flow of hydrogen gas into and out of the detector volume. A gauge calibration system schematic is shown in Figure 1. The pressure external to the satellite, which is to be measured, is $P_0$. A calibration is performed in the neighborhood of $P_0$ by introducing a known pressure increment within the gauge envelope. The pressure increment is produced by applying a temperature pulse to a thermal diffuser located within the gauge enclosure. The temperature pulse increases (exponentially) the gaseous diffusion rate thru the wall of the diffuser, producing a flux of calibrating gas $\dot{Q}$. The resulting gas pressure within the gauge enclosure is

$$P_d = P_0 + \frac{\dot{Q}}{C},$$

where $C$ is the gas conductance between the diffuser and the exterior. Ionic pumping of the gauge is considered to be negligible compared with the gas conductance to the exterior of the satellite. By regulating the diffuser temperature and thus the flow of calibrating gas, $P_d$ may be varied over a prescribed pressure range.

During the first phase of the program, performance goals were defined for a calibration system applicable to a pressure (or partial pressure) detector on a satellite executing an elliptical orbit. These goals are as follows:

1) System to calibrate a gauge at pressures of $1 \times 10^{-7}$ Torr and $1 \times 10^{-9}$ Torr by increasing the pressure within the gauge to $1 \times 10^{-6}$ Torr and $1 \times 10^{-7}$ Torr, respectively.

2) The calibrating pulses to have less than a 1% effect in subsequent pressure measurements.

3) The rise-time of the calibration pulse to be less than 3.5 minutes. The pressure then to remain steady at the calibrating level for 10 seconds. The pressure level due to calibrating gas must then decrease to negligible values in less than 15 minutes.
FIGURE I
SCHEMATIC OF SATELLITE CALIBRATION SYSTEM
4) Maximum power consumption of the thermal value to be less than 5 watts.

5) Number of calibrations required: 12. One per month over a 12-month period.

6) Volume of gas to be carried: less than 100 c.c.

7) Telemetry rate for pressure data: 20-60 samples/seconds. Telemetry rate for temperature data: 20 samples/second.

8) Satellite temperature: -10 to +50°C.

9) Volume of gauge and conductance to be less than 330 c.c.

10) Conductance to exterior of satellite to be greater than 10 l/second.

11) Time constant of system for pressure variations to be less than 0.03 seconds.

12) Roll frequency of satellite: 3 cps.

13) Mass of system to be a minimum.

During phase one, two prototype calibrators, Mark I and Mark II were designed, constructed, and tested and the results of those tests used to design and construct Mark III Calibrator. The work performed in that phase was summarized in a final report issued in November 1965. The objective of this phase of the program is to experimentally evaluate Mark III Calibrator and to determine its compatibility with the above performance goals.

Figure 2 shows a detail schematic of Mark III and Figure 3 shows Mark III joined to a Redhead normal magnetron gauge (NRC 552). In the construction of Mark III only materials suitable for operation at high temperatures were used. For example, all braze joints required were made with gold-nickel braze (M. P. 950°C), thus allowing high degassing temperatures in order to obtain improved low pressure performance.

A vacuum jacket was built around the stainless steel tube
SECTIONAL SKETCH OF MARK III CALIBRATOR
FIGURE 3
MARK III CALIBRATOR JOINED TO MAGNETRON GAUGE
below the diffuser section to more precisely define the diffusing area and to reduce the thermal isolation of the diffuser section. This was done to improve low pressure performance and increase the rate of cool down and thus the pressure decay rate following a calibration pulse. In addition, a thick OFHC copper plate was brazed to the diffuser section to assure that the diffuser area would be isothermal. The thermocouple was attached directly to the copper plate to provide accurate temperature data during rapid transients. Finally, the normal magnetron gauge was used to measure $P_d$ so that reliable pressure measurements could be extended to the $10^{-12}$ Torr range.

3. EXPERIMENTAL APPARATUS AND PROCEDURE

The Mark III Calibrator and associated magnetron gauge were joined to an oil diffusion pumped ultra-high vacuum system, having two anti-migration LN$_2$ cold traps. This system is shown in Figure 4. Figure 5 is a schematic of the system. In Figure 4, a modulated Nottingham gauge is shown joined to the system in place of the reference magnetron gauge. The circular table below the top cold trap is the base of a bakeout oven which was used to degas the top trap, system gauges, high vacuum valve, and Mark III Calibrator. During system bakeout, the lower anti-migration cold trap was filled with LN$_2$. Prior to starting the 4" diffusion pump, the lower cold trap was heated to 450°C with the 2" diffusion pump operating so as to maintain the system pressure low during bakeout. During the course of the program, the system was degassed at 425°C for five periods greater than 24 hours each and over two weekend periods at 320°C and 380°C.

Data were taken with the system cooled to room temperature (-22°C). Continuous recording on a Honeywell Electronik 17 two-pen recorder was made of the reference magnetron and diffuser magnetron collector currents using Keithley 610R and 410 electrometers. The diffuser copper-constantan thermocouple EMF was measured with a Keithley 148 Nanovoltmeter and continuously recorded with a Moseley 680 autograph recorder. The thermocouple wire was precision grade with a standard error limit of ±0.2°C. The temperature was obtained from the National Bureau of Standards thermocouple table. For EMF's greater than the standard range, an extrapolation was made of the table using.
FIGURE 4
MARK III CALIBRATOR AND ASSOCIATED GAUGES
ATTACHED TO VACUUM SYSTEM FOR TESTING
FIGURE 5

SCHEMATIC CONFIGURATION OF SYSTEM FOR MARK III PROTOTYPE PERFORMANCE TESTING
a quadratic fit that agreed extremely well in the 300-400°C range. Heat sink temperature, when obtained, was measured using a thermistor in a Wheatstone bridge circuit. Heat sink temperature was varied by varying the temperature of a massive brass block clamped to the heat sink with brass filings in between. The brass block was in thermal contact with a variable set point constant temperature circulator and was insulated with several layers of aluminized Mylar.

Linde regent grade ultra-pure hydrogen obtained in metal cylinders from J. T. Baker Chemical Company was used. A batch analysis by Linde of the hydrogen used showed the following impurities:

- $H_2O$: 2 PPM
- $O_2$: 3 PPM
- $CH_4$: 1 PPM
- $N_2$: 1.8 PPM

The all-metal gas transfer system was evacuated using LN$_2$ trapped mechanical pumps. The gas pressure was measured with a rotating piston gauge primary pressure standard (CEC type 6-201-0001). The hydrogen gas was then captured by closing a crossover valve around a differential pressure gauge (MKS Baratron Type 77H-1068) which was used to continuously monitor the gas pressure in the Mark III diffuser volume.

Power was supplied to the diffuser heater from a regulated low voltage power supply (ERA model TR040M). The current and voltage to the heater were measured with 1% laboratory meters (Weston Model 901). All measurements were made with either 14.6 psi or 29.5 psi $\pm$1% hydrogen pressure in the diffuser manifold.

The data were obtained over a period of 1½ calendar months, and the measurements were of two distinctly different types as follows:

1) Steady-state measurements were made of the equilibrium pressure attained in the system after applying a constant voltage to the diffuser heater. The pressure of the system after reaching the steady-state (generally defined as less than ½ percent
variation in measured pressure during a period greater than several minutes), the temperature of the diffuser, the voltage and current to the diffuser heater, and in some cases the heat sink temperature were observed.

2) Transient measurements were made of the quasi-steady-state pressure obtained by applying a 3 minute heating pulse at constant applied voltage and then switching into the circuit for 30 seconds a series resistance of such a magnitude that the diffuser temperature and thus the system pressure remains constant to within +2% for thirty seconds. All the quantities measured in the steady-state runs, with the exception of the heat sink temperatures, were also measured in the pulse runs. In addition, the voltage and current to the diffuser heater were measured during the plateau period of the pulse.

The lowest system pressures were attained immediately after cool down from a bakeout. The lowest pressure measured during the program was $9 \times 10^{-12}$ Torr (equivalent $N_2$) at the system pressure gauge. It is believed that the system pressure is limited by the rate of decomposition of the DC 705 oil used in the diffusion pump since the ultimate pressure was not steady but was composed of closely spaced random amplitude pulses dependent to some extent on the diffusion pump heater power. The lowest pressure measured at the reference and diffuser gauges was $1.5 \times 10^{-11}$ Torr (equivalent $N_2$). The magnetron sensitivity was determined from the data of Feakes, et al(2), for nitrogen and corrected to hydrogen by using 2.2 A per Torr above $10^{-9}$ A, which is the break point below which the NRC 552 becomes non-linear. Below $10^{-9}$ A the hydrogen calibration was assumed to follow the same power law as found by Feakes, et al(2). Their data were adjusted for hydrogen by multiplying the current axis by 0.488 to obtain hydrogen pressure.

4. RESULTS

4.1 Preliminary Experiments

An initial low temperature run was made to determine the effect of heat sink temperature variation on the calibration
The heat sink was initially cooled to $257^\circ K$. Upon admitting 14.6 psi of hydrogen to the diffuser no variation in $P_d$, the pressure in the calibrator magnetron gauge, was found. The temperature of the heat sink was slowly raised to $352^\circ K$. No variation in $P_d$ was noted, even for a temperature of the diffuser as high as $342^\circ K$. It was concluded from the experiment that the time required to set up equilibrium diffusive flow in the stainless steel tube was longer than 10 hours for temperatures near $300^\circ K$.

Reference to the time to set up equilibrium will be made in a manner analogous to the work of Barrer\(^3\). For the general case of flow through a plane sheet of thickness $\ell$ in which the concentration is maintained at a constant $C_o$ on one side and $C_x=0$ on the other, the approach to equilibrium flow is as shown qualitatively in the Q-t plane in Figure 6. The backwards extrapolation of the equilibrium flow rate meets the abscissa at a time $\tau$ which is called the lag time and is found to be related to the diffusion constant as follows

$$\tau = \frac{\ell^2}{6D} \quad (2)$$

Actually, the hydrogen concentration at the high pressure surface of the diffuser wall is not constant as the temperature increases. The total hydrogen supply volume is large compared to the internal volume of the diffuser. The volume external to the diffuser remains at room temperature even though the temperature of the gas within the diffuser internal volume may increase by several hundred degrees. Therefore, the pressure throughout the hydrogen supply volume (including the diffuser internal volume) remains substantially at the initial hydrogen charge pressure (generally about 1 atmosphere) independent of diffuser temperature. Thus, the hydrogen density in the diffuser internal volume decreases like $T^{-1}$ as the diffuser temperature increases. The number of hydrogen molecules colliding with unit area of the high pressure surface of the diffuser wall in unit time, therefore, decreases like $T^{-\frac{1}{2}}$. From this effect alone, the hydrogen concentration in the diffuser wall at the high pressure surface must decrease as the temperature increases. However, the hydrogen solubility in stainless steel increases with temperature faster than $T^\frac{1}{2}$ (based on solubility data in the literature\(^4\)). Thus, the total effect of temperature is such that
FIGURE 6
INITIAL DIFFUSION OF GAS THROUGH A SOLID BODY

S = System pumping speed
Slope = PS

Q, Torr liters
Time, t, seconds
the hydrogen concentration in the high pressure surface of the diffuser increases with temperature.

4.2 Steady-State Diffusion Experiments

To establish an initial hydrogen concentration gradient within the stainless steel diffuser tube, the sink was kept near room temperature by convective cooling with room air and the diffuser was heated by applying power to the heater. No increase in $P_d$ was observed after 10 minutes of heating at temperatures up to $450^\circ$K. Upon increasing the temperature to $530^\circ$K, the first diffusion of gas was noted causing $P_d$ to increase within several minutes time by two orders of magnitude. It was concluded that the lag time at $520^\circ$K was less than 1000 seconds. The diffuser temperature was increased to $550^\circ$K to establish equilibrium hydrogen concentration in the diffuser.

Over a period of several weeks, many separate experiments were carried out to define the functional form of the steady-state pressure versus temperature over the range of pressure from $10^{-12}$ to $10^{-6}$ Torr (H$_2$). The diffuser temperature was varied over the range 280°K to 770°K. Measurement of temperature at the high end of this range required extrapolation of the thermocouple EMF table as discussed earlier. Figure 7 is a plot of log $P_d$ versus 1/T for the data obtained in these experiments. It is noted that there is a large deviation from linearity of the data in the region of low pressure. A careful look at the data shows that the pressure at which deviation from linearity occurs depends upon the pressure attained in the previous diffuser experiments and thus does not act like a typical background due to outgassing.

In Figure 8, the data for the steady-state experiments is replotted, but with the initial background of each experiment substracted. It is seen that the data are grouped closely about a straight line over the whole range of pressure investigated. This is the expected behavior for diffusive flow of hydrogen through the wall of the stainless steel diffuser tube. The slope of the plot yields the effective activation energy for the diffusion process.

The least squares fit to the straight line was calculated
DIFFUSER GAUGE PRESSURE FOR STEADY STATE EXPERIMENTS

FIGURE 7
FIGURE 8
STEADY STATE DIFFUSER GAUGE PRESSURE, CORRECTED FOR BACKGROUND
and is shown on Figure 8. The relative standard error of estimate for the least squares fit is 1.3% to all the data. This low error indicates, among other things, that the effective diffusion area is constant, a fact influenced by the improved design of the Mark III diffuser shorting bar and the use of a guard vacuum around the hydrogen supply tube.

In order to test for curvature, a second order least squares fit was tried but this procedure did not lower the standard error of estimate. This is not surprising for there is no physical basis for expecting the quadratic term. However, by determining the least squares fit to a straight line for different parts of the whole pressure range, there was found to be a systematic increase in slope and decrease in standard error of estimate as more of the lower pressure points were neglected. For example, considering only the $10^{-8}$ to $10^{-6}$ Torr (H$_2$) points, the slope is 9% higher than for the $10^{-12}$ to $10^{-6}$ points. This variation is summarized in Table I. It may also be seen that the relative standard error of estimate of the least squares fit decreases as the lower pressure data is dropped out. There are several reasons for this behavior. First, as the temperature of the diffuser is increased there is a tendency for it to become more isothermal and thus the parts of the tube beyond the copper shorting strip can contribute comparatively more to the flow than at lower temperatures. Second, as will be shown later, in the lower part of the temperature range, the hydrogen concentration within the diffuser wall is higher than the equilibrium concentration corresponding to that range. Third, as discussed later in section 4.7, the hydrogen flux through the diffuser was observed to depend on hydrogen supply pressure more strongly than the $\frac{1}{3}$ power. This implies that at high temperature a molecular hydrogen flux component becomes observable (presumably due to molecular hydrogen flow along grain boundaries). Thus, the total flux observed in the calibrator at high temperatures may be the sum of the atomic hydrogen diffusion through the metal and molecular hydrogen diffusion along grain boundaries. Finally, as has already been pointed out, the hydrogen concentration in the high pressure surface of the diffuser increases with temperature.
TABLE I  
STEADY-STATE EXPERIMENT SUMMARY

<table>
<thead>
<tr>
<th>Pressure Range, Torr(H₂)</th>
<th>A</th>
<th>B, °K</th>
<th>Error, percent</th>
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<tbody>
<tr>
<td>10⁻¹² - 10⁻⁶</td>
<td>-2.512</td>
<td>-2764</td>
<td>1.3%</td>
</tr>
<tr>
<td>10⁻¹¹ - 10⁻⁶</td>
<td>-2.460</td>
<td>-2789</td>
<td>1.2%</td>
</tr>
<tr>
<td>10⁻¹⁰ - 10⁻⁶</td>
<td>-2.315</td>
<td>-2863</td>
<td>1.0%</td>
</tr>
<tr>
<td>10⁻⁹ - 10⁻⁶</td>
<td>-2.298</td>
<td>-2870</td>
<td>0.6%</td>
</tr>
<tr>
<td>10⁻⁸ - 10⁻⁶</td>
<td>-2.020</td>
<td>-3040</td>
<td>0.4%</td>
</tr>
</tbody>
</table>

where A and B are the constants in the least squares fit of the data to

\[ \log(P_d - P_{\text{background}}) = A + \frac{B}{T} \],

and the error given is the relative standard error of estimate for the least squares fit.
The lowest pressure points were measured in an experiment to determine the effect of heat sink temperature variation (to simulate the variations in temperature of a spacecraft). This run was made after a large concentration of hydrogen had been established in the diffuser. The diffuser heater was not activated but rather the diffuser was heated by conduction from the heat sink. The fact that these points fit so well to the straight line for the higher temperatures obtained by using the diffuser heater indicates the success, in the Mark III design, of the copper shorting bar in maintaining the diffuser isothermal and the diffuser guard vacuum in accurately defining the diffusing area.

4.3 Diffusion Constant and Lag Time

Some steady diffusion points were obtained for a set of equilibrium temperatures such that each temperature was less than the previous temperature. These data contained evidence that the hydrogen concentration in the diffuser did not arrive at its new equilibrium concentration within the same time interval required to establish a new equilibrium temperature. For example, after a constant pressure experiment at a diffuser temperature of 769°K, the temperature was lowered to 569°K and was stable after 11 minutes. However, the pressure continued to decrease for an additional 10 minutes to about 95% of its value at the time of reaching temperature equilibrium. It, thus, becomes obvious that knowledge of the effective diffusion lag time, \( \tau \), as a function of temperature is required.

To obtain \( \tau \), a step change in pressure was generated on the high pressure side of the diffuser and the variation in diffusion rate with time was observed. The effect of this change in pressure on a \( Q - t \) plot is shown in Figure 9. In analogy with the earlier definition of \( \tau \), the lag time derived from such an experiment for a change in low side pressure from \( P_Q \) to \( P_F \) is indicated in Figure 9. By analytical calculus it may be seen that \( \tau \) is given by
A step change in high side pressure is made at time $t_1$.

$S = \text{System pumping speed}$

$Q$, Torr Liters

Time, $t$, seconds

CHANGE OF FLOW RATE OF GAS THROUGH A SOLID BODY
The lag time, \( \tau \), was determined for 5 temperatures and the results are given on a semi-log plot versus \( 1/T \) in Figure 10. An attempt to determine \( \tau \) at \( 500^\circ K \) was unsuccessful due to the excessive time involved although a lower limit of 1000 seconds was obtained for that temperature. The least squares fit to the data is also plotted. It may be seen that \( \tau \) becomes long compared with the usual cooldown time of hundreds of seconds at the comparatively high temperature of \( 580^\circ K \) and that for room temperature \( \tau \) is about \( 10^7 \) seconds.

The effective diffusion coefficient \( D^* \), for \( P_{\text{H}_2} = 14.6 \) psi was obtained from equation (2) and the lag time, \( \tau \). The results are shown in Figure 11 along with data reported by Flint(4) and by Lewin(5). In addition, the effective diffusion coefficient was obtained from

\[
D^* = 1.32 \frac{S_p(H_2) P_d \ell}{A} \text{cm}^2/\text{sec},
\]

where \( P_d \) is the pressure measured in the steady-state runs, \( S_p(H_2) \) is the effective pumping speed for hydrogen at the diffuser, \( \ell \) is the thickness of the diffuser, and \( A \) is its area. The pumping speed of the system was determined for argon in an auxiliary experiment with a porous Vycor plug whose conductance is accurately known. Using free molecular flow theory \( S_p \) for hydrogen was calculated to be 24 liters/second. The value of \( D^* \) thus determined is also plotted in Figure 11 where close agreement is seen with the least squares fit to the points obtained from the pressure change technique.

### 4.4 Thermal Pulse Diffusion Experiments

Pulses of hydrogen gas with flat tops up to 30 seconds in duration were generated, starting from various initial diffuser temperatures. Background was subtracted from the pressure reached at the plateau similar to the method used in reduction
FIGURE 10
EFFECTIVE LAG TIME FOR MARK III DIFFUSER

\[ \frac{1}{T}, \quad o_k^{-1} \]

* \( \tau \) too long to measure by this method

\( \tau, \text{sec} \)
FIGURE 11

EFFECTIVE DIFFUSION COEFFICIENT FOR MARK III DIFFUSER
of the steady-state data. Table II summarizes the average value of pertinent parameters for each sub-group of pulses having the same peak temperature. These data were taken over a period of more than a week at various pulse amplitudes and initial temperatures. Only 10 seconds of the plateau were considered in that summary, the pressure constancy over the interval being better than +1% in general. Figure 12 is a plot of the logarithm of \((P_{\text{plateau}} - P_{\text{initial}})\) versus \(1/T\) for the average values above, also, the least squares fit to a straight line is plotted. The least squares slope is seen to differ from that of the steady-state points for the same pressure range by less than 6%. Some variation would be expected since it was found in the steady-state experiments that temperature equilibrium was not reached in the three minutes used in the pulse runs but rather took up to 25 minutes in some cases. In addition, the number of steady-state high pressure points is small (one point above \(3 \times 10^{-7}\) Torr (H₂)) and, therefore, the high pressure data is less reliable. For example, leaving out the point at \(10^{-6}\) Torr causes the least squares slope to change from -3045 to -2929. The latter slope is within 2% of the steady-state slope for points in the same pressure range.

In order to test the effect of initial temperature on the data, all runs with a plateau temperature in the vicinity of 515°K were corrected to 515°K, using the least squares fit for the pulse runs. Figure 13 shows a plot of \((P_{\text{plateau}} - P_{\text{initial}})\) for 515°K versus initial temperature. It is seen that the effect of initial temperature is negligible if the background subtraction scheme is followed.

### 4.5 Pressure Decay Following a Calibration Pulse

It has already been shown that the diffusion time lag, \(\tau\), is short at high temperatures. It, therefore, is anticipated that the pressure decay rate following a calibration pulse would be a function of the diffuser temperature decay rate only (at least for high to moderate diffuser temperatures). However, it is found experimentally that the observed pressure decay rate is slower than that calculated from the observed temperature decay rate. Thus, the hydrogen pressure in the calibrator is higher than that corresponding to steady-state diffusion at the instantaneous temperature of the diffuser during cooldown.
Least Squares Fit:

$\log P = -2.090 - \frac{3045}{T}$

**FIGURE 12**

PULSE PLATEAU PRESSURE, CORRECTED FOR BACKGROUND
### TABLE II

**SUMMARY OF THREE MINUTE PULSES WITH TEN SECOND PLATEAUS**

<table>
<thead>
<tr>
<th>Pulse Series No.</th>
<th>N</th>
<th>$T_{i}, \text{oK}$</th>
<th>$T_{p}, \text{oK}$</th>
<th>$P - P_{i}, \text{torr}$</th>
<th>$E, %$</th>
<th>$V, V$</th>
<th>$W_{\text{max}}, \text{W}$</th>
<th>$R, \text{Ohm}$</th>
<th>$I_{p}, \text{A}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8</td>
<td>352</td>
<td>522</td>
<td>$1.07 \times 10^{-8}$</td>
<td>4.4</td>
<td>7.60</td>
<td>1.98</td>
<td>3.6</td>
<td>0.166</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>352</td>
<td>521</td>
<td>$1.03 \times 10^{-8}$</td>
<td>0</td>
<td>7.60</td>
<td>1.91</td>
<td>3.6</td>
<td>0.166</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>352</td>
<td>526</td>
<td>$1.10 \times 10^{-8}$</td>
<td>0.5</td>
<td>7.60</td>
<td>1.94</td>
<td>3.6</td>
<td>0.166</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>352</td>
<td>512</td>
<td>$0.92 \times 10^{-8}$</td>
<td>2.3</td>
<td>7.50</td>
<td>1.84</td>
<td>3.6</td>
<td>0.168</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>381</td>
<td>525</td>
<td>$1.16 \times 10^{-8}$</td>
<td>0.7</td>
<td>7.60</td>
<td>1.98</td>
<td>3.6</td>
<td>0.165</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
<td>381</td>
<td>525</td>
<td>$1.15 \times 10^{-8}$</td>
<td>0</td>
<td>7.60</td>
<td>1.79</td>
<td>3.6</td>
<td>0.165</td>
</tr>
<tr>
<td>7</td>
<td>2</td>
<td>381</td>
<td>529</td>
<td>$1.22 \times 10^{-8}$</td>
<td>0</td>
<td>7.60</td>
<td>1.67</td>
<td>3.6</td>
<td>0.166</td>
</tr>
<tr>
<td>8</td>
<td>4</td>
<td>323</td>
<td>606</td>
<td>$8.58 \times 10^{-8}$</td>
<td>0.5</td>
<td>10.90</td>
<td>3.00</td>
<td>3.0</td>
<td>0.200</td>
</tr>
<tr>
<td>9</td>
<td>4</td>
<td>323</td>
<td>433</td>
<td>$1.00 \times 10^{-9}$</td>
<td>0</td>
<td>5.45</td>
<td>1.09</td>
<td>5.5</td>
<td>0.140</td>
</tr>
<tr>
<td>10</td>
<td>3</td>
<td>323</td>
<td>436</td>
<td>$1.00 \times 10^{-9}$</td>
<td>0.6</td>
<td>5.40</td>
<td>1.12</td>
<td>4.5</td>
<td>0.142</td>
</tr>
<tr>
<td>11</td>
<td>3</td>
<td>323</td>
<td>606</td>
<td>$8.83 \times 10^{-8}$</td>
<td>0.6</td>
<td>11.0</td>
<td>3.85</td>
<td>3.0</td>
<td>0.200</td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>323</td>
<td>777</td>
<td>$1.17 \times 10^{-6}$</td>
<td>3.2</td>
<td>19.0</td>
<td>10.64</td>
<td>2.2</td>
<td>0.254</td>
</tr>
<tr>
<td>13</td>
<td>2</td>
<td>294</td>
<td>512</td>
<td>$8.19 \times 10^{-9}$</td>
<td>0</td>
<td>7.60</td>
<td>2.28</td>
<td>3.6</td>
<td>0.170</td>
</tr>
<tr>
<td>14</td>
<td>1</td>
<td>297</td>
<td>511</td>
<td>$8.40 \times 10^{-9}$</td>
<td>-</td>
<td>7.60</td>
<td>2.22</td>
<td>3.6</td>
<td>0.185</td>
</tr>
<tr>
<td>15</td>
<td>1</td>
<td>297</td>
<td>517</td>
<td>$8.65 \times 10^{-9}$</td>
<td>-</td>
<td>7.60</td>
<td>2.09</td>
<td>3.6</td>
<td>-</td>
</tr>
</tbody>
</table>
where,

\[ \begin{align*}
N &= \text{Number of data points considered} \\
T_i &= \text{Initial temperature of diffuser} \\
T_P &= \text{Diffuser temperature during pulse plateau} \\
P_i &= \text{Initial pressure in diffuser magnetron} \\
P_P &= \text{Pressure in diffuser magnetron during pulse plateau} \\
E &= \text{Standard error in } (P_P - P_i) \text{ for pulse series} \\
V &= \text{Voltage applied to system} \\
W_{\text{max}} &= \text{Maximum measured power dissipation} \\
I_P &= \text{Current through diffuser during pulse plateau}
\end{align*} \]

Note: \( W_{\text{max}} \) was the power dissipation observed seconds after the voltage was applied. The initial power drawn with the filament cold was slightly higher.
FIGURE 13
EFFECT OF INITIAL TEMPERATURE ON PULSE EXPERIMENTS
In addition to hydrogen adsorption on the surfaces of the electrodes and vacuum enclosure, there are at least two other possible causes of this behavior. First, the hydrogen concentration within the diffuser wall at any instant may be that corresponding to a higher temperature if relaxation of the concentration distribution is a slower process than cooldown (this must happen if the diffuser temperature is sufficiently low). The solubility of hydrogen in stainless steel increases with temperature. The hydrogen concentration distribution corresponding to some higher temperature may be temporarily "frozen-in" during rapid cooldown of the diffuser. Thus, the hydrogen concentration at the vacuum surface of the diffuser would always be higher during cooldown than an equilibrium concentration corresponding to the instantaneous diffuser temperatures. Therefore, the rate at which gas could be desorbed from the vacuum surface would be higher than that corresponding to the steady-state diffusion process at the instantaneous temperature. If recombination to form molecular hydrogen on the vacuum surface is the rate limiting step in the total permeation process, the "frozen-in" nonequilibrium concentration suggested above would probably not be observable. Based on the assumption that the hydrogen concentration becomes "frozen" at about 700°K, (a conservative assumption based on the data in Figure 10), quantitative estimates of the total number of hydrogen atoms in or near the surface of the diffuser which would be available for desorption into the vacuum can account for only a small fraction of the total number of hydrogen molecules observed in the calibrator after the diffuser heater power was turned off, obtained from the time integral of the observed pressure and known conductance to the pumping system.

The second possible cause of the observed reduction in pressure decay rate following a calibration pulse, may be hydrogen liberated by the magnetron gauge. During the calibration pulse about 2% of the total diffuser flux is pumped by the magnetron gauge. After the diffuser flux is turned off and the pressure in the calibrator begins to decay, the hydrogen ion current to the magnetron cathode decreases. However, the quantity of hydrogen buried in the cathode corresponds to a higher ion incidence rate. This gas must continue to diffuse out of the cathode yielding an apparent hydrogen source until the quantity of gas remaining in the cathode is again in equilibrium with the (lower) ion current. It is not obvious how much time this recovery process requires. Since the cathode is at room temperature the unaided diffusion process would be slow. However,
the cathode is continually bombarded with energetic ions which produce considerable disturbances on an atomic scale (temperature spikes) and assist the liberation of hydrogen from the cathode.

It is, of course, possible that a substantial fraction of the hydrogen observed in the calibrator after the diffuser heater power is turned off comes from hydrogen desorption from the surface within the calibrator where it had been adsorbed during the high pressure part of the calibration pulse.

Several experiments were conducted to determine the actual source of hydrogen which produced the apparent reduction in pressure decay rate after a calibration pulse. The diffuser temperature was maintained at 501°K for a time comparable with the lag time $\tau$ at 501°K such that equilibrium concentration was established in the diffuser wall. After the diffuser heater power was turned off the pressure and diffuser temperature were measured as a function of time. The observed pressure is plotted in Figure 14, along with the pressure calculated for steady-state diffusion from the observed instantaneous temperature. The difference between the observed pressure time history and that calculated from the instantaneous diffuser temperature could result from either desorption or re-emission of hydrogen pumped ionically. From these results, it is not possible to determine the location of the gas source.

In another experiment the diffuser temperature was maintained at 436°K for an interval which was less than 2% of $\tau$ at 436°K. After the heater power was turned off the pressure and diffuser temperature were recorded as a function of time. Figure 15 presents the observed pressure and the pressure calculated from steady-state diffusion for the observed instantaneous diffuser temperature. It may be seen that the difference between the two curves is substantially less than in Figure 14. It is not possible to give an unambiguous explanation for the closer agreement between the two curves since maintaining peak diffuser temperature for a short period compared to $\tau$ would prevent the establishment of equilibrium concentration at 436°K and also maintaining peak pressure for a short period would reduce the number of ions that could be buried in the magnetron cathode, and at the same time, reduce the total number of hydrogen molecules adsorbed on the surfaces of the calibrator.
OBSERVED AND CALCULATED PRESSURE DECAY FOR MARK III, COOLED RAPIDLY FROM EQUILIBRIUM AT HIGH TEMPERATURE
FIGURE 15

OBSERVED AND CALCULATED PRESSURE DECAY FOR MARK III, COOLED RAPIDLY FROM NON-EQUILIBRIUM AT HIGH TEMPERATURES
The importance of this effect may be appreciated from the following experimental results. The diffuser was heated to 615°K corresponding to \( P_d = 2 \times 10^{-7} \) Torr (H\(_2\)) and equilibrium hydrogen concentration distribution was established. Heater power was removed and after cooldown the diffuser maintained at 295°K for 2.5 days. This period is short compared to the time lag period at 295°K which may be determined by extrapolating the measured time lag data given in Figure 10 to yield \( \tau = 116 \) days. The observed pressure after the 2.5 day decay period was \( 3 \times 10^{-11} \) Torr, which corresponds to the steady-state diffusion rate for a diffuser temperature of 345°K, if there were no other sources of hydrogen.

4.6 Three Level Pulse Experiments

The practicability of producing calibration pulses in the neighborhood of \( 10^{-9}, 10^{-8}, \) and \( 10^{-7} \) Torr (H\(_2\)) all with a single insertion resistance and fixed power supply voltage by varying the heating period prior to resistor insertion was investigated. The voltage was chosen such that the time required to reach \( \sim 10^{-7} \) Torr was less than 3 minutes. The value of the resistor inserted in series with the diffuser heater was chosen such that the peak pressure remained constant within 1.0% for 30 seconds for a pulse height of \( \sim 10^{-8} \) Torr (H\(_2\)). The results are shown in Figure 16. It may be seen that the peak heights of the high and low pressure pulses do not remain constant to better than \(+14\%\) and \(+23\%\) respectively.

From these results, it is concluded that the power dissipated in the diffuser heater must be different for each different height calibration pulse. This may be most efficiently accomplished by providing a number of resistors, one tailored to satisfy the dissipation required in the diffuser heater to maintain constant temperature for each calibration pulse height desired.

4.7 Diffusive Flow Dependence on Pressure

The flux for a diatomic gas which reacts with the surface and diffuses through a solid as atoms, recombining to desorb into the vacuum as molecules is given by

\[
\dot{Q} = \frac{S(T)D(T)A}{\lambda} \frac{1}{2} P_h \exp\left(-\frac{E}{RT}\right),
\]

(5)
FIGURE 16
THREE LEVEL PULSE
where, \( S(T) \) is the gas solubility in the solid, \( D(T) \) is the diffusion coefficient, \( A \) is the area of the solid thru which the gas is diffusing, \( l \) is the length of the solid, \( P_h \) is the high side pressure, and \( E \) is the activation energy for the diffusion process.

This is considered the flow mechanism in Mark III Calibrator. The pressure on the vacuum side of the diffuser \( P_d \), is related to this flow rate by

\[
\dot{Q} = P_d S_p ,
\]

where, \( S_p \) is the effecting pumping speed of the system, including the effects of ionic pumping by the gauge. Thus, if the high side pressure \( P_h \) is changed at constant temperature, it is expected that

\[
\frac{P_{d_i}}{P_{d_f}} = \left( \frac{P_{h_i}}{P_{h_f}} \right)^{1/2}
\]

where, \( i \) refers to initial and \( f \) to final pressures, respectively. This relation has been tested experimentally and the results are given in Table III. It may be seen that deviations from monatomic diffusion increase with temperature (except for the point at 651°K which has a relatively small deviation). This implies that a small fraction of the hydrogen is diffusing as molecular hydrogen. The deviation from atomic diffusion appears to increase exponentially with temperature.

4.8 Activation Energy for Diffusion

The activation energy in the diffusion process may be obtained from the slope of the Arrenius plots such as Figures 7, 8, or 11. If only the data above \( 4 \times 10^{-10} \) Torr \( (H_2) \) is considered, in order to leave out the effect of the "background" then the average slope from these three figures is found to be

\[
\frac{E}{k} = -6540 \pm 50 \text{ Degrees},
\]
TABLE III
COMPARISON OF OBSERVED $P_d$ WITH DIFFUSION THEORY
FOR MARK III CALIBRATOR

<table>
<thead>
<tr>
<th>$P_{hi}$, psi</th>
<th>$P_{hf}$, psi</th>
<th>Temp, °K</th>
<th>$\frac{P_{di}}{P_{df}}$</th>
<th>Deviation from Theory, percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>14.6</td>
<td>29.5</td>
<td>594</td>
<td>0.7062</td>
<td>+0.4</td>
</tr>
<tr>
<td>14.6</td>
<td>29.5</td>
<td>607</td>
<td>0.6950</td>
<td>-1.2</td>
</tr>
<tr>
<td>29.5</td>
<td>14.6</td>
<td>651</td>
<td>1.448</td>
<td>+1.8</td>
</tr>
<tr>
<td>14.6</td>
<td>29.5</td>
<td>656</td>
<td>0.7370</td>
<td>+4.8</td>
</tr>
<tr>
<td>29.5</td>
<td>14.6</td>
<td>770</td>
<td>1.597</td>
<td>+12.2</td>
</tr>
</tbody>
</table>

The deviation from theory in the last column is defined as:

$$\frac{P_{di}}{P_{df}} - \left(\frac{P_{hi}}{P_{hf}}\right)^{\frac{1}{2}} - \left(\frac{P_{hi}}{P_{hf}}\right)^{\frac{1}{2}}$$
from which it follows that the activation energy for diffusion of Hydrogen thru stainless steel is 13.0 kilocalories per mole.

5. CONCLUSION AND DISCUSSIONS

The principal design changes in Mark III Calibrator (relative to Mark II) were made with the expectation of achieving a well defined isothermal diffuser area, faster thermal response, and a larger dynamic range. From the linearity of Figures 8 and 12, it may be concluded that the actual diffusing area is independent of temperature and that the diffuser is isothermal. These data also imply that the diffuser hydrogen flux dynamic range is about $3 \times 10^5$ for the temperature interval 777$^\circ$K to 323$^\circ$K. The latter number was defined to be the maximum spacecraft temperature and, therefore, the maximum heat sink temperature.

Data similar to that presented in Figure 16 verify that the peak pressure of a calibration pulse may be maintained for time intervals of the order of 30 seconds with less than 1.0% pressure variations, but these data also imply that separate insertion resistors are required for each calibration pulse height if in-flight step calibrations of the detector are to be performed over a relatively large dynamic range. However, if data sampling is frequent, calibration may be continuous over some specified time interval, for example, between 10 and $10^2$ seconds. Thus, the full dynamic range of the diffuser may be used by fixing the diffuser heater circuit resistance such that the desired temperature rise rate is obtained for the onboard power supply available. In this continuous calibration mode, the instantaneous calibration pressure is determined from the measured instantaneous diffuser temperature and the calibration range is determined by the heating period.

For in-flight calibrations corresponding to external pressures in the mid to high pressure range of the detector, the observed pressure decay rate is such that 15 minutes after the calibration pulse the background associated with the calibration pulse (from all sources) is only a few percent of the external pressure if the calibration pulse is no more than an order of magnitude higher than the external pressure. However, near the low end of the detector pressure range, the observed decay rate
indicates that considerable attention must be given to minimization of the residual flux following a calibration pulse. The following actions are considered necessary or desirable in applications of the techniques developed here to in-flight calibration:

1) The continuous calibration mode is favored over the step calibration mode.

2) The calibration pulse height should be no larger than an order of magnitude above the external pressure near the high pressure end of the dynamic range of the detector and perhaps no more than a factor of 3 above the external pressure near the low pressure end of the detector dynamic range.

3) The calibration pulse period should be as short as possible.

4) The pumping speed of the detector should be as small as possible.

5) The external conductance should be small but large compared to the pumping speed of the detector.

6) The internal surface area of the detector should be as small as possible.

7) The diffuser area should be chosen such that the maximum calibration required is obtained only at the maximum safe diffuser temperature (probably several hundred degrees higher than the maximum temperature used here).

These actions assure that the quantity of gas liberated to produce a given height calibration pulse is minimized, that gas adsorption within the detector is minimized, and that the area of the diffuser is minimized, thus, minimizing the time required for recovery after calibration. The useful calibration pressure dynamic range achieved in a specific flight application will depend on the degree to which these requirements are satisfied.

The results obtained in this work may be summarized, for
application to the design of an in-flight calibrator, as follows: The equilibrium hydrogen flux liberated from an isothermal thin wall, 304L stainless steel diffuser tube is given by:

\[
N_{H_2} = C_1 \frac{A}{\lambda} P_h \exp \left( -\frac{C_2}{T} \right), \text{sec}^{-1},
\]

where, 

- \( C_1 = 2.668 \times 10^{15}, \text{sec}^{-1} \text{cm}^{-1} \text{Torr}^{-\frac{1}{2}} \)
- \( C_2 = -6608, ^\circ\text{K} \)
- \( A = \text{Area of diffuser, cm}^2 \)
- \( \lambda = \text{Wall thickness of diffuser, cm} \)
- \( P_h = \text{Hydrogen supply pressure, Torr} \)
- \( T = \text{Diffuser temperature, } ^\circ\text{K} \)

The hydrogen density within the detector enclosure is then given by:

\[
n_{H_2} = \frac{N_{H_2}}{C_{H_2}}, \text{cm}^{-3},
\]

where, \( C_{H_2} = \text{Enclosure exhaust conductance for hydrogen at the temperature of the enclosure, cm}^3/\text{sec} \).

Due to uncertainties in estimating the effective area for hydrogen diffusion in the diffuser used, \( C_1 \) may be in error by as much as 10%. The value of \( C_2 \) given above was taken from the high temperature portion of the data and may therefore predict a hydrogen flux, which is low at low temperatures.
REFERENCES


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—National Aeronautics and Space Act of 1958

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