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FIFTH QUARTERLY REPORT
1 JANUARY 1970 to 31 MARCH 1970
STUDIES IN FUNDAMENTAL CHEMISTRY
OF FUEL CELL REACTIONS
NGR 39-010-002

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ELECTROCHEMISTRY LABORATORY

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FIFTH QUARTERLY REPORT

1 JANUARY 1970 to 31 MARCH 1970

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:

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PROJECT PERSONNEL

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SECTION I

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: Testing of the efficiency of the rhodizonic acid (RA) graphite paste electrode with newly designed cell.

Results of Work in This Period: The cell employed in study of RA graphite paste electrode is schematically presented in Fig. 1. Both the paste electrode and the counter electrode compartments were provided with inlets and outlets for the passing of N_2 . The two compartments were separated by cationic exchange membrane to prevent diffusion of the dissolved portion of RA to the working electrode compartment. The RA graphite paste electrode was made by mixing 5 g of solid RA with a wet mixture of graphite and activated charcoal in a ratio of 1:1. The activated charcoal was employed in order to hold adsorbed RA at its surface and prevent the loss of RA by dissolution in the surrounding

electrolyte. The potential of the paste electrode was measured against the saturated calomel electrode through a Luggin capillary adjacent to the glass frit separating the paste electrode from the solution. All potential values were corrected for IR drop through both solution and glass frit.

The discharge characteristics of the RA graphite paste electrode with Pt as a counter electrode in 1.2 N NaCl are presented in Fig. 2. The main loss in the current efficiency was observed during the first discharge. The discharge was followed by changing to the initial potential, and discharging again. Up to ten discharge and charge cycles of the RA paste electrode were performed during which the current efficiency remained practically unchanged and equal to 100% of the first discharge cycle.

The current efficiency for the reduction of RA to hexahydroxy benzene (HHB) according to the reaction⁽¹⁾



has been followed in separate experiments by potentiometric titration of the reduction

product with K_3FeCy_6 (the reduction being performed upon a Pt screen cathode of 100 cm^2 area). The analysis of the results shows that the loss in the current efficiency is partially due to the formation of the stable intermediate tetrahydroxyquinone. A detailed analysis of these results will be presented in the next report period.

Specific Aims for The further study of the current efficiency
Next Report of the RA graphite paste electrode.
Period: Detailed mechanistic study of the electrode
reaction.

References:

1. Third Quarterly Rept., July-Sept., 1969, NGR Contract 39-010-002.

Captions to Figures:

1. Schematic representation of cell used in determining charge-discharge characteristics of the RA-HHB couple.
2. Discharge characteristics (expressed as total coulombs passed *vs.* potential) as a function of total current. Electrode area 3.1 cm^2 .

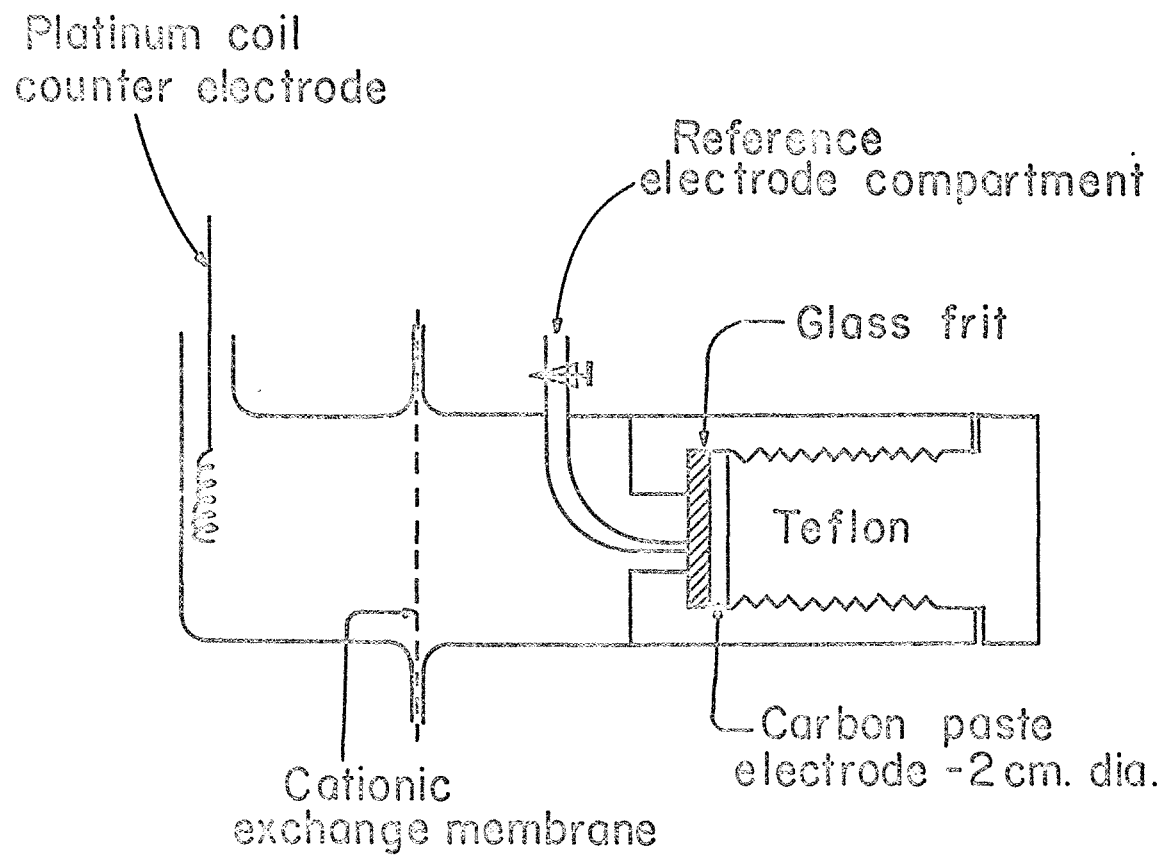


Fig. 1

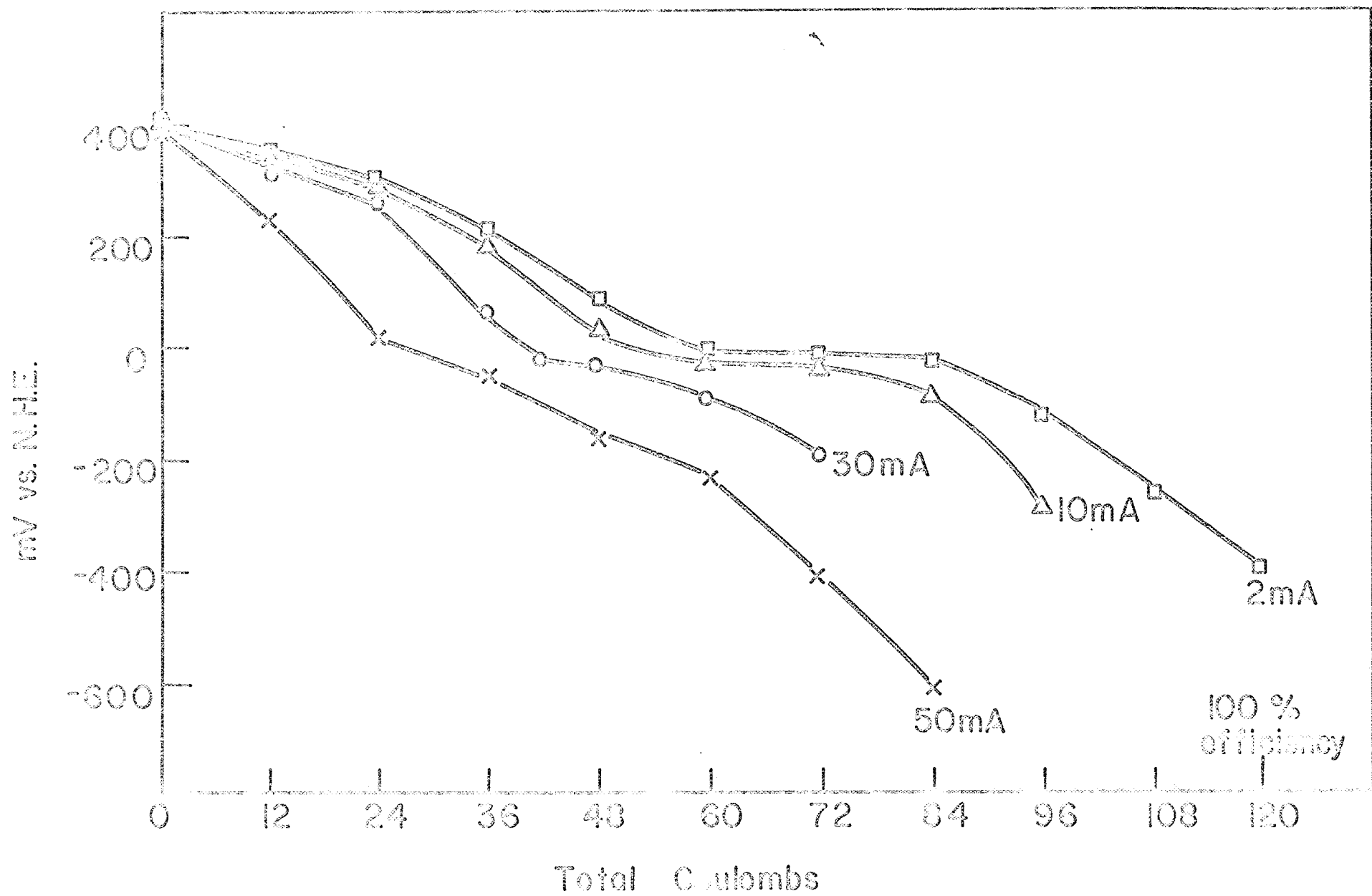


Fig.2

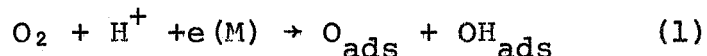
SECTION II

Title of Project: Oxygen Dissolution Reaction: A Theoretical Study

Long-term Aims: To calculate theoretically the rate of the oxygen dissolution reaction and then study the properties of the metal electrode on which the rate of the above reaction depends most. Such a study, if successful, will be of great help in developing a catalyst for the oxygen dissolution reaction.

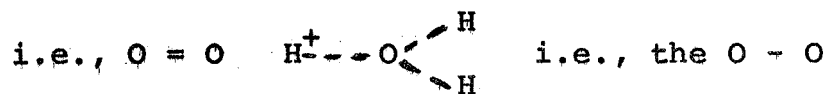
Specific Aims for This Period: The aim for this period was to look into the initial state of the reaction in detail.

Results of Work in This Period: As mentioned in the previous report, the rate determining step of the oxygen dissolution reaction is:

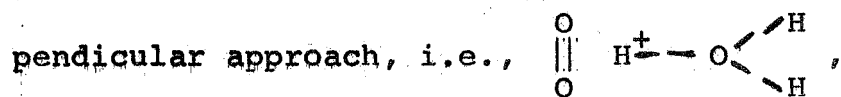


Thus the initial state for this reaction consists of an O_2 molecule and a proton in solution, and the electron in the metal. It is pretty obvious from the above rate determining step that in this step a three-body process is occurring. The O_2 and H^+ have to arrive at a particular configuration, and the moment such a configuration is

established the electron transfer from the metal to the species in solution occurs. Now, we considered the various possible ways in which the H_3O^+ can approach the O molecule near the electrode surface. There are a large number of ways in which such a process can occur. However, two distinct possibilities were ultimately considered to be important, namely (a) a co-linear approach,



bond in the molecule and the $\text{H}^+ - \text{O}$ bond in H_3O^+ lie in the same line, and (b) the per-



where the $\text{H}^+ - \text{O}$ bond in H_3O^+ is perpendicular. In the latter case the species has

a C_{2v} symmetry. Now the problem was to decide which of these approaches plays the predominant role in the rate determining step of the oxygen dissolution reaction. Since there is no experimental evidence for the occurrence of any of those species in any form or any state, we had to decide on some theoretical criteria. The criterion that was decided upon was the following:

It is usually agreed that whenever two configurations can be formed for the same species, the preferred configuration is the

one having the lower energy. So it was decided that we will do a SCF-LCAC-MO calculation for both the configurations as the H^+ approaches the oxygen molecule and whichever configuration gives the lower energy will be considered to be the preferred configuration. However, what we have said up til now is truly valid for the gas phase. But, unfortunately, we have the solvent in our case to take care of. It is easily imaginable that the solvent will play a very important role in deciding the configuration of the above-mentioned species. It is extremely difficult to take the effect of the solvent into account quantum-mechanically because the problem then becomes too immense to be solved. So the effect of the solvent has to be taken into account in some classical manner.

Specific Aims for In the next period we would like to decide
Next Period: on the exact manner in which the solvent effect can be taken into account. Then we would like to decide in detail our model for the final state.

SECTION III

Title of Project: Zinc Electrode Study for Silver-Zinc Batteries

Long-Term Aims: Prevention of dendrites' growth and of decrease of active surface on the Zn electrode.

Specific Aims for This Period: While crystallization process is also involved, we first deal with organic adsorption in order to understand dendrites' growth inhibition.

Previous studies⁽¹⁾ invite to investigate adsorption of large organic cations (tetraalkylammonium... , hemulphogene, 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, Z 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 cur-

rent density not to be decreased in the discharge region of potential.

Most of the usual methods of adsorption studies are forbidden for our purpose: electrocapillary, varied sweep or pulse methods, hydrogen coverage measurement . . . because we want to 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 range to be investigated. After an investigation using diffusion theory⁽³⁾ which failed, we started to use thin film ellipsometry measurements.

Results of the
Work:

Ellipsometry method relate the changes in intensity and polarization, of an elliptically polarized light beam, to the characteristics of the surface reflecting the light beam.⁽⁴⁾

One needs a smooth and flat electrode as a mirror. We were able to obtain such a zinc surface by mechanical polishing, but the brightness was never very good, perhaps due to oxide layer, and optical parameters differences between grains of different crystal faces.

Inside the cell we must maintain this mirror-like surface, i.e., have neither dissolution nor H₂ bubble formation. In

1 N KOH solution the rest potential of Zn was -1.5 V (SCE) and it was possible to vary the potential between this value and -1.7 V (SCE): in this cathodic region the corrosion is decreased while the hydrogen evolution reaction is not too strong and bubble formation is avoided. Moreover, the surface must remain oxide-free and this appeared to be very difficult: the ellipsometry measured values of polarization angle and intensity were found generally to vary with time and to correspond to those of an oxide film on the surface.

The compound to be observed must have a $\frac{R}{V}$ larger than that of water. In the absence of accurate values in the literature for our tetraalkylammonium salts we measured the refraction index of tetraalkylammonium hydroxide: increase of the refractive index with concentration proves (OH^- is optically the same as water) that these larger cations are optically active for ellipsometric measurements. We had to avoid the presence of chloride or bromide salts because these anions are optically very active, and change in their adsorption on the Zn surface would mask change in adsorption of our cation.

Because the electrochemically unstable An surface we couldn't use a wide potential region as in other adsorption studies by ellipsometry.⁽⁴⁾ We thus put our compounds in the solution and measured the change in ellipsometric values (change in the refractive index of the solution due to the addition of compounds may disturb the optical system) before and after. No change was observed, when we were expecting absorption.⁽⁵⁾

We tried also on platinum whose stability allows better experimental conditions: no change was observed, but we didn't find adsorption data of tetraalkylammonium salts on platinum in the literature; perhaps as for iron⁽⁶⁾ there is on platinum no adsorption of these species.

Specific Aims for Next Report Period: Since ellipsometry gives no results, the last method to be used, according to the previous report, is the radiotracer method.⁽²⁾ The accuracy of this method decreases when the concentration of the adsorbing species increases in the solution. The high concentrations (10^{-4} - 10^{-2} M) to be used may be a problem. We expect first to study some of the tetraalkylammonium salts which are available from sulfur as C_{14} labelled compounds. A Zn tape will be in a cell for

adsorption to occur, then will be taken out of the cell through a slit and the activity of the thin film of solution which remains on the metal surface will be counted by a proportional gas flow counter.

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