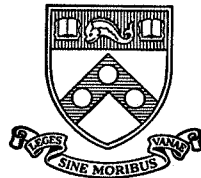FOURTH QUARTERLY REPORT

1 OCTOBER 1969 to 30 DECEMBER 1969

STUDIES IN FUNDAMENTAL CHEMISTRY
OF FUEL CELL REACTIONS

NGR 39-010-002

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UNIVERSITY OF PENNSYLVANIA

ELECTROCHEMISTRY LABORATORY

PHILADELPHIA, PENNSYLVANIA 19104

FOURTH QUARTERLY REPORT

1 OCTOBER 1969 to 3 DECEMBER 1969

STUDIES IN FUNDAMENTAL CHEMISTRY
OF FUEL CELL REACTIONS

NGR 39-010-002

Submitted to:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546

Submitted by:

Professor John O'M. Bockris

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University of Pennsylvania
Philadelphia 19104

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SECTION 1

Woon-Kie Paik

J. O'M. Bockris, Supervisor

Title of project: Adsorption of anions at the solid-electrolyte interface: an ellipsometric study.

Long-term aims: Measurement of ionic adsorption on solid electrodes.

Investigation of the nature of the adsorption forces.

Specific aims for this period: Final interpretation of the ellipsometric data for ionic adsorption. Discussions on adsorption forces. Writing a paper for publication.

Results of work in this period: The optical model developed in the last period for the interpretation of the ellipsometric observations on the adsorption system was refined further. The correction for the "electromodulation" effect of the optical properties of a metal surface was also recalculated in this period, with slightly different approaches from those adopted in the last period. These revisions were necessary to clarify the physical basis of the model and to minimize the number of assumptions.

The experimentally observed ellipsometric parameter $\delta\Delta_{obs}$ was equated to the sum of the direct adsorption effect and the "electromodulation" effect:

$$\delta\Delta_{obs} = \delta\Delta_{ads}(q_-) + b\delta q_M .$$

1

The coefficient b is calculated using the free electron model of metal and the ellipsometric equations. Then q_- (the amount of anions adsorbed) was calculated from $\delta\Delta_{ads}$ using the ellipsometric equations and a modified Lorenz-Lorentz equation for the refractive index of the double layer.

The results of the final analysis are shown in Figures 1-7.

Some of the features of these results are:

1. Anions adsorb on different metals to markedly different degrees (Fig. 6).

2. The potential (with respect to NHE) of different metals at which adsorption starts increases in the order:

$$Ag < Au < Ni < Hg < Pt < Rh$$
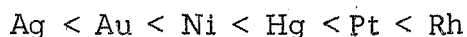
3. Adsorbability of anions as measured by $(\frac{\partial q_-}{\partial V})_c$ or by $(q_-)_V$ increases in the order:

$$ClO_4^- < SO_4^= < Cl^- < Br^- \ .$$
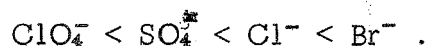
4. At low degrees of adsorption, there is usually exponential rise of adsorption, with increasingly positive potential, followed by inflections of the curve.

5. q_- plotted against $\log c$ follows straight lines in most systems.

6. For less adsorbing ions (ClO_4^- and $SO_4^=$), the value of $(\frac{\partial q_-}{\partial \log c})_V$ is small and increases with increasingly positive potential and with adsorbability of anions. For strongly adsorbing anions (Cl^- and Br^-), $(\frac{\partial q_-}{\partial \log c})_V$ is large and either increases or decreases with potential.

The potential dependence of the adsorption on different metals is noted to be related to the pzc's of the metals, as in Figure 7, with

Fig. I Adsorption of anions on Ni and Rh
electrode
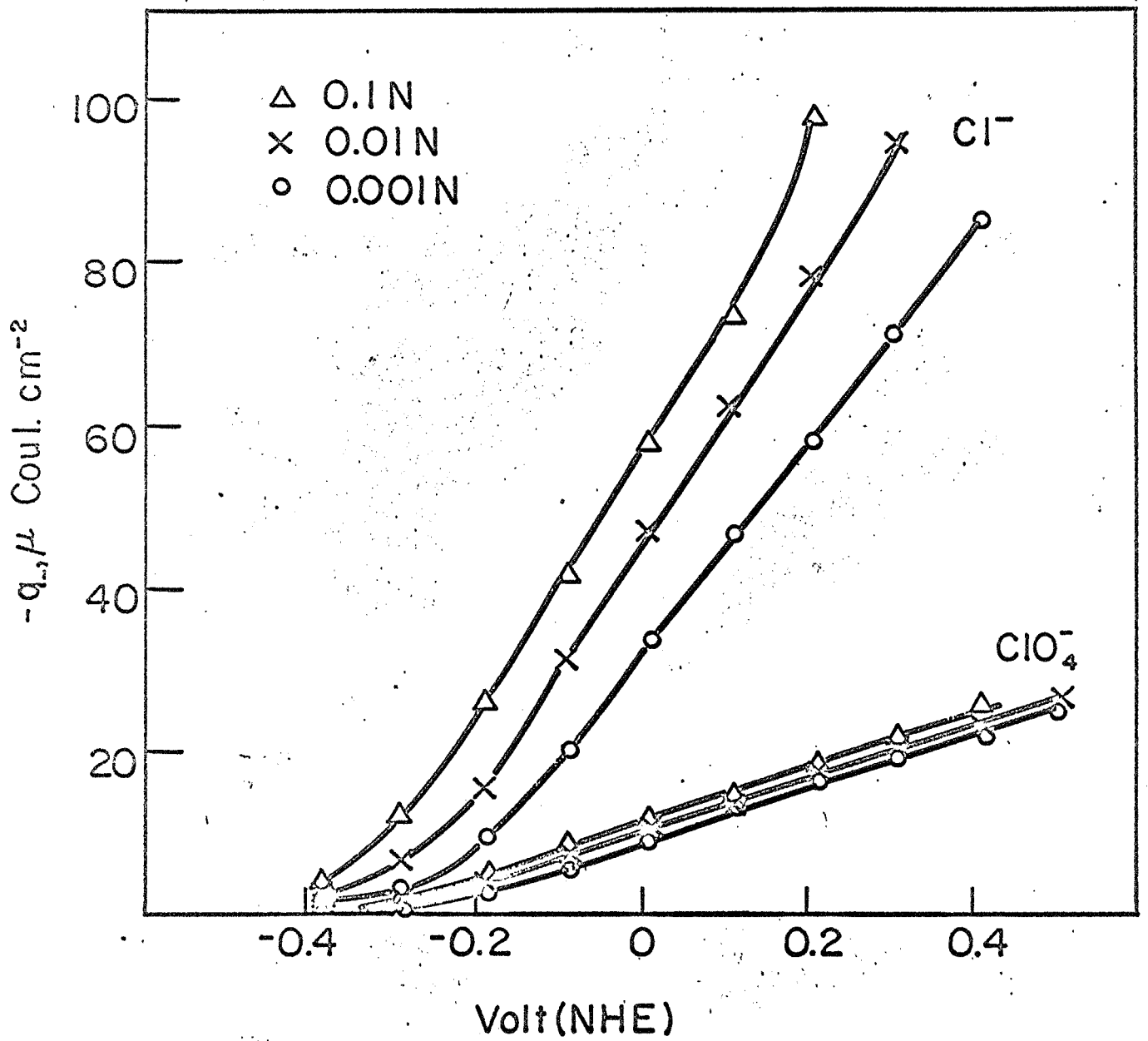
Fig. 2 Adsorption of Cl^- and ClO_4^- on Ag electrode

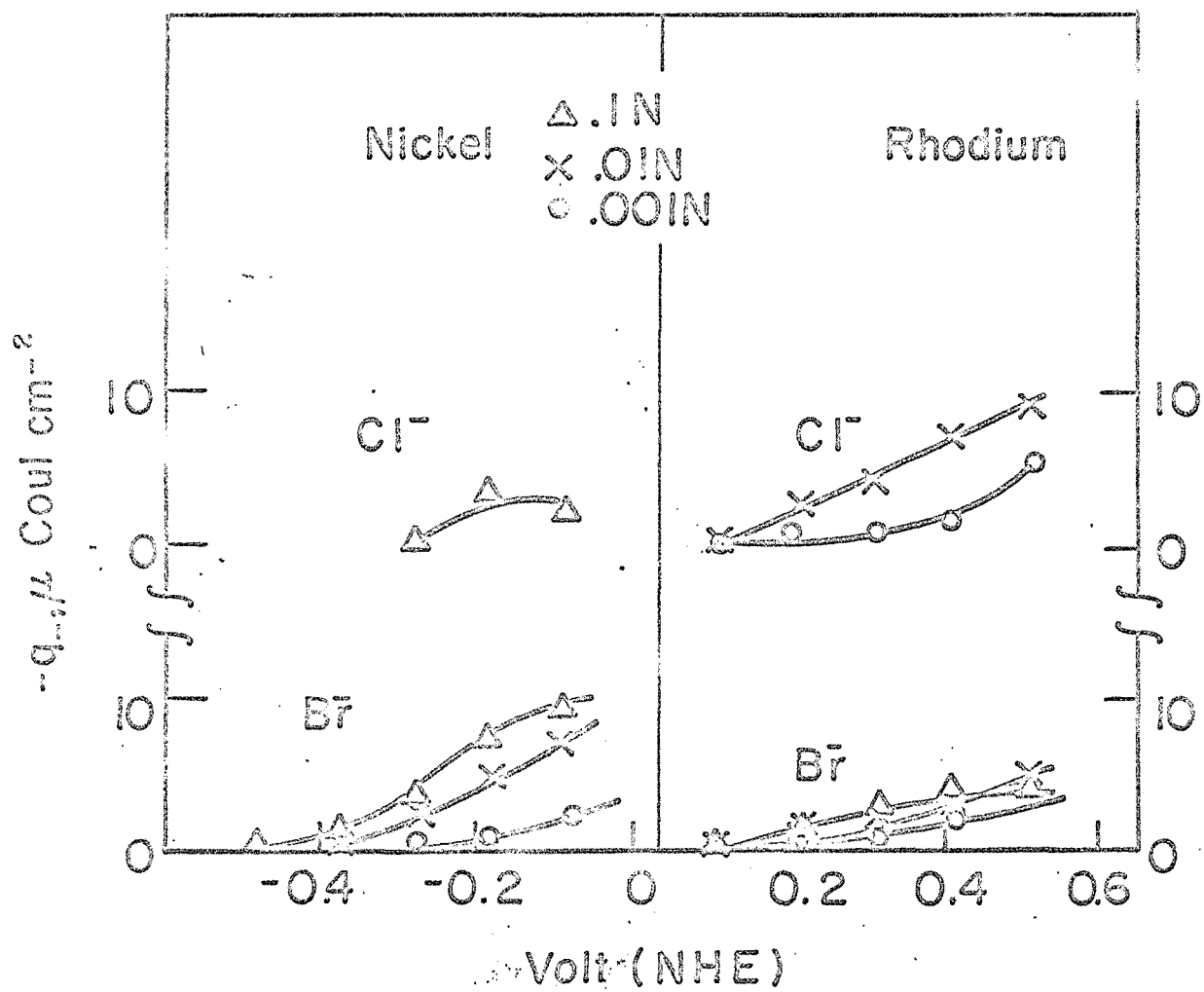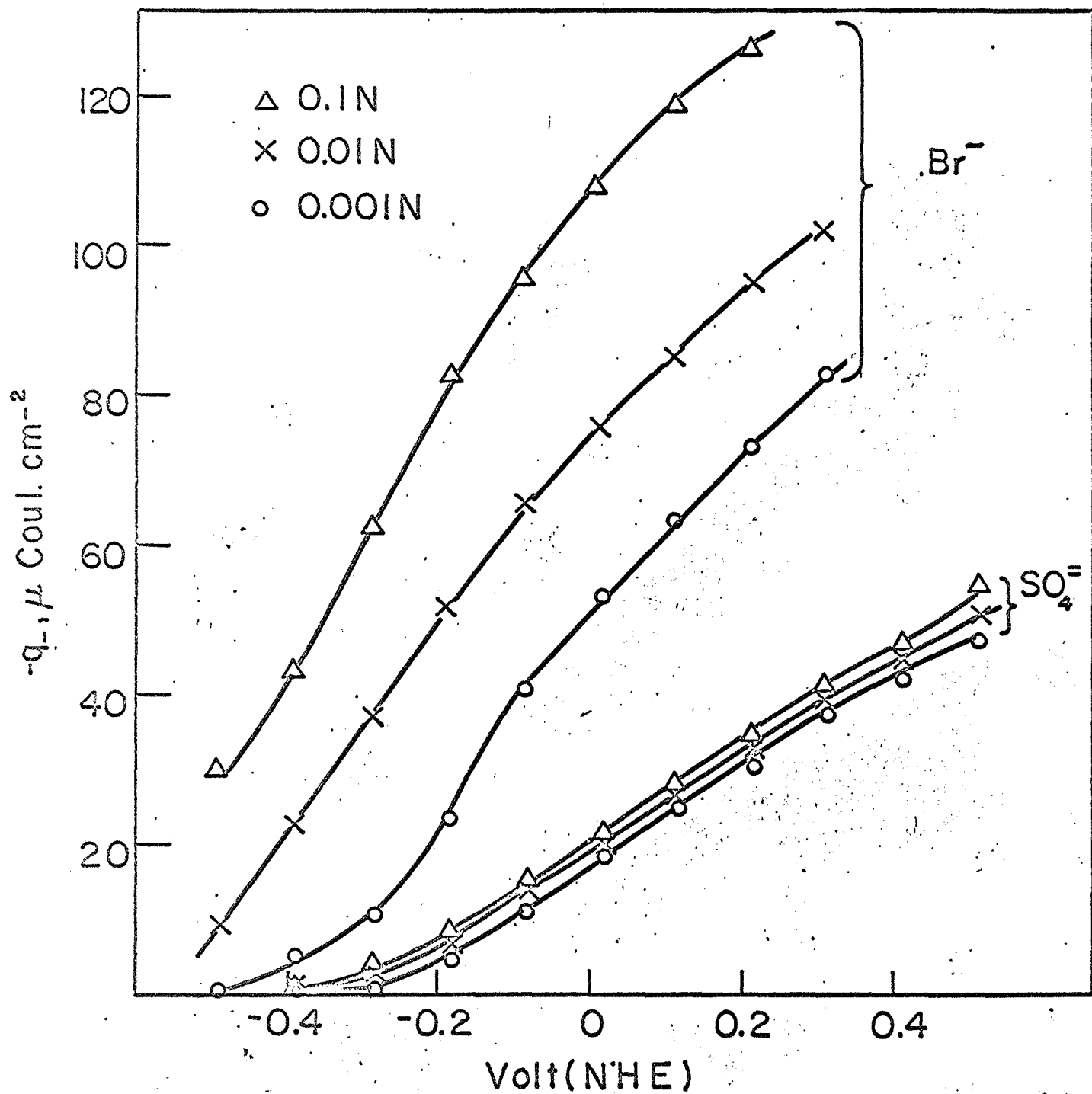Fig.3 Adsorption of Br^- and $SO_4^=$ on Ag electrode
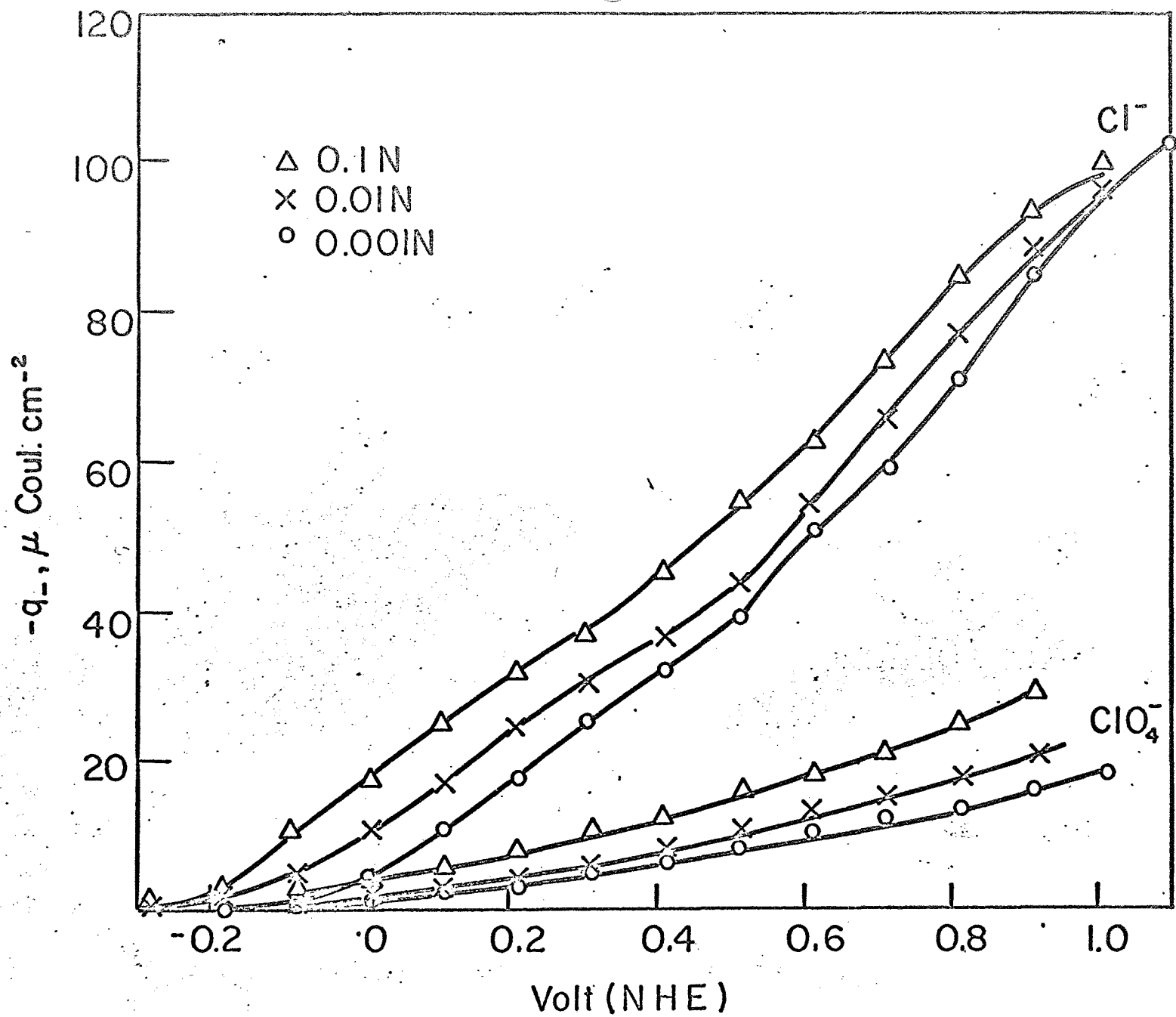
Fig. 4 Adsorption of Cl⁻ and ClO_4^- on Au
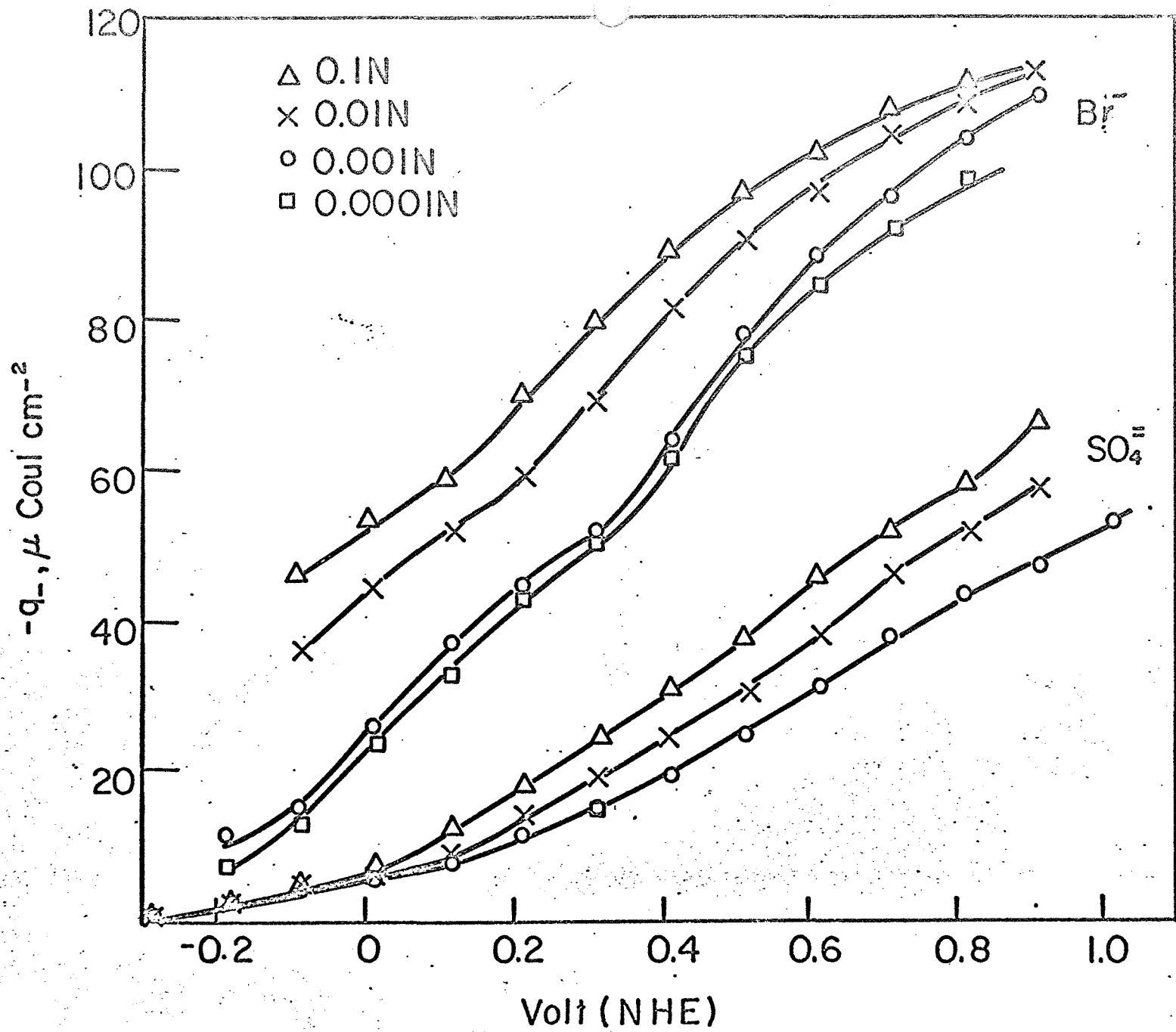
Fig.5 Adsorption of Br^- and $SO_4^=$ on Au
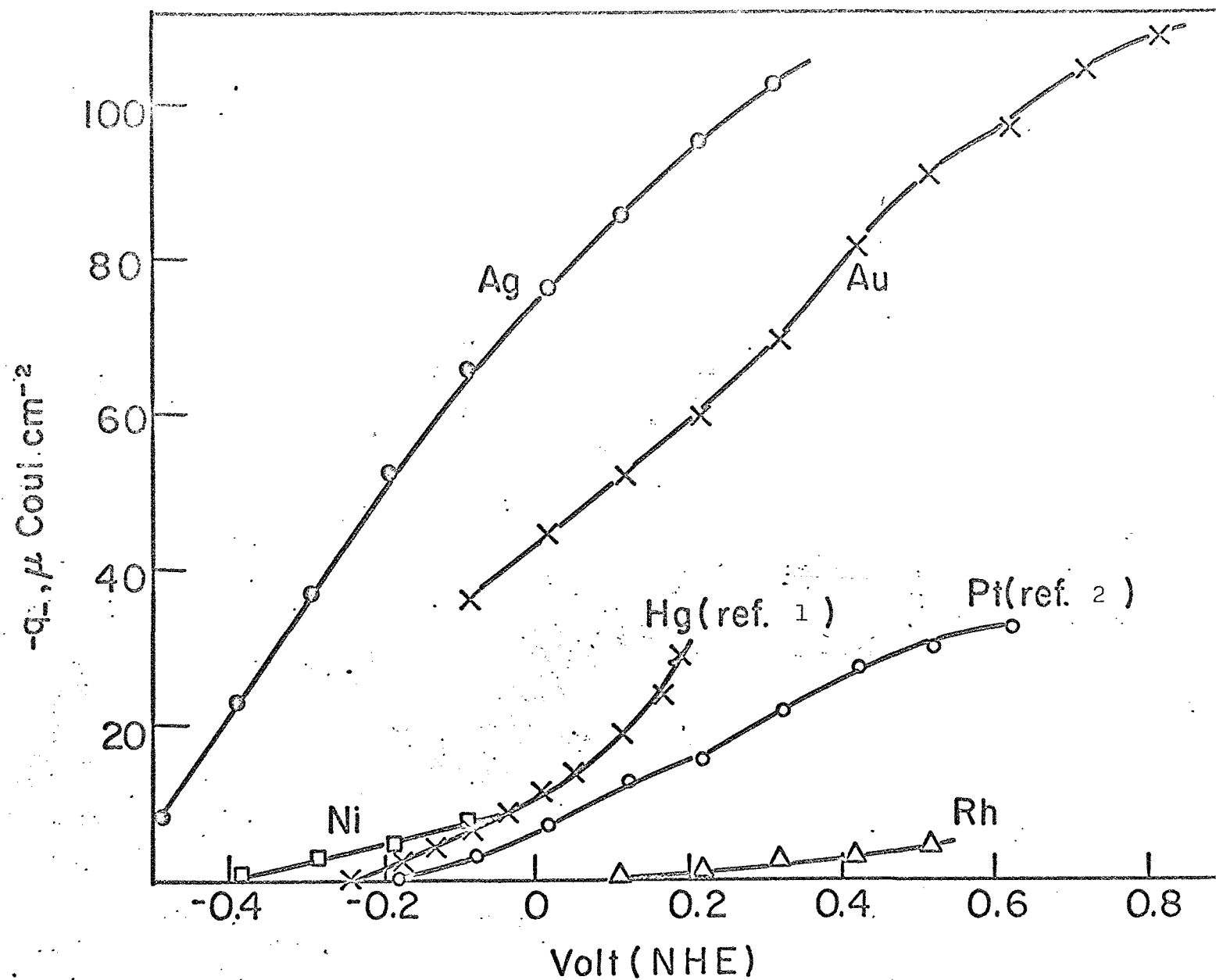
Fig. 6 Adsorption of Br⁻ from $\frac{1}{100}$ N Br⁻ solution on metals.
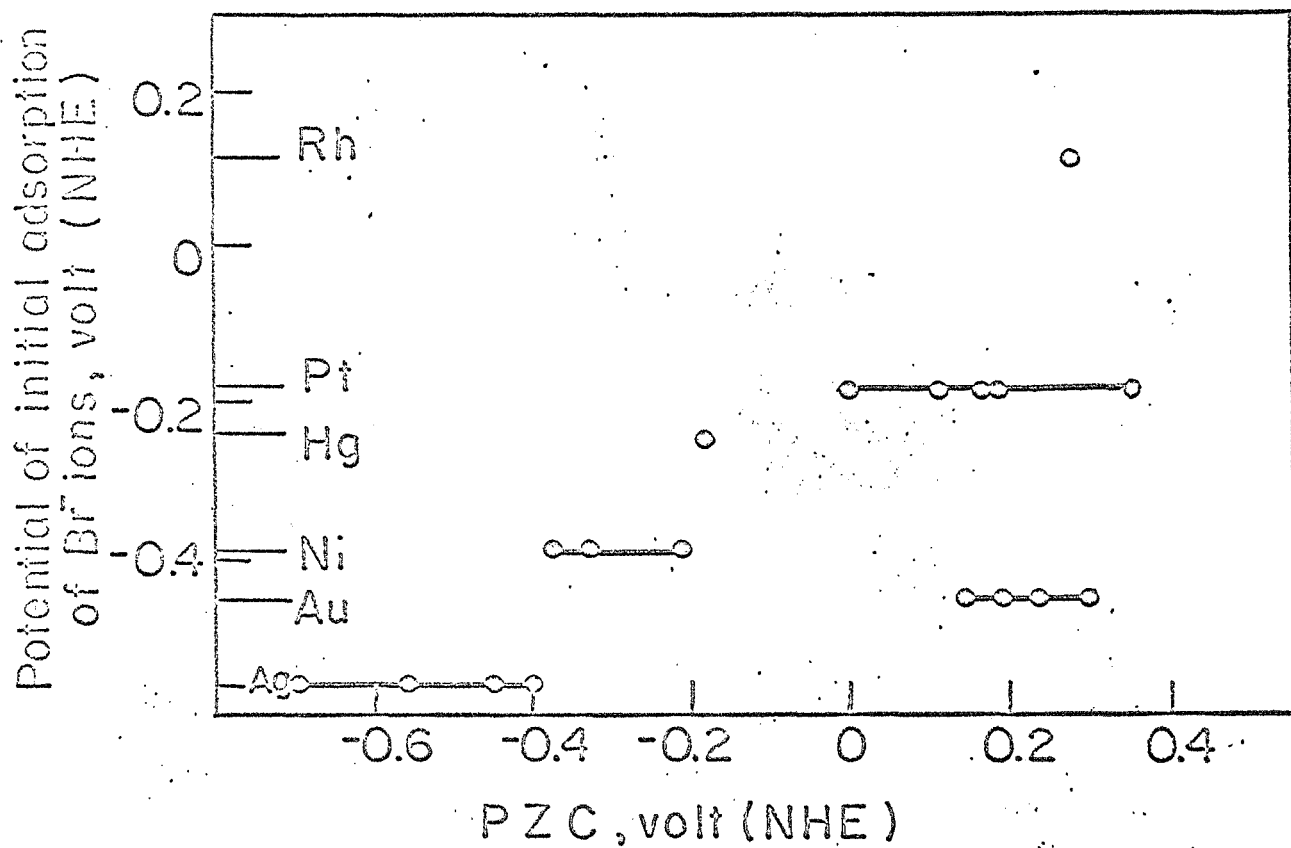
Fig. 7 Potential of initial adsorption of Br⁻ compared with PZC

the exception of gold and possibly platinum.

A paper has been written on the basis of the above results and is ready to be submitted for publication.

Specific aims for
next period:

Study of the kinetics of ion adsorption. Ellipsometric fast transients will be measured for the adsorption of anions on solid metals.

This experiment will result in the separate rate constants of diffusion-controlled and migration-controlled adsorption processes and the "electro-modulation" effect. The relative contributions of the above processes to the ellipsometric parameters will be examined.

Electronic control devices of the electrochemical system and the electronic measuring devices for the rapidly changing optical responses are being developed for this purpose.

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Divna Cipris

J. O'M. Bockris, Supervisor

Title of project:	Reversibility of Organic reactions
Long-term technological aims:	Investigation of capability of organic compounds for use in high energy secondary batteries.
Specific aims for this period:	Preliminary testing of rhodizonic acid, RA (mixed with carbon black) as a cathode, coupled with Mg as an anode, under conditions similar to those in practical systems.
Results of work in this period:	0.5 g of RA mixed with carbon black and/or graphite (ratio 1:1 or 1:2), coupled with Mg (electrolyte 250 $g\ell^{-1}$ $MgBr_2$), was discharged through different resistors. Corresponding V-t and I-t curves are shown in Figures

1 and 2 (as an example). The testing cells in these experiments were

the U-shaped tube (anodic and cathodic compartments being separated by

fritted glass), or the Morehouse-Glicksman type of cell.[1] It has been

shown that the latter is not suitable for testing of RA, due to the relatively

high solubility of RA (up to 1 mole ℓ^{-1}). Mainly, RA gets dissolved in

the surrounding electrolyte, and the process becomes diffusion limited.

Also, the amount of RA which diffuses into the anodic compartment is lost

for the reaction. Therefore, the performance (see Table 1) of the RA-Mg
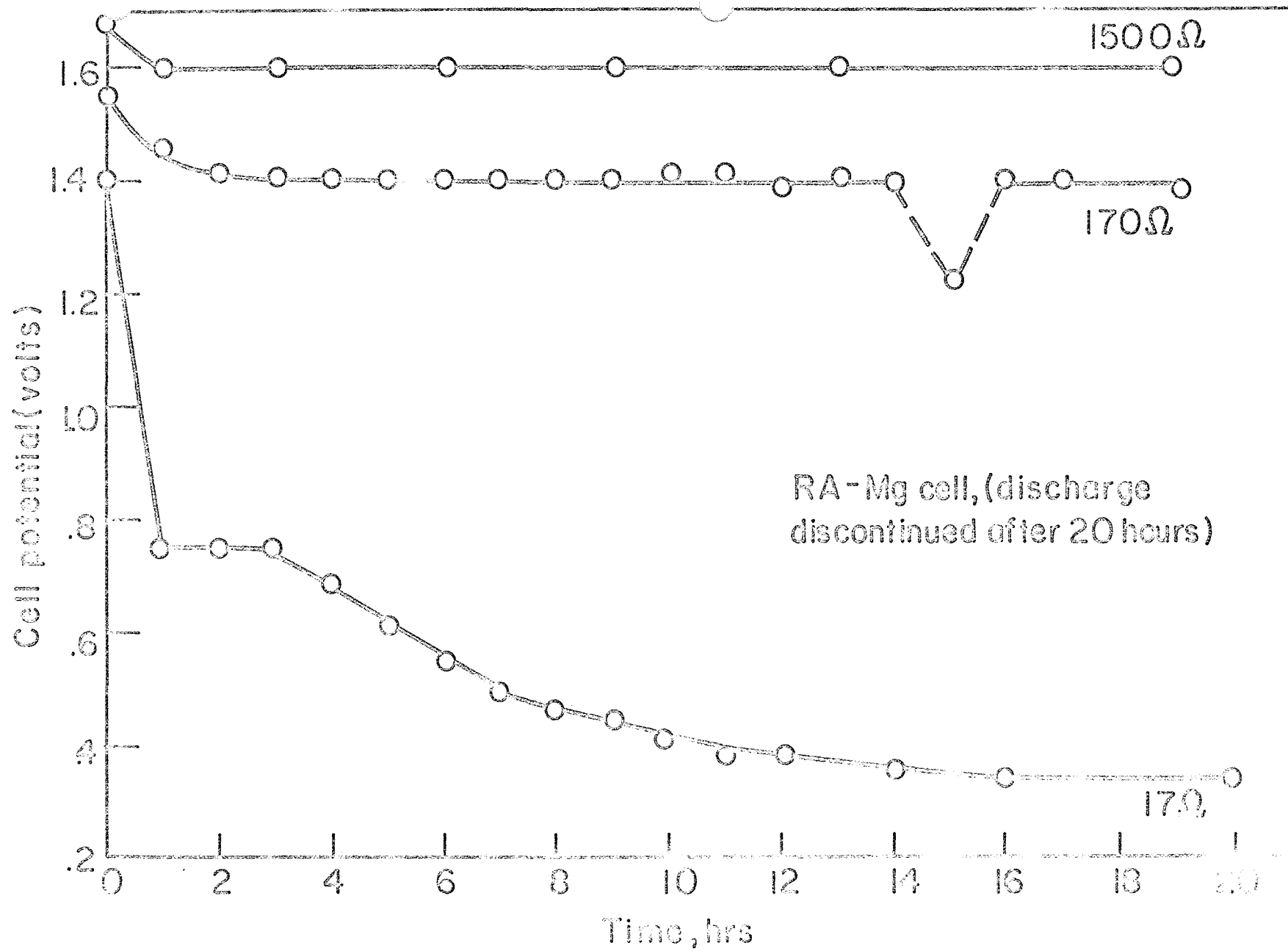
Fig. 1
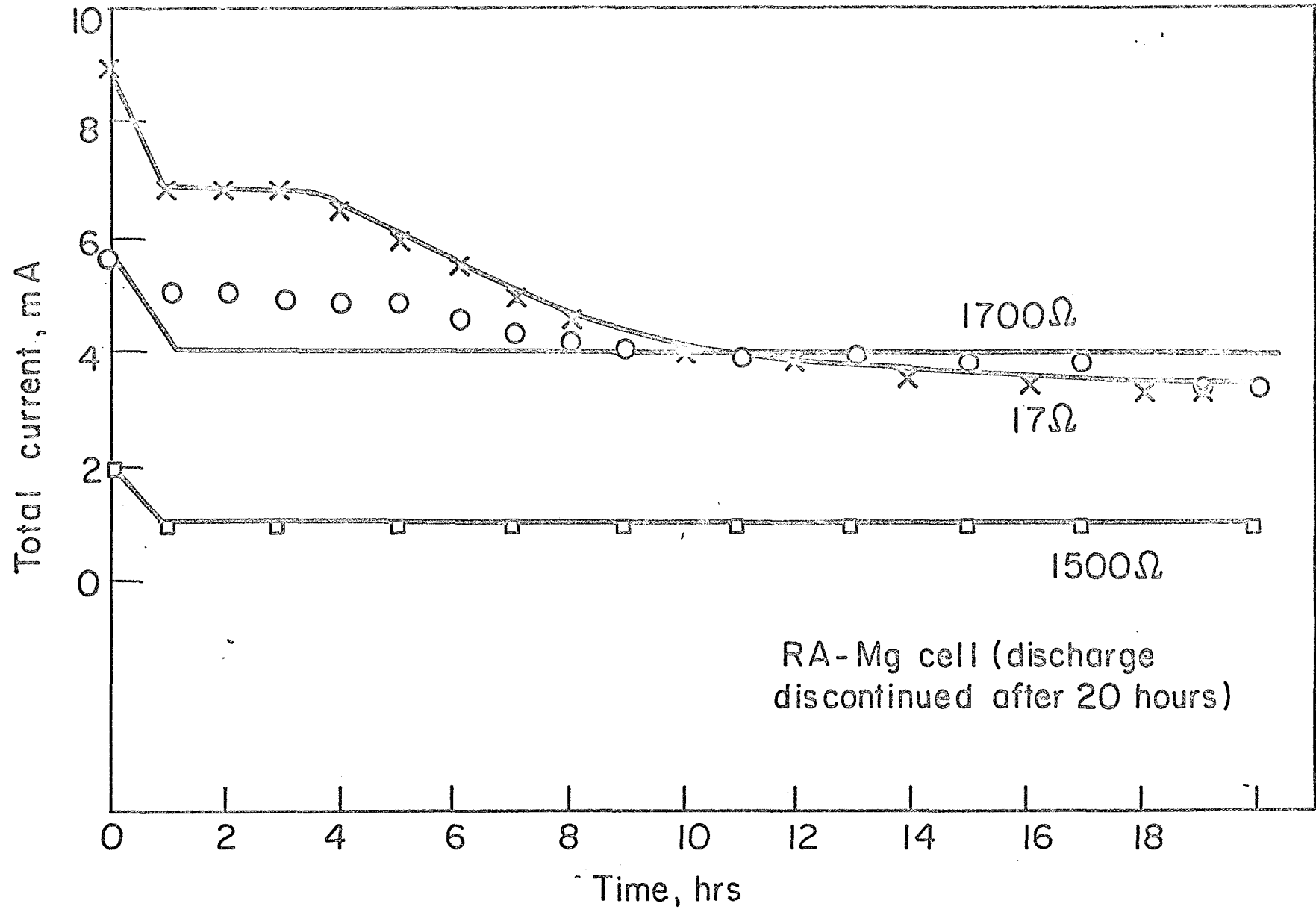
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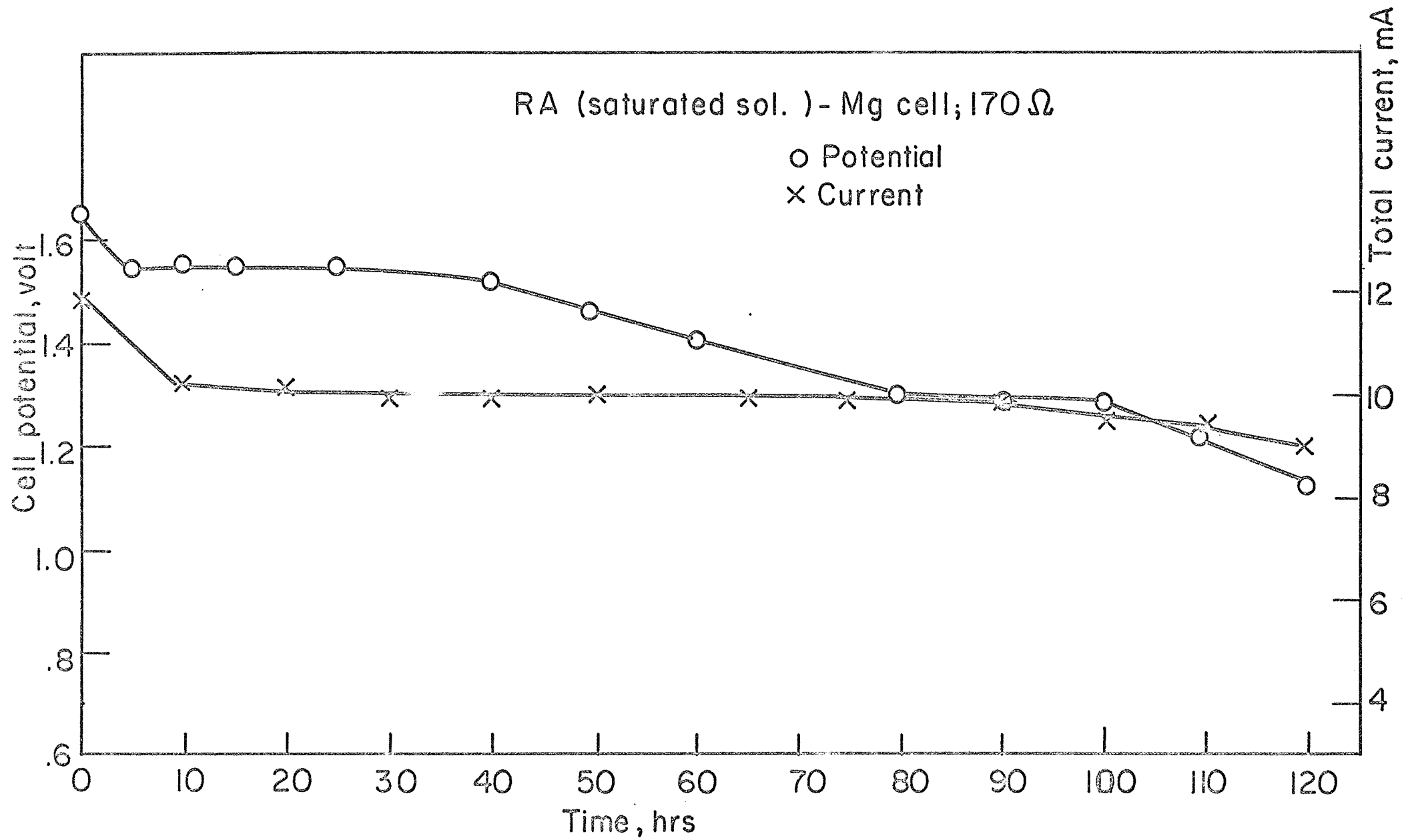
Fig. 3

TABLE 1. Discharge characteristics of RA-Mg couple
         through different resistors

R Ω	i mA	V volts	watt hrs/lb ideally	watt hrs/lb assuming 0.8 efficiency	Remarks
1500	1	1.6	395	316	)
					) Diffusion
170	4	1.4	345	275	(controlled
					) process
17	6	0.75	184	148	)

cell can be improved after these two limitations have been eliminated.

A new cell has been designed which will permit the testing of RA under the optimal conditions in both the discharge and the recharge process (the latter after replacing the electrode with Pt). The use of a cation-transfer membrane will be employed for separation of cathodic from anodic compartments. (Preliminary results with the cation-transfer membrane give better V-t and I-t characteristics, although the reaction is still under diffusion control; see Figure 3).

Specific aims for next report period: Testing of carbon paste RA electrode in discharge and charge processes (using the new cell). Determine coulometric efficiency of the reaction.

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1. C. K. Morehouse and R. Glicksman, J. Electrochem. Soc., 103, 94 (1956).

Gennadi A. Kokarev

J. O'M. Bockris, Supervisor

Title of project:	Investigation of the behavior of iron in alkaline solution
Long-term aims:	Inhibition of iron corrosion in batteries, etc.
Specific aims for this period:	Examination of anodic efficiency of iron electrode in iron-oxygen cell.
Results of work in this period:	The curves of Faradaic efficiency and self-discharge have been obtained in KOH and KOH + KCl solutions. The problems of the iron electrode can be summed up in the following way:

1. Passivation sets in during the discharge stage, and ferrous hydroxide does not form more than about 100-200 layers. This makes a high capacity difficult to obtain.

2. The difficulty in charging the battery arises because of the corresponding hydrogen evolution, and self-discharge, i.e., spontaneous corrosion with ensuing passivation of the iron.

Taking as a basis for our experiment, we calculated projected electrical and physical characteristics of an iron-oxygen single cell (weight one pound without electrolyte):

1. Nominal voltage   0.9 V

2. Capacity 40^{*} A/hr

3. Discharge time
   (for iron electrode)   22 hrs

4. Specific energy   20 watt hrs/lb

5. Energy density   9.5 watt hrs/in^3

*The factor of the use of active material --0.4

Specific aims   The examination of some organic compounds which
for next period:
                      would act as corrosion inhibitors of the iron.

                      Potentiostatic transient apparatus for measurements

                      of the surface coverage with organics will be set up.

R. Sen

J. O'M. Bockris, Supervisor

Oxygen-dissolution reaction — A theoretical study.

The rate determining step of the oxygen dissolution reaction was shown[1] to be

$$O_2 + H^+ + e \rightarrow OH_{ads} + O_{ads}$$
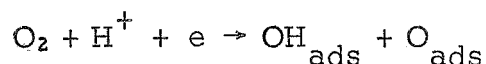
The objective of this project is to calculate theoretically the rate at which the above reaction will proceed and try to find out on what factors the important phenomena of electrocatalysis depends.

Very few attempts have been made to explain such a complex phenomena. In fact there are only two approaches worth mentioning. The Levich[2] approach though mathematically complex is physically questionable, whereas the Bockris-Mathews[3] approach is quite approximate. So it was decided, that in this problem, the essential features of the Bockris-Mathews approach will be kept, but an attempt will be made to obtain a more quantitative solution. In the present calculation we decided to consider the initial state to be made up of the electrode and the O_2 molecule physically adsorbed on it, and the H^+ attached to water molecules in the double layer. Due to bond stretching as well as fluctuation in the solvent the energy of the initial state varies with the distance between O and H^+ as shown in Fig. (1)(a). The final state consists of OH and O_{ads} on the electrode, (Fig. (1)(b). If the adsorption energy is calculated we can get energy as a function of M – O distance.
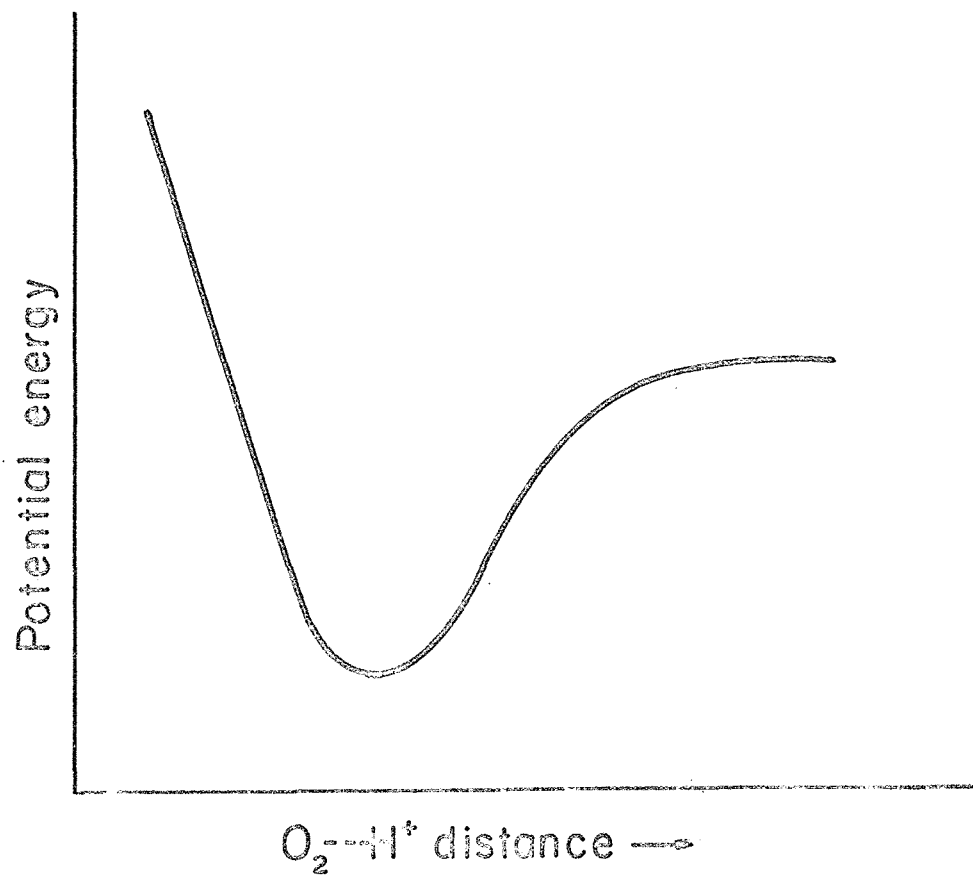
Fig. I(a) Variation of PE as a function of the O_2---H^+ distance
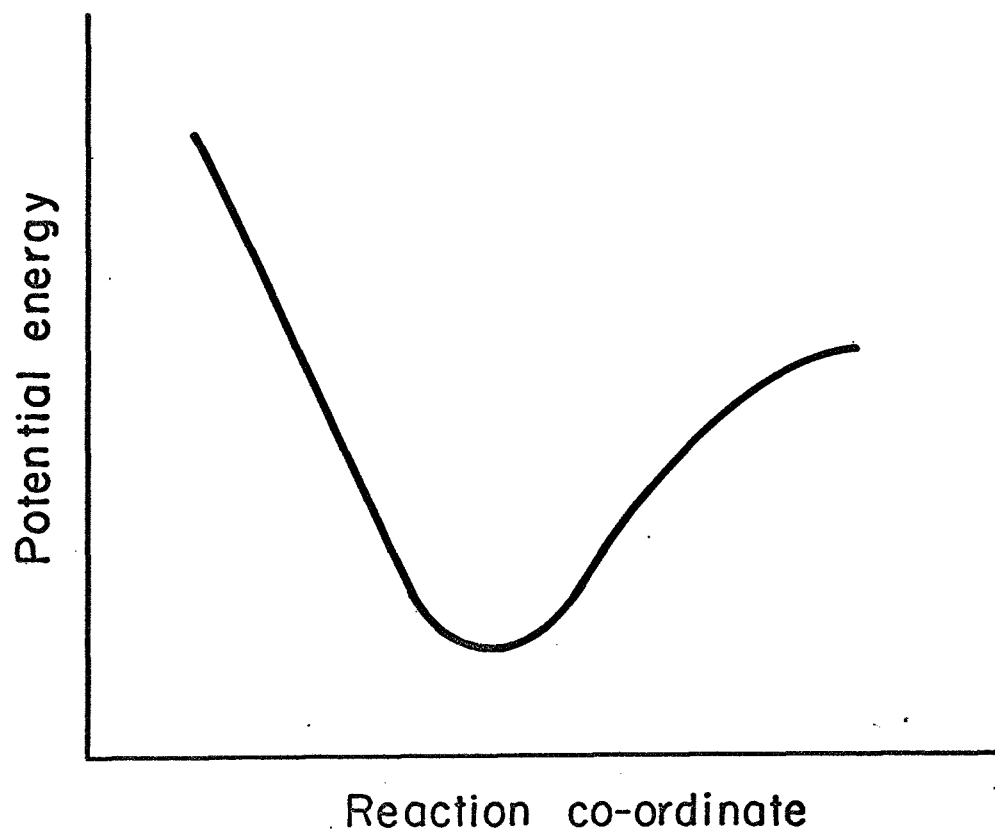
Fig. I (b) Variation of PE as a function of the reaction co-ordinate. This curve is synthesized from the two curve arising out of the variation in energy with M---O and M---OH bond distance.
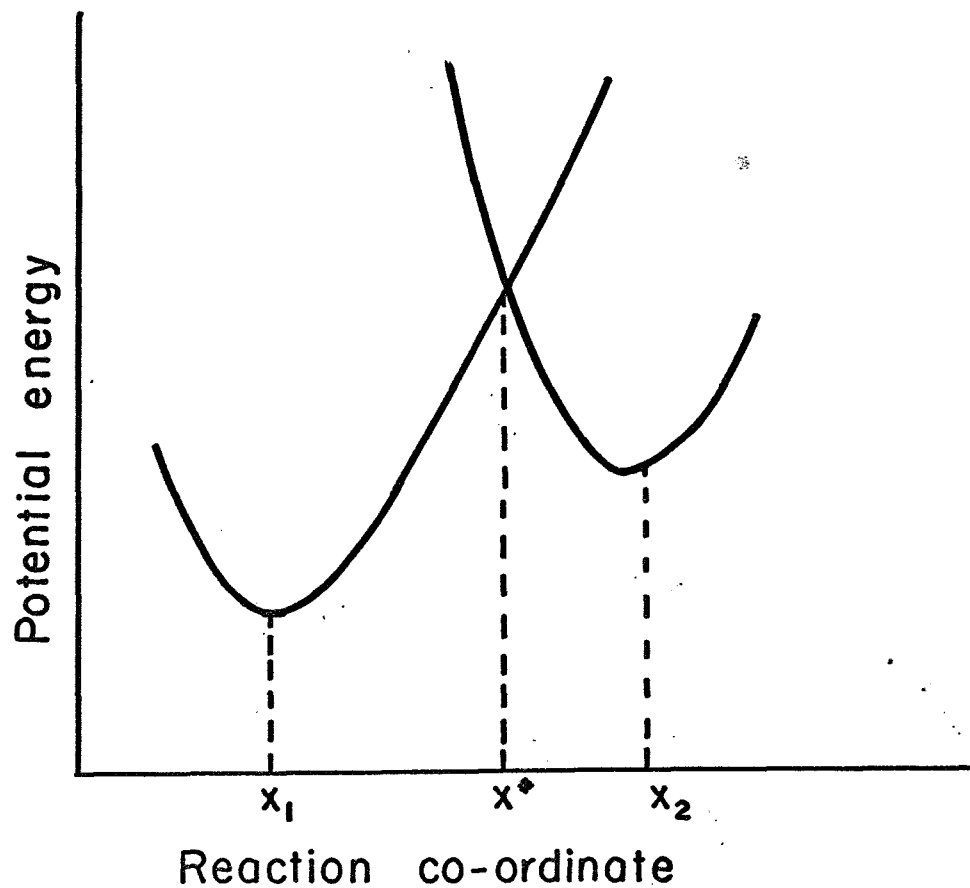
Fig.I(c) The curve on the RHS is the initial state curve, where the LHS are represents the final state.

The energy will be affected by lateral interaction with the water also
present on the electrode. If we put the energy distance curve of both the
initial and final states into the same plot, the two curves intercept, Fig. (1)(c). It
is at this point tunnelling occurs across a PE barrier. Thus, the problem
of tunnelling across an unsymmetrical barrier has to be solved.
Moreover, the tunnelling is occurring from a metal surface where for all
practical purposes a plane wave function is present, to an ion with
definitely a hydrogenic wave function. So correction for such a case
must also be taken into account.

The proposed solution of the model can be done as follows.

(1) Calculation of initial state: - The hydrogen bond interaction
can be calculated using Schuster's[4] method. The effect of the rest of the
solvent can be heated as a perturbation.

(2) Adsorption in the final state can be calculated by Van der Avoird's[5]
method, by calculating the exchange energies.

(3) Tunnelling - there is no definite solution for the case we have in
hand and this has to be developed along the lines suggested by Fonash[6].

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6. A. Fonash, Ph.D. Thesis, University of Pennsylvania 1967

SECTION 5

David LaPointe

J. O'M Bockris, Supervisor

Title of project:	Sodium Diffusion in Crystals
Long-term technological aims:	The purpose of this project is to study theoretically the dependence of the activation energy for Na^+ ion diffusion through a crystal lattice, on the various structural parameters. The reason for such a study is to find out why β-alumina has such a low activation energy for Na^+ ion diffusion, and if possible, to hypothesize an ideal lattice which will have very low activation energy for Na^+ diffusion.
Specific aims for this period:	In the previous report it was mentioned that a model was set up similar to the Burton and Jura[1] model which allowed us to calculate the activation energy for self diffusion in solids under a certain set of approximations. The aim for this period was to test this model for as many cases as possible.
Results of work in this period:	Work on the project during this period was centered on consideration of the magnesium oxide (MgO) lattice structure. It as felt that since most work of this type in the literature was concerned with alkali halides, an investigation of an alkaline oxide system would be a good method for evaluating the formulae which had been derived

previously, and would introduce a number of new variables into the program.

Since most constant, on an atomic scale such as the exponential "hardness" factor ρ and the pre-exponential constant β were not readily available, they had to be calculated. These calculations could only be good approximations, since the data used to calculate the constants were related to the continuum properties of the crystal (i.e., thermoelastic properties such as bulk modulus and other elastic properties), and the constants were to be used in non-continuum aspects of the crystal.

The equation to calculate the energy or potential of an ion i with charge Z_i and radius R_i was:

$$\mu_i = \sum_{j \neq i} \left(\frac{Z_i Z_j q^2}{r_{ij}} + B_{ij} \exp\left[(R_i + R_j - r_{ij})/e \right] \right) \tag{1}$$

where q is the unit of electronic charge, and B_{ij} is given by:

$$B_{ij} = B\left(1 + \frac{Z_i}{h_i} + \frac{Z_j}{h_j}\right) \tag{2}$$

where h_i is the number of electrons in the outer shell of ion i. B is a constant of the crystal.

Equation (1) was used to calculate the lattice potential of a magnesium ion in the crystal at rest. Our calculations gave $U = -940 \pm 20$ kcal/mole, which is very close to literature values of 936-944 kcal/mole.[1]

The energy of migration of the magnesium ion to a vacancy across the sublattice was calculated, using a diffusion path through the tetrahedral
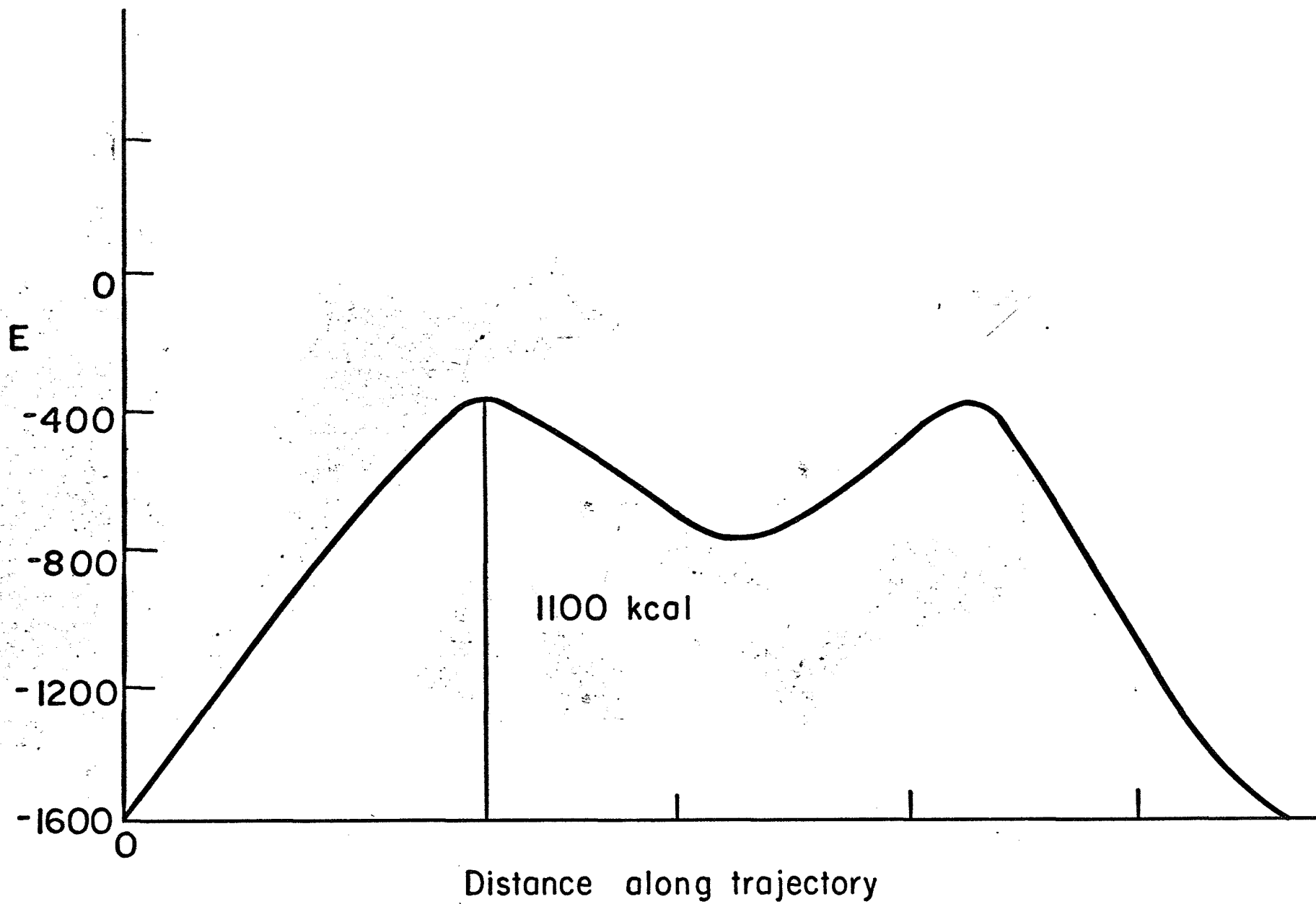
Energy vs. position along trajectory

void between the oxygen ions in the unit cell. The energy contour gave two "humps" of ~ -400 kcal/mole each and a minimum between the humps of -800 kcal/mole. Since the effect of creating a vacancy is to lower the lattice potential for the magnesium ion to -1540 kcal/mole, that is the magnesium ion which is to diffuse, the activation energy is approximately 1000-1100 kcal/mole through an unrelaxed lattice.

Plans for the future should include a careful calculation of relaxation effects.

All calculations involving summations were done on an IBM 360/75 computer which calculated the energy of the diffusing ion by equation (1) at a number of points along the diffusion trajectory.

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Jean Ricodeau

J. O'M. Bockris, Supervisor

Title of Project: Zinc electrode study for silver-zinc batteries

Long-term technological aims: Prevention of dendrite growth and of decrease of active surface on the zinc electrode.

Specific aims for this period: While crystalline process is also involved, we first deal with organic adsorption in order to understand dendrite-growth inhibition. Previous studies[1] invite to investigate large organic cations (tetraalkylammonium, hemolthogenes, triton . . . .) and to select those which would adsorb on the cathodic side, Zn deposition, i.e., charging potential region, and desorb on the anodic side, Zn dissolution, i.e., discharge potential region of a battery. Dendrite growth is expected to be inhibited if we can decrease the exchange current density by adsorption of organic species in the charge potential region while the battery efficiency need the exchange current density not to be decreased in the discharge region of potential.

Most of the usual methods of adsorption studies are forbidden for our purposes: electrocapillary, various sweep or pulse methods, hydrogen

coverage measurement, ... because we want operate on a solid metal

which is far from "noble," while all these methods need to avoid large

currents as those expected in the potential region to be investigated.

Among the usual methods, there is the radio tracers measurements. But

because the uncommercial active compounds and the high concentrations

to be used (10^{-4} – 10^{-2} molar), this decreasing the accuracy of that

measurement, we first tried to avoid the method, and used the following

ideas.

Results of work: When adsorption occurs, we can consider it to give

a perturbation in the concentration of the adsorbed

species in the solution nearby the electrode. This

perturbation will propagate and disappear by diffusion

process. The time needed to have again an unperturbed

solution in the neighborhood of the electrode is given by the geometry,

the diffusion laws, and the characteristics (adsorption isotherm and

coverage) of the perturbation. Assuming for the adsorption reaction a

linear isotherm and a diffusion controlled rate of adsorption in unstirred

solution, one expects the coverage θ to vary as the square root of time.[2]

$$\frac{d\theta}{d(t^{\frac{1}{2}})} = \frac{2D^{\frac{1}{2}}}{\pi^{\frac{1}{2}} \kappa \Gamma_{max}} (\theta_{equil} - \theta_o)$$

If during adsorption we have at the electrode an electron transfer

reaction, change in coverage produces change in current density.

Recording the current versus the square root of the time after the

beginning of the adsorption process, we can get the time τ (Fig. 1)
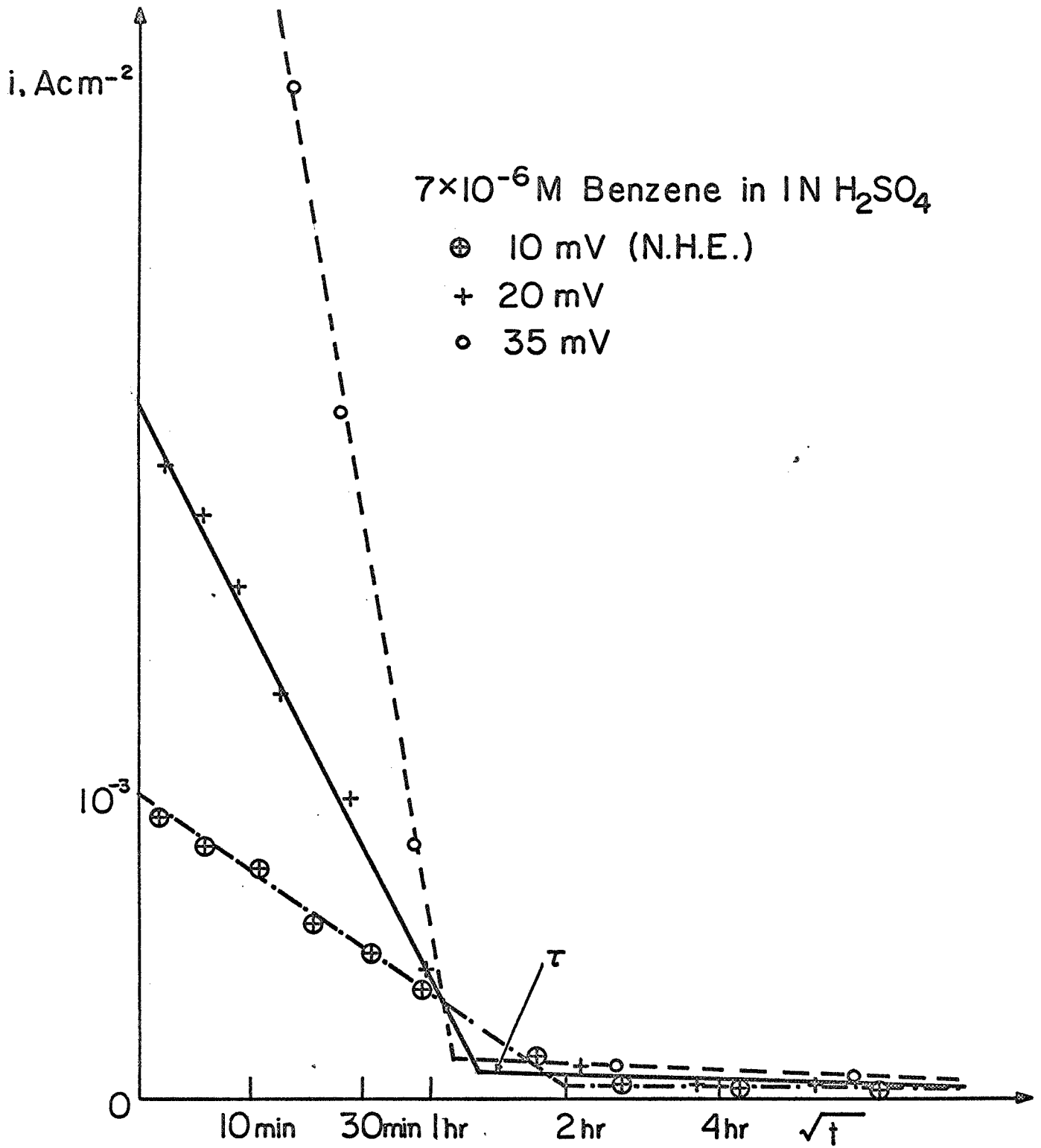
Fig.1

needed to reach a steady state which corresponds to the equilibrium of adsorption. With the knowledge of this time and the theory of diffusion we expect to determine the amount of adsorbed species.[3] To check the validity of this method we tried it on platinum with benzene adsorption, because the coverage curves in this case are well known.[4] Our task, to measure a current (hydrogen evolution) while previous studies tried to avoid it, places our work at the border of the former data. For various potentials (+100 mV to −100 mV) (NHE) and for varied concentrations of benzene (10^{-7} − 10^{-5} M) in 1 N sulfuric acid solutions, we found straight lines of the current versus $\sqrt{t}$ with a time τ ranging from 1 hour to a few hours (Fig. 1). A plot of τ versus the potential gives (Table 1) a part of the expected bell-shaped curve wer? were expecting in this cathod region (Fig. 2).

These results could seem good. But —

Convection in the solution for so large a time must be a determin?? ming factor which makes the diffusion equations meaningless.

Then we did some counter experiments in stirred conditions and found that while the current vs time variation was expected to be given by the coverage law $\Gamma_{(t)} = \Gamma_{max}[1 - \exp(\frac{-Dt}{\kappa \tau})]$ it was again approximately a $\sqrt{t}$ variation as in unstirred conditions. On the other hand, the time τ was not reduced as much as expected.

Plots of the final current versus potential give curved plots in a semi-logarithmic scale which cause doubts about a constant current-

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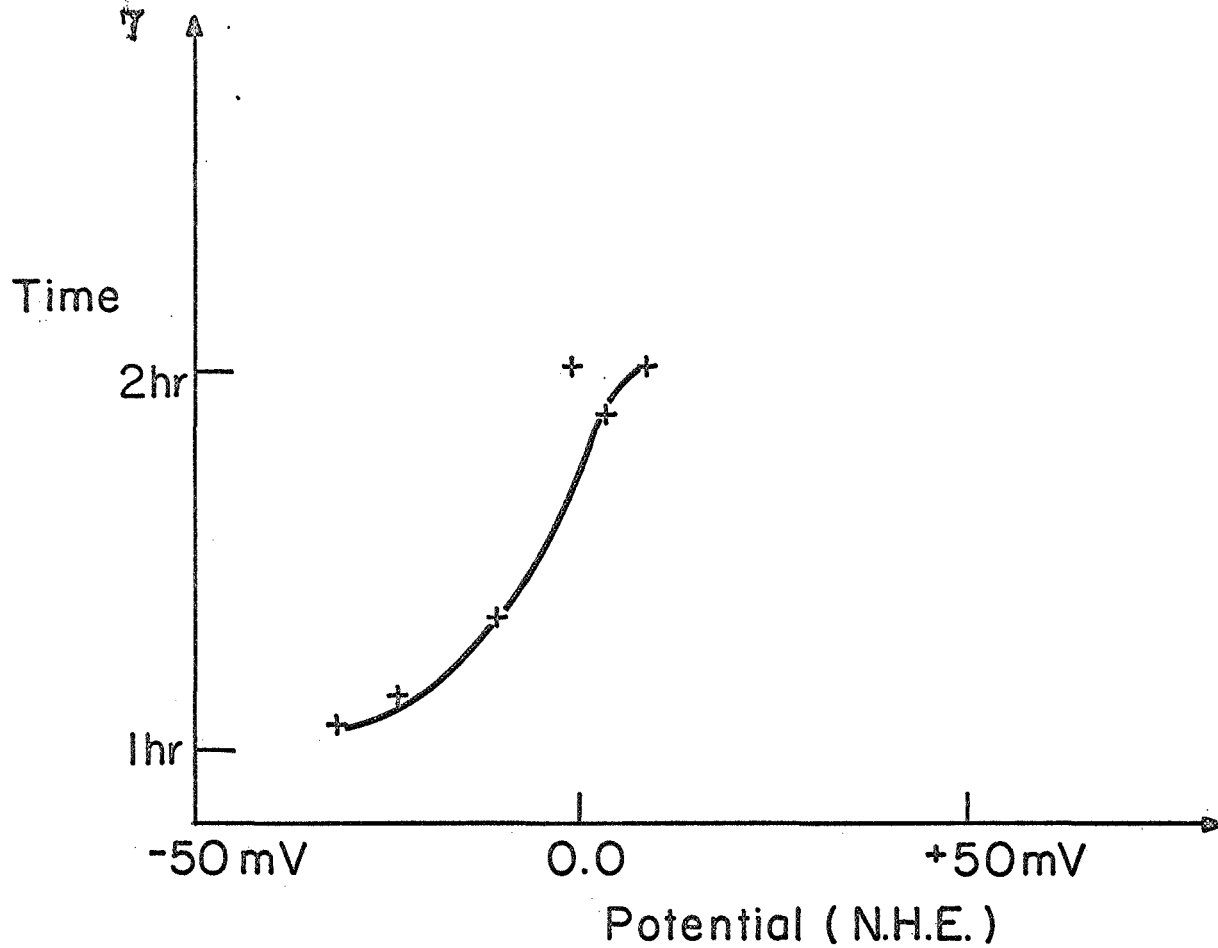
Fig. 2

Table I

Potential	τ
-35 mV (N.H.E.)	1 hr 5 mn
-25 mV	1 hr 10 mn
-10 mV	1 hr 20 mn
0.0 mV	2 hr
+5 mV	1 hr 50 mn
+10 mV	2 hr
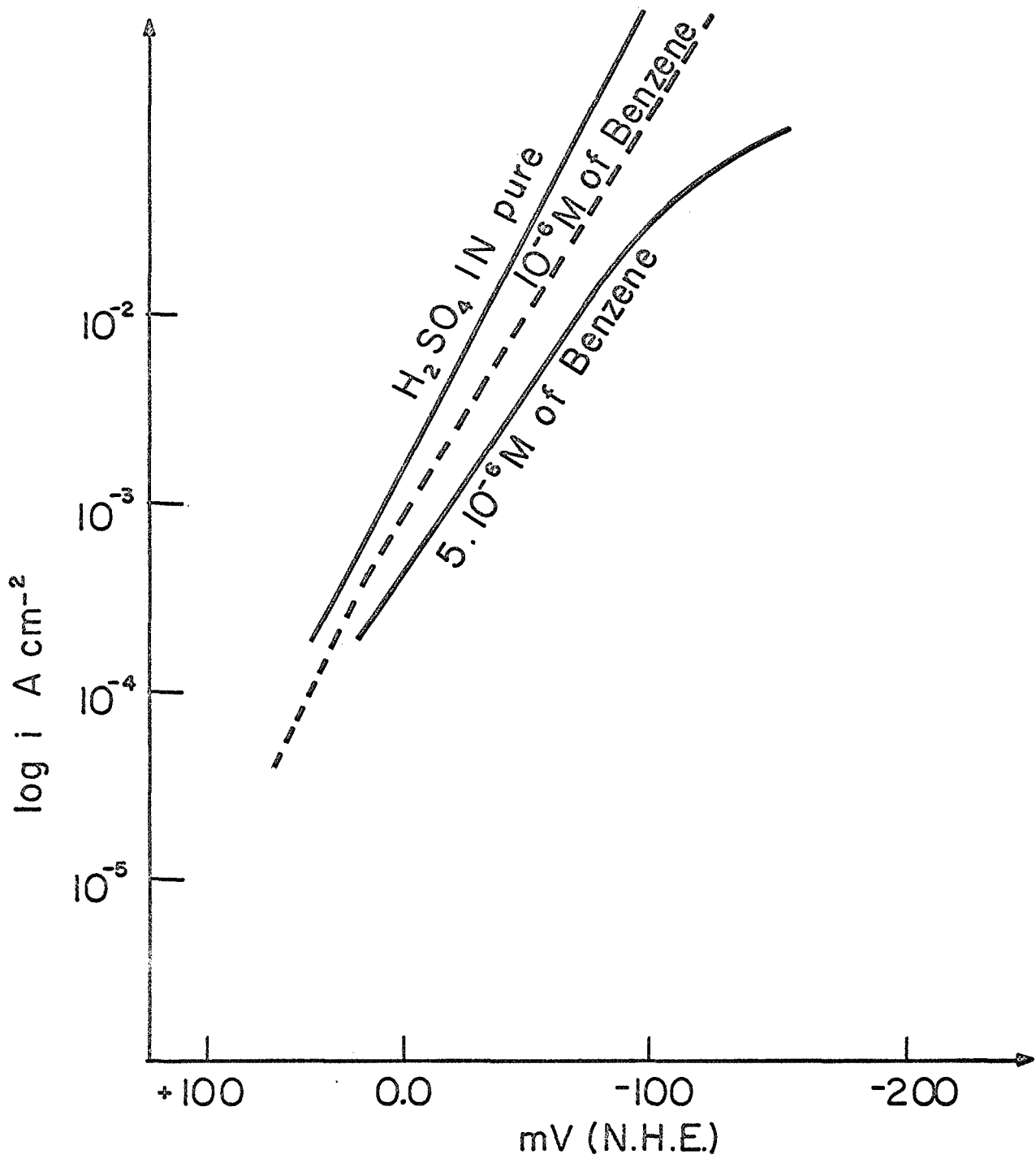
Fig. 3

coverage relation (Fig. 3). Thus, the test made on platinum with

benzene adsorption suggested not using such a method on zinc.

Specific aims for next report period: Since we don't want to use a diffusion method we need to look for another way to investigate adsorp-

tion on Zn electrode near its reversible potential.

The more classic is the radiotracer method which exists in varied

forms.[6]

The accuracy of these methods decrease when the concentration of

the adsorbing species increases in the solution. The high concentra-

tion to be used ($10^{-4} - 10^{-2}$ M) may be a problem.

Together with such radiotracer methods we are planning to obtain

complementary information by ellipsometric measurements.

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   at the Electrochemical Society fall meeting, Detroit, October
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