

# Potential of a Platinum Electrode at Low Partial Pressures of Hydrogen or Oxygen

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## ABSTRACT

The open-circuit potential on bright platinum in 1M  $\text{H}_2\text{SO}_4$  was studied as a function of oxygen or hydrogen partial pressure from  $10^{-2}$  to  $10^{-7}$  atm. The very low rates of  $\text{H}_2$  or  $\text{O}_2$  flow which were required were produced by a special gas generator. Potentials predicted by the Nernst equation for the  $\text{H}^+/\text{H}_2$  couple were not observed below  $10^{-4}$  atm  $\text{H}_2$  partial pressure. A Nernst relation with a slope of 0.06 v per decade of  $\text{O}_2$  partial pressure was found.

The  $\text{O}_2$  leak into the closed system used was found to be about  $10^{-7}$  atm. Based on the assumption that dissolved  $\text{O}_2$  decreases the effective  $\text{H}_2$  partial pressure at the electrode surface, an equation was developed for obtaining the effective  $\text{H}_2$  partial pressure.

## PROBLEM STATUS

This is an interim report on one phase of the problem of electrode mechanisms; additional work on this problem is continuing.

## AUTHORIZATION

NRL Problems C05-06 and C05-13  
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# POTENTIAL OF A PLATINUM ELECTRODE AT LOW PARTIAL PRESSURES OF HYDROGEN OR OXYGEN

## INTRODUCTION

The Nernst equation relating hydrogen partial pressure and potential of a platinum electrode in acid solution has been experimentally verified previously for  $H_2$  partial pressures ranging from hundreds of atmospheres down to 0.01 atm (1). It is often assumed to be valid at much lower  $H_2$  pressures. One purpose of this study was to check this assumption and to determine the conditions under which normal Nernst behavior prevails. The potential behavior of Pt in acid solutions equilibrated with oxygen at partial pressures ranging down to less than  $10^{-6}$  atm was also determined.

Previous work at this Laboratory (2,3) had shown that substantial amounts of  $O_2$  and  $H_2$  could dissolve in the "skin" of a Pt electrode and affect its catalytic activity (and also appear as an unexpected participant in chemical reactions). We differentiate here between solution of a gas into the bulk of the metal in the usual sense of the word "solution" and a kind of rapid selective absorption of gas atoms into a relatively few atomic layers of metal immediately below the surface atomic layer. This layer immediately below the surface we shall call the "derma" of the metal, by analogy to biological usage. This type of absorption can then be termed "dermasorption." These experiments were designed to give additional information on the ways gases absorb in Pt, particularly on the time required to sorb and desorb them, and the effects of sorbed gases on open-circuit potentials.

For meaningful measurements in electrochemical systems having small exchange current densities, conditions inside the cell must be rigorously controlled. The system utilizing Pyrex pipe recently built in this Laboratory (4-6) gave such control. Being gas-tight, it permitted effective exclusion of air and other atmospheric contaminants.

## EXPERIMENTAL METHOD

### Apparatus

The electrochemical cell (6) contained a miniature glass reference electrode, a Pt bead electrode of about  $0.17 \text{ cm}^2$  apparent area, a Pt wire electrode 5 cm long with a 0.064-cm diameter, and a Pt gauze electrode of very large area. The surface-to-volume ratios of the bead, wire, and gauze were 26, 62, and  $450 \text{ cm}^{-1}$ , respectively. The electrolyte was 1M  $H_2SO_4$  maintained at  $25 \pm 1^\circ C$ . The helium purification train was identical with that described previously (5), except that an improved electrolytic  $H_2/O_2$  generator replaced the conventional design previously used. Hydrogen was treated as before in a Serfass hydrogen purifier, saturated with water, and introduced into the gas train between the  $H_2/O_2$  generator and the cell. Kern high-vacuum greasless stopcocks (Viton-A diaphragm) at the ends of a T-connection between the He and  $H_2$  lines were used to block the flow of either gas. The  $H_2$  line was always back-flushed (from cell to Serfass) with He and left filled with pure He when not in use.

The  $H_2/O_2$  generator\* shown in Fig. 1 was used to generate pure  $H_2$  or  $O_2$  at low rates. We shall describe this device as an  $H_2$  generator with the understanding that the same principles also apply to the  $O_2$  case. For our purposes, it was important that  $H_2$  added to the helium stream not be contaminated with trace amounts of  $O_2$ , so the generator was designed to avoid crossdiffusion of electrode products while retaining the advantages of electrolytically generated gas.† Crossdiffusion is impeded in four ways: (a) a very long path, including a glass frit, separates the two compartments; (b) buffer compartments act very much like plates in a conventional condenser— $H_2$  diffusing downward from the working electrode is stripped by the incoming pure He, while in the upper chamber, the He +  $H_2$  stream coming from the electrochemical cell creates an  $H_2$ -rich region below the  $O_2$ -rich counter electrode compartment; (c)  $H_2$ -rich gas bubbles flowing over the Pd-tube counter electrode impede  $O_2$  bubble formation; and (d)  $H_2$  gas introduced on the inside (i.e., the room side) of the Pd tube dissolves into the Pd, and the anodic reaction is largely  $H \rightarrow H^+ + e$  (7). In practice, visible bubble formation at the counter electrode is only seen at currents in excess of 20 to 50 ma. Hydrostatic pressure difference between the two chambers automatically compensates for the pressure drop through the cell and associated bubblers.

Perhaps the greatest advantage of this design is that at very low  $H_2$  or  $O_2$  generation rates no gas is formed at the counter electrode at all. The anodic reaction at the counter electrode is the oxidation of H (dissolved in Pd) to  $H^+$ ; a steady supply of H (dissolved) is provided by  $H_2$  introduced inside the tube. The cathodic reaction at the counter electrode is the reduction of  $H^+$  to H; this  $H_2$  dissolves in the Pd tube, diffuses to the inside surface, and reacts with the  $O_2$  there (in this case the inside of the Pd tube is open to air) to form  $H_2O$ .

#### Procedure

Glassware was cleaned with boiling  $HNO_3$  followed by 12 to 15 rinses with triply distilled water and/or by heating to the annealing temperature. Preliminary solution pre-electrolysis and calibration of the glass reference electrode in 1 atm of  $H_2$  were carried out as before (6): the  $H_2$  line was back-flushed and filled with He, and the desired partial pressure of  $H_2$  or  $O_2$  in He (known to  $\pm 5\%$ ) was obtained by generating gas at the required rate. Helium flow was always  $88 \pm 4$  cc/min, pressure within the cell was 13 torr over atmospheric, and open-circuit potentials of the three electrodes were measured with a Keithley 603 Electrometer and reported versus the normal hydrogen electrode (N.H.E.), as before (6).

Although the experimental system was gastight, there is no question but that a minute amount of leakage of  $O_2$  from air‡ took place by diffusion through Viton-A O-rings connecting the various sections of glass pipe. This leakage was unimportant as long as the partial pressure of  $O_2$  or  $H_2$  was more than  $10^{-5}$  atm. However, at partial pressures of these gases approaching  $10^{-6}$  atm and lower, the leak rate of  $O_2$  from the air was significant and was taken into account. Since the He flow was constant, leak rates will for convenience frequently be discussed in terms of the net partial pressure of gas resulting.

\*Fig. 1 shows an improved design and differs from the generator employed in this study in one respect; instead of using the circular disks shown, baffles in the lower buffer compartments were formed by pushing in the sides of the glass walls as is commonly done in vapor condensers.

†A conventional H-cell generator cannot be used for partial pressures below  $10^{-4}$  to  $10^{-5}$  atm because of effects due to crossdiffusion of the gases produced.

‡It should be pointed out that the air in the laboratory was purified by constant recirculation through an absolute filter to remove aerosols and through a carbon bed to remove gases and vapors. Smoking was not permitted in the laboratory.

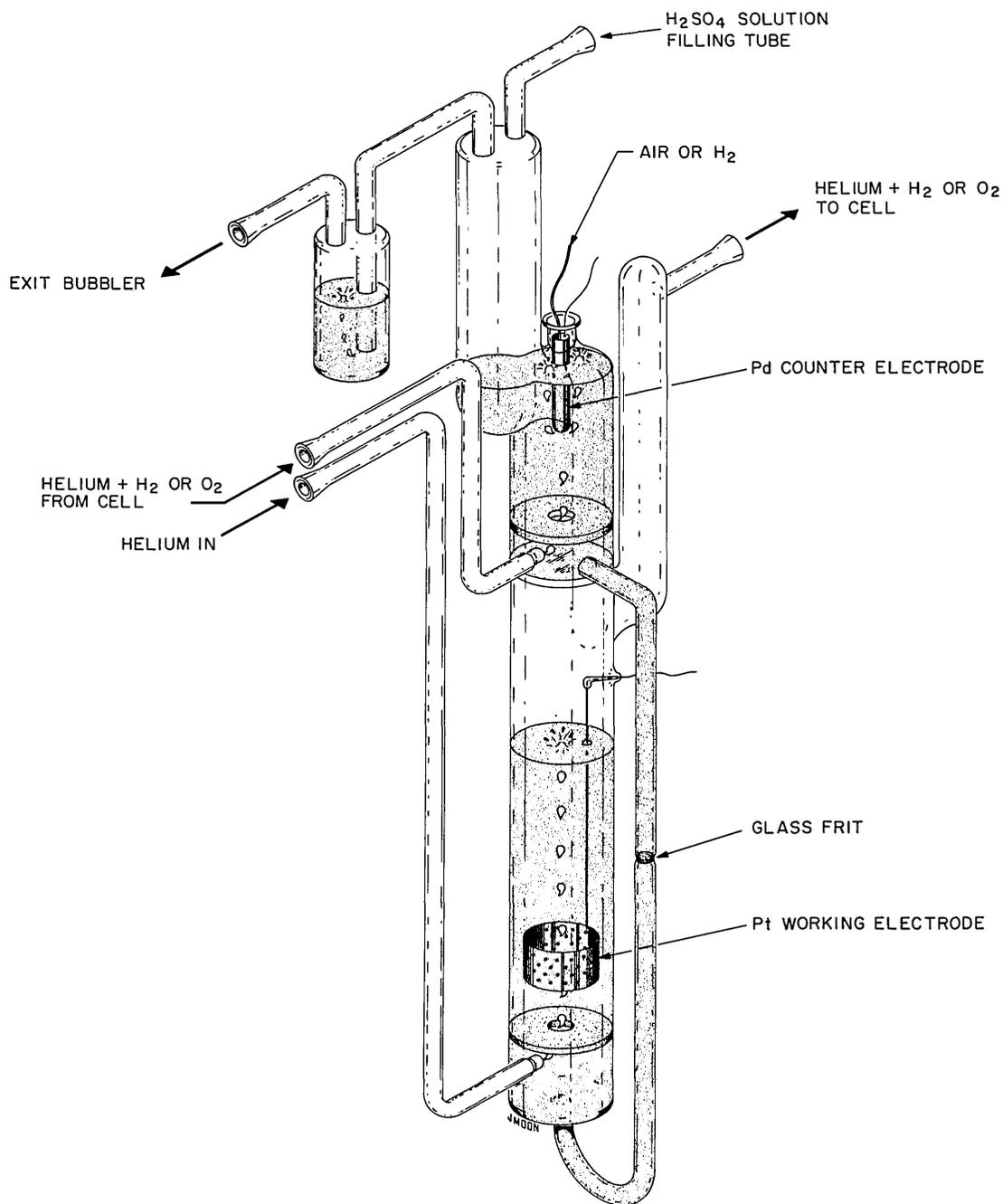


Fig. 1 - Electrolytic  $\text{H}_2$  or  $\text{O}_2$  generator

## RESULTS AND DISCUSSION

### Experimental Results

The experimental results are shown in Fig. 2. Values were obtained with increasing and decreasing gas partial pressures and were accepted after a steady state had been attained (usually within  $\pm 10$  mv). Results for  $\text{O}_2$  pressures above  $10^{-4}$  atm confirm

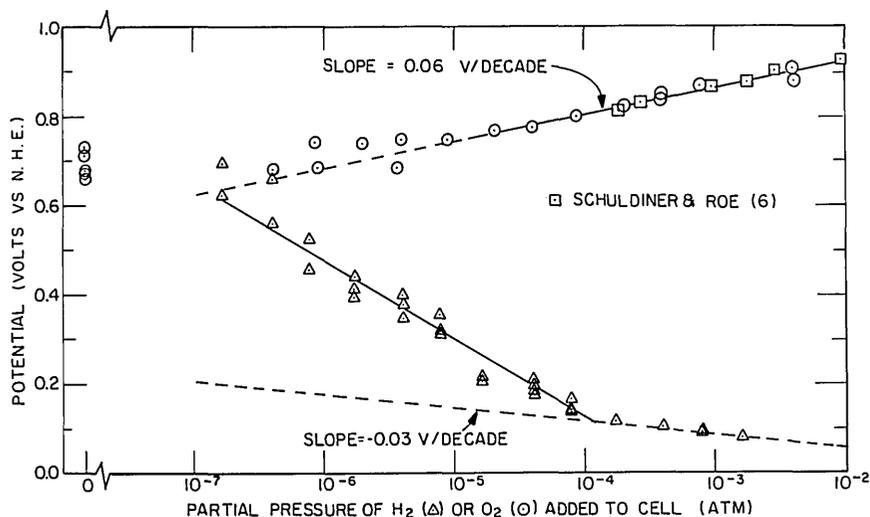


Fig. 2 - Effects of H<sub>2</sub> and O<sub>2</sub> partial pressure on Pt rest potentials. The points are experimental results. In the H<sub>2</sub> region, the dashed line gives reversible H<sup>+</sup>/H<sub>2</sub> potentials as predicted by the Nernst equation. In the O<sub>2</sub> region, the dashed line is an extrapolation of the linear region found at higher pressures.

previous work from this Laboratory (6). Potentials under H<sub>2</sub> at partial pressures less than 10<sup>-4</sup> atm deviate markedly from those predicted for the reversible H<sup>+</sup>/H<sub>2</sub> couple by the Nernst potential equation. Before interpreting these data, however, the response time, extent, and probable effects of O<sub>2</sub> leaking into the system from its surroundings must be considered.

#### Response Time

The time required to come to a true steady state was variable, depending on the H<sub>2</sub> or O<sub>2</sub> partial pressure, the previous gas partial pressure at which the electrode was equilibrated, and the surface-to-volume ratio of the electrode. The time increased as the surface-to-volume ratio decreased, as partial pressure decreased, and as the change from previous pressure increased. Periods of several days were required at the lowest pressures and three or more days for open-circuit potentials under pure He (i.e., He containing only that O<sub>2</sub> leaking into the system). False, apparently steady state, potentials were often observed for many hours. They were always between the last potential obtained and the new one being approached.

The above behavior could consistently be interpreted, by assuming that for an open-circuit potential to represent truly steady state conditions the amount of H<sub>2</sub> or O<sub>2</sub> dissolved in the Pt must be allowed to come to equilibrium with the amount at the surface. The long times required for this, and the surface/volume effect, imply that the partition must ultimately be between gas dissolved in the bulk of the metal and gas on the surface. If sufficient time was allowed, all three electrodes invariably came to the same potential within ±5 mv. These results then, coupled with earlier results (2), suggest that dermasorbed gases can exchange relatively rapidly with the surface, and that gases dissolved in the bulk of the metal must exchange slowly with the dermasorbed layer (and thus with the surface).

Effect of O<sub>2</sub> Leakage

For the case of O<sub>2</sub> added to the He, any O<sub>2</sub> leak adds to the partial pressure of O<sub>2</sub> actually in the system. The case of O<sub>2</sub> leaking into a system to which H<sub>2</sub> is being added is more complex. Very little is known about the effects of trace amounts of O<sub>2</sub> in H<sub>2</sub> on the potential of a Pt electrode, but an estimate may be made by assuming that any O<sub>2</sub> at the Pt surface will react with H<sub>2</sub> until only an effective net excess of H<sub>2</sub> remains. Since reaction of H<sub>2</sub> with O<sub>2</sub> could take place only at the platinum surface, the H<sub>2</sub> concentration would be reduced only at or very near to the Pt-solution interface. An "effective" H<sub>2</sub> partial pressure, i.e., the pressure that would result in the same electrode potential in the absence of a leak, was calculated by assuming Henry's law and complete reaction of O<sub>2</sub> and H<sub>2</sub> present in solution at the interface. The net excess concentration of H<sub>2</sub> in solution was used to calculate the pressure of H<sub>2</sub> in the gas phase that would be in equilibrium with it in the absence of any leak, and this pressure was used as the effective H<sub>2</sub> partial pressure.

$$(P_{H_2})_{\text{effective}} = \frac{\alpha_H (P_{H_2})_{\text{added}} - 2\alpha_O (P_{O_2})_{\text{leak}}}{\alpha_H} \quad (1)$$

where  $\alpha_H$  and  $\alpha_O$  are the solubilities of H<sub>2</sub> and O<sub>2</sub> in 1M H<sub>2</sub>SO<sub>4</sub> at 25°C, taken as 0.0149 and 0.023 std cc/ml H<sub>2</sub>SO<sub>4</sub>, respectively (8). (At H<sub>2</sub> generation rates lower than the O<sub>2</sub> leak rate a net excess of O<sub>2</sub> resulted.)

Lorch (9) studied chemical polarization of the H<sub>2</sub> electrode by O<sub>2</sub>, and his experimental data were used to check the assumptions implicit in Eq. (1). The results, given in Table 1, show agreement within 0.5 mv for H<sub>2</sub> partial pressures near 1 atm. In the next section we assume that this model also applies at very low partial pressures of both O<sub>2</sub> and H<sub>2</sub>.

Table 1  
Polarization of the H<sub>2</sub> Electrode by O<sub>2</sub>

P <sub>O<sub>2</sub></sub> * in Gas Phase (atm)	(P <sub>H<sub>2</sub></sub> ) <sub>effective</sub> † Calculated (atm)	E (mv vs N.H.E.)	
		Experimental	Calculated ‡
0.0092	0.97	0.8	0.4
0.0161	0.94	1.3	0.8
0.0252	0.89	1.9	1.5
0.0547	0.77	3.8	3.4
0.100	0.59	7.1	6.9

\*Lorch (9), mixture with H<sub>2</sub>, total pressure 1 atm.

†Calculated using Eq. (1).

‡Calculated from the Nernst equation,  $E = -0.0295 \log (P_{H_2})_{\text{effective}}$ .

Extent of O<sub>2</sub> Leakage

The potential behavior of the Pt electrode was a sensitive indicator of the background O<sub>2</sub> concentration. The fact that addition of H<sub>2</sub> to the He stream sufficient to give a partial pressure of  $4 \times 10^{-7}$  atm H<sub>2</sub> caused the potential to drop ~0.1 v indicated

that the  $P_{O_2}$  could not have been much larger than this. Furthermore, plotting effective  $H_2$  pressures versus observed potential under the assumption of  $O_2$  leaks of  $10^{-6}$  and  $10^{-7}$  atm (Figs. 3 and 4) gives an unreasonable picture when  $10^{-6}$  atm\* is assumed and organizes the data coherently when  $10^{-7}$  atm is assumed.

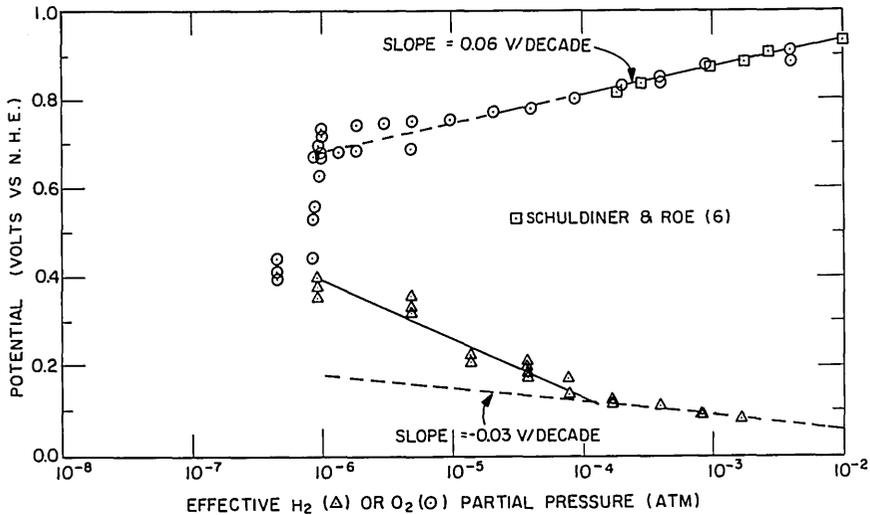


Fig. 3 - Platinum rest potentials vs  $H_2$  or  $O_2$  partial pressures corrected for an  $O_2$  leak of  $1 \times 10^{-6}$  atm

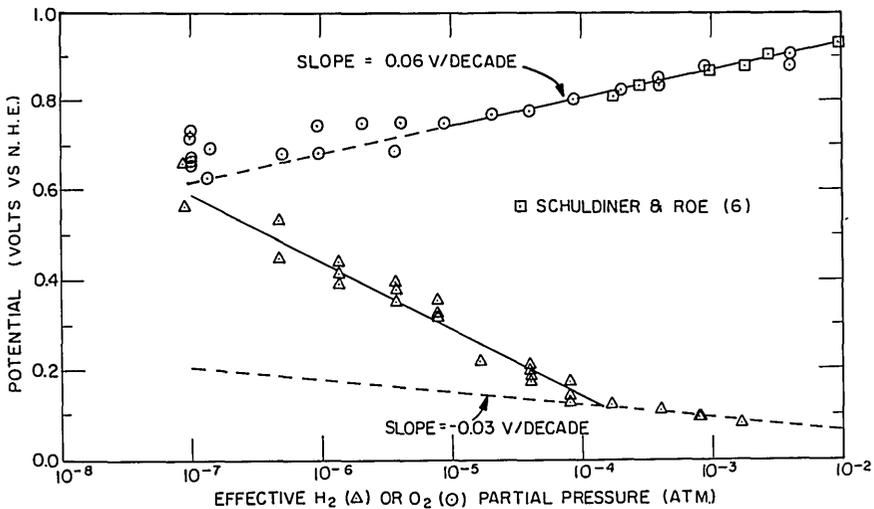


Fig. 4 - Platinum rest potentials vs  $H_2$  or  $O_2$  partial pressures corrected for an  $O_2$  leak of  $1 \times 10^{-7}$  atm

\*For example, the corrections made in Fig. 3 compress potential values between 0.45 and 0.75 v into the range of partial pressures between  $0.85$  and  $1.00 \times 10^{-6}$  atm. In this region the individual points should have been erratic—in fact, they were very steady and fairly reproducible as shown in Fig. 2.

The above estimate based on electrochemical evidence is in reasonable agreement with the  $10^{-6}$  to  $10^{-8}$  atm  $O_2$  partial pressure due to leaks that would be estimated a priori within such a flowing system, since Viton-A O-rings are suitable for use in vacuum systems operating in the  $10^{-6}$ -torr region. It was also confirmed by analyses using a modified CED 21-103c mass spectrometer that indicated that  $O_2$  concentration in the nominally pure He flowing through our system was less than 1 ppm.\*

#### Pt Rest Potentials versus $P_{H_2}$ or $P_{O_2}$

The preceding discussion indicates that Fig. 4 most closely represents the potential behavior of these systems. Clearly, a reversible  $H^+/H_2$  couple cannot control electrode potential below  $10^{-4}$  atm  $H_2$ . What does control potential is, at present, a moot question. It seems quite unlikely that an  $O_2$  leak of the order of  $10^{-7}$  atm could be responsible for this potential behavior at  $H_2$  partial pressures several orders of magnitude larger. Schuldiner (10) showed that the relationship between exchange current density  $i_0$  and  $P_{H_2}$  was

$$i_0 = 1.43 \times 10^{-3} P_{H_2} (1 - \theta_0)^2, \quad (2)$$

where  $\theta_0$  is the fraction of available Pt surface covered with atomic hydrogen at  $P_{H_2}$ . At low  $H_2$  partial pressures,  $\theta_0$  will undoubtedly be very small compared to unity and for all practical purposes  $(1 - \theta_0)^2$  will approach unity. Hence, at  $P_{H_2} = 10^{-4}$  atm,  $i_0 \approx 1.4 \times 10^{-7}$  amp/cm<sup>2</sup>, and at  $P_{H_2} = 10^{-7}$  atm,  $i_0 \approx 1.4 \times 10^{-10}$  amp/cm<sup>2</sup>. If a mixed potential involving  $H^+/H_2$  and, say, the  $O_2/HO_2$  (7) reactions were potential determining,  $i_0$  for the  $O_2/HO_2$  reaction would have to be of the same order of magnitude as the  $H^+/H_2$  reaction. It is very unlikely that the  $O_2/HO_2$  or any other possible potential-determining reaction involving  $O_2$  at  $10^{-7}$  atm would have an  $i_0$  comparable to the  $H^+/H_2$  reaction. All literature indications are that any equilibrium reactions on Pt involving  $O_2$  at 1 atm have  $i_0$  values of not more than  $10^{-10}$  amp/cm<sup>2</sup>.

It is also unlikely that these potentials are caused by polarization by the electrometer ( $10^{14}$  ohms shunted by  $10 \mu\mu\text{f}$ ), for then one would expect an even more marked effect on the presumably more easily polarized Pt/ $O_2$  system. In addition, electrometer measurements were the same for electrodes which were not permanently connected and for which potential measurements were made in a few minutes after connecting the electrometer circuit.

It should be stressed that the unexpected relation between potential and  $H_2$  partial pressure below  $10^{-4}$  atm is an experimental fact and does not depend on any assumptions about how  $H_2$  may interact with  $O_2$ . The same kind of behavior would be expected in any real system from which  $O_2$  is imperfectly excluded. It is believed that the deviation from Nernst behavior may be due to the rather complex sorption of  $H_2$  on Pt (5), where even at 1 atm of  $H_2$  there are only a small number of weakly bonded H atoms which are in rapid equilibrium with  $H_2$  and  $H^+$  (10,11).

\*Such a concentration is at or below the lower detection limit of the instrument. The amount of  $O_2$  was determined from repeated measurements and blanks, with due consideration of wall effects. Interestingly, the mass spectrometer data supported the view that  $O_2$  entering the system did so by diffusion through the Viton O-rings, for the  $N_2/O_2$  ratio was about 3. The  $N_2/O_2$  ratio in the He as received was about 6, and there was no reason to assume equal diffusion rates for  $N_2$  and  $O_2$  through the rings. If a gross leak existed, the ratio should have been 4; and if the  $O_2$  resulted from inadequate purification of the He, the ratio should have been at least 6, since that was the ratio in the unpurified He and since the purification train was designed specifically to remove  $O_2$ .

It is interesting that the Pt/O<sub>2</sub> system fits a Nernst relation at substantially lower O<sub>2</sub> partial pressures than does Pt/H<sub>2</sub>. The O<sub>2</sub> rest potential follows the same Nernst relation previously obtained at this Laboratory (6). These results similarly indicate a potential-determining equilibrium



assuming a constant activity of HO<sub>2</sub> adsorbed on the Pt surface.

The open-circuit potentials for "pure" He, i.e., at 10<sup>-7</sup> atm O<sub>2</sub> in Fig. 4, are somewhat scattered. This may be the result of an extremely long time being required for establishment of true equilibrium between absorbed, dermasorbed, and chemisorbed O<sub>2</sub> at such low partial pressures. Also, small variations in the O<sub>2</sub> leak from time to time are possible. These values differ from the previously reported values of 0.2 to 0.4 v (6). The reason for the discrepancy may be that in the previous work a Pd-tube electrode at about 50 mv (N.H.E.) was present in the cell. We have observed that at very low O<sub>2</sub> partial pressures such a Pd electrode will cause the Pt electrode to become less positive. The Pd may either have contributed a small amount of H<sub>2</sub> to the cell or acted as an effective O<sub>2</sub> getter to give O<sub>2</sub> partial pressures virtually equal to zero. A combination of these two effects is also possible. The effect of H<sub>2</sub>-containing Pd at very low O<sub>2</sub> partial pressures requires further investigation.

#### ACKNOWLEDGMENTS

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nernst equation Platinum electrode Hydrogen electrode Oxygen Low partial pressure Oxygen leakage Dermasorption						

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