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INVESTIGATIONS LEADING TO THE DEVELOPMENT OF A PRIMARY ZINC-SILVER OXIDE BATTERY OF IMPROVED PERFORMANCE CHARACTERISTICS

SUMMARY REPORT NO. 3

Contract No. NAS 8-5493

Control Number TP3-83728 (1F)

CPB 13-1600-63

GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

Reporting Period
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THE EAGLE-PICHER COMPANY
COUPLES DEPARTMENT
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I. PURPOSE

The immediate objective during this extended contract period shall be the design of a zinc-silver oxide cell capable of activated stand periods and recharge abilities as follows:

A. Stand period (or useful life) - thirty days
B. Stand temperature - 90° F
C. Cycle capability - six cycles in thirty days
   1. Five cycles removing 25% depth
   2. A final discharge of 100% capacity
D. Battery voltage during discharge - 28 ± 2.0 volts (1.40 ± 0.10 volts per cell)

Related studies will be carried out as required to achieve this goal.

II. ABSTRACT

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Effort is being made to develop a cell design capable of limited cycle life while retaining the high rate and high density characteristics of the primary zinc-silver oxide system.

A literature search has been carried out with emphasis on references pertinent to improved separator systems.

A preliminary design study has been begun to determine more specifically the areas subject to design modification.

Studies relative to optimization of the zinc plate densities over the useful range of discharge current densities have also been begun.

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III. FACTUAL DATA AND DISCUSSION

A. General

The zinc-silver oxide system has been investigated extensively as indicated by the references included in the appendix. Specific studies have included individual investigations of both electrodes, as well as separator materials. Segments of other studies have dealt with electrolyte concentration and additives.

It is indicated that it is possible to construct highly reliable, high energy density, primary zinc-silver oxide batteries, while reliability of the secondary battery comes at the expense of either battery energy density or high rate capabilities. Yet it is possible to construct zinc-silver oxide batteries, either vented or sealed, capable of a considerable number of cycles.[1, 3] The goal of this program shall be the design of a cell capable of limited cycle life while retaining the high energy density and close voltage regulation features characteristic of the primary system.

It appears that utilization of the cathode and anode active materials has essentially reached a plateau, although references occasionally mention the application of chemical additives or other innovations. For instance, Dirkse[2] has discussed the addition of palladium to the positive electrode, as have Landers and Keralla.[3] It is anticipated, therefore, that the most advantageous approach will involve the assimilation and application of technique and knowledge revealed in the pertinent references. This is perhaps most true of evaluation of separators and methods of their improvement, because of programs which have dealt largely or entirely with separator studies.

B. Separator Materials

1. Design Factors

As silver migration, dendritic zinc growths, dissolution of zinc, and separator degradation remain limiting factors in the operation of secondary zinc-silver oxide cells, a great amount of study has been accomplished in recent years relative to separators. Almost all silver zinc development programs have included phases of study devoted either to screening or special treatment of separator materials and the selection of the optimum combination thereof. To understand the importance of selecting the optimum separator combination, it is only necessary to examine the function of this material and the effect of multiple layers upon cell energy density and voltage regulation characteristics.

As indicated by Figure No. 1, equilibrium cell voltage during discharge can be represented by an essentially linear function of current density based upon the total superficial area of the positive group (if all other design factors are held constant). Figure No. 2 indicates possible effects of quantity of separator material in a cell upon discharge voltage at constant current density. Figure No. 3 relates the effects of varying the number of plates and quantity of separation in a cell upon the possible capacity in the cell. These three figures indicate the problems of design involved in optimizing the design of a cell for a specific application.

K+E 10 X 10 TO ½ INCH 46 1473
7½ X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

FIGURE NO. 1

EFFECT OF DISCHARGE CURRENT DENSITY
UPON MAXIMUM MONOVALENT PLATEAU VOLTAGE

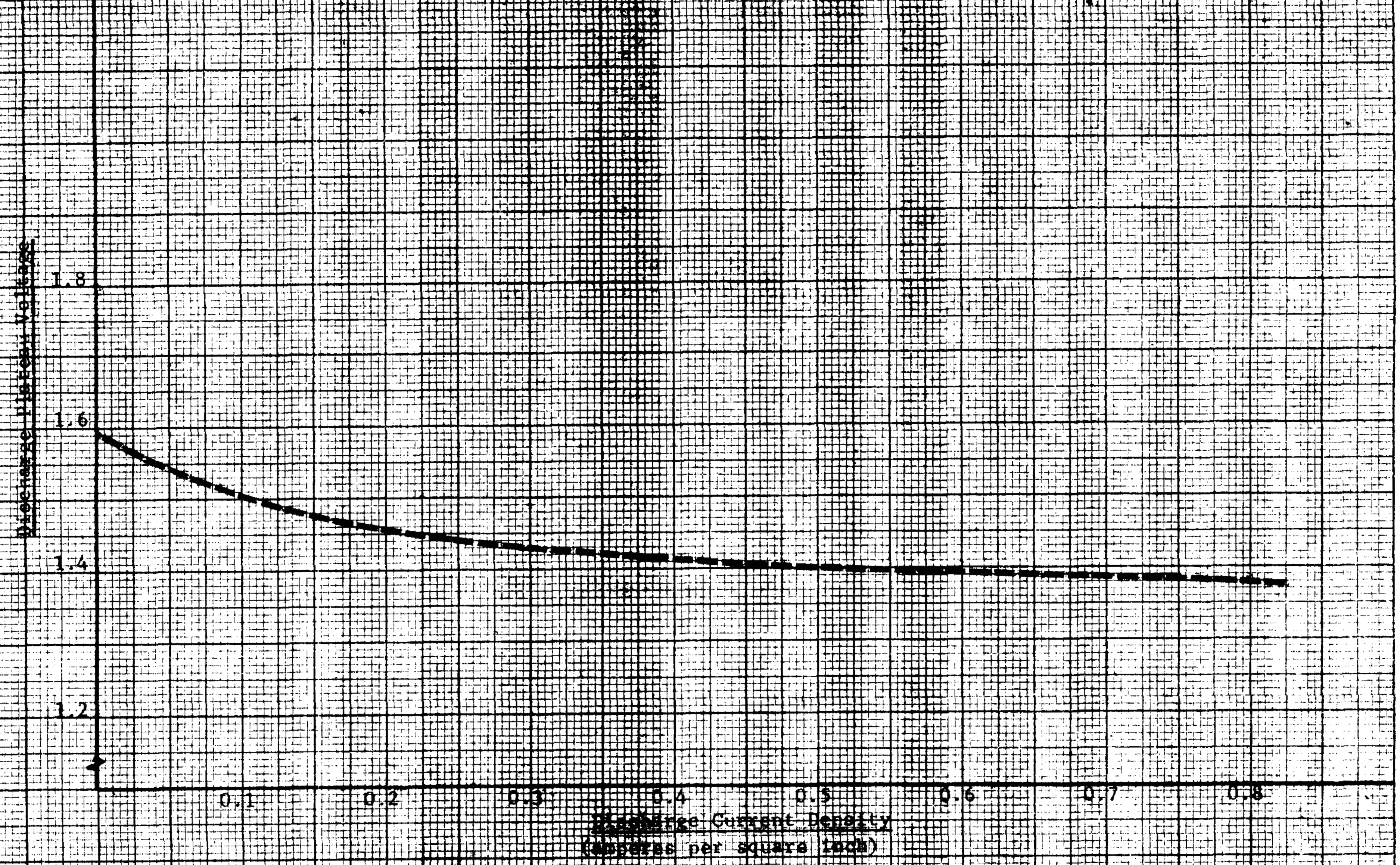


FIGURE NO. 2

EFFECT OF SEMIPERMEABLE MEMBRANE
ON DISCHARGE VOLTAGE

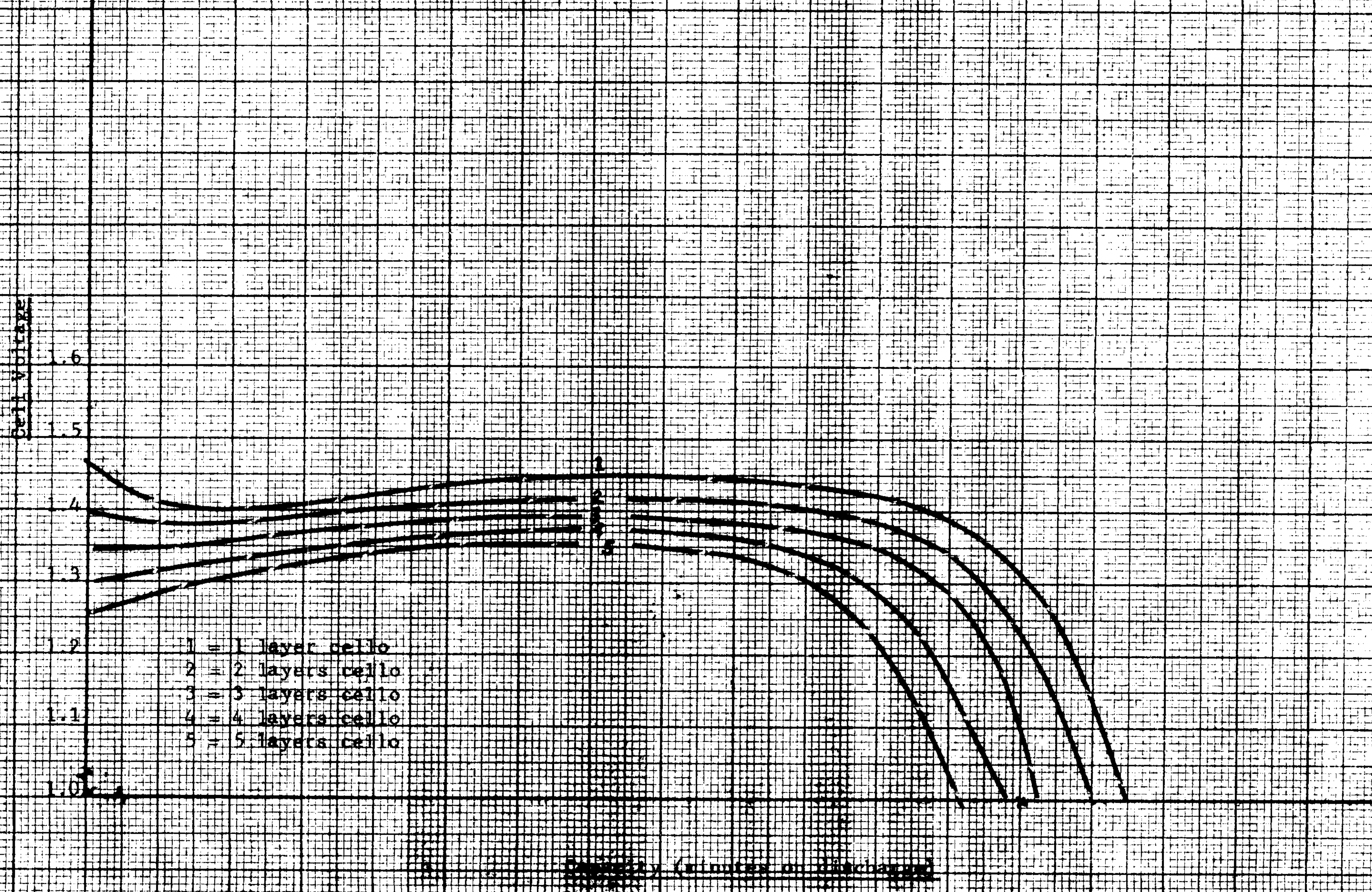
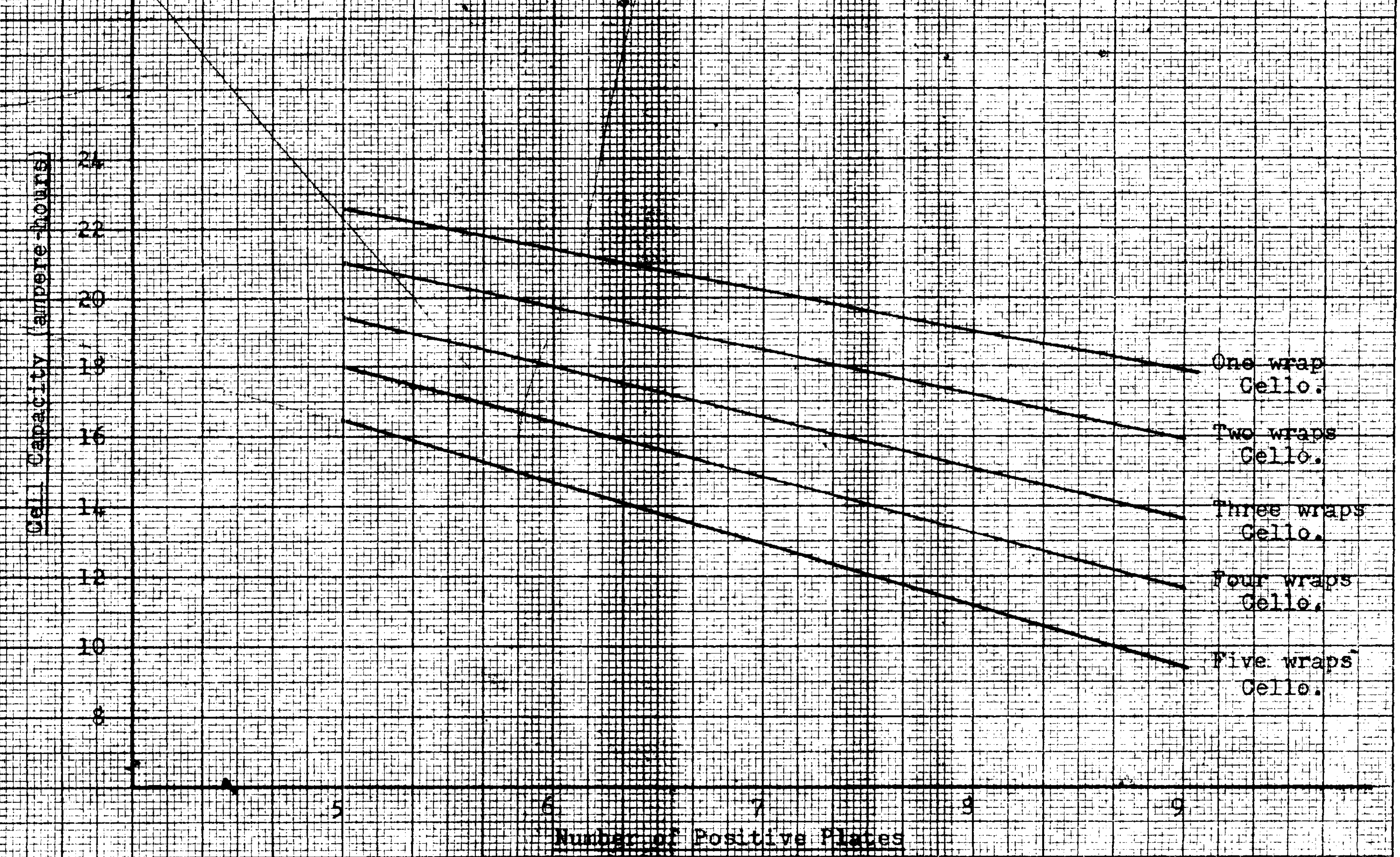


FIGURE NO. 3
TYPICAL EFFECT OF CONSTRUCTION
VARIABLES UPON CELL CAPACITY



Very frequently, a battery application demands an average cell voltage within definite limits, such as 1.40 ± 0.10 volts. If longer activated life is required, a common method of achieving this aim is the use of a more membranous separator. The greater thickness of separator may be sufficient to demand an increase in number of plates to offset the adverse effect of increased separation upon cell voltage and voltage regulation. As indicated by Figure No. 3, this in turn decreases the possible cell capacity and energy density.

These practical design criteria reveal the importance of selecting the optimum separator system and the use of this system such that limited cycle life may be obtained without making the sacrifices outlined above.

2. Discussion of Separator Treatments

Many studies have included screening tests which reflect the relative abilities of prospective separator materials to meet one or more of the requirements of an acceptable product. The criteria used commonly considered in evaluating separators include the following:

A. Dimensional changes upon wetting
B. Electrolyte retention or absorptivity
C. Degradation of the material in caustic solution
D. Ability to retard silver transport
E. Resistance to oxidation
F. Electrical resistance
G. Physical strength

Some of these properties have been evaluated and reported for most of the commonly available commercial products.

The use of glycerine-free cellulosic membranes has been widely followed by makers of secondary alkaline cells because of the ability to retard silver transport and their relatively low electrical resistance, as well as their low cost and ready availability. However, because of the degradation of these materials in caustic and oxidizing environments, many attempts to find substitutes or to improve the properties of cellulosics have been made.

One treatment of cellulosic materials which has been widely reported is the deposition of silver within the material by use of formaldehyde or other reducing agent. (4) It has been reported that such treatment may reduce swelling but has little or no effect upon oxidation resistance as determined by attack of alkaline permanganate solution. (5)

Studies have resulted in data relating the progressive deterioration of cellophane in KOH/ZnO systems under oxygen, including the depolymerization of the material as a function of time and temperature. It was found that the maximum degree of swelling of cellophane and, hence, the greatest degree of attack upon cellophane, occurs in KOH concentrations ranging from 26-33 percent by weight. (5) Another manufacturer has reported data describing the loss of strength of cellophane in caustic solutions at temperatures down to -35° C. (6)

Among the treatments of cellophane have been addition of grafts of styrene and isopropylacrylamide monomers as well as methacrylic acid.[5, 7] Indications are that these treatments may not greatly increase the resistance to KOH attack without undue increase in the electrical resistance of the membrane. One of the more recent treatments has been that of cellophane by antioxidants such as m- and p-phenylenediamine. It has been indicated that such treatments may result in remarkably improved oxidation resistance while electrical resistance remains essentially unchanged (about 0.010 ohm-inch^2 for the membrane tested). However, this decreased reactivity of the membrane caused inability to retard silver transport. It is now concluded that a separator should be able to react with dissolved and colloidal silver in order to prevent its migration.[2, 5, 6] Later reports from Electric Storage Battery Company indicate that an isocyanate treatment of cellulosics may hold some promise. Preliminary indications were that results might include improved strength retention and greater oxidation resistance at the expense of slightly increased electrical resistance.

Recently, attempts have been made to evaluate or study separator systems by means of radioactive tracer techniques.[2] This involves measuring the rate of transport of an isotope of silver through the system under study. However, Dirkse verified that this means of evaluation is confused by the fact that cellulosic materials effectively remove dissolved and colloidal silver from the electrolyte. Therefore, very little silver passes through a "fresh" cellulosic membrane (assuming no grafting or special treatment of the membrane). Again, it is implied that only reactive membranes are capable of preventing silver transport.

3. Conclusions

In view of the difficulty observed in obtaining reproducible results relative to silver loading of membranes and transport through membranes, it is doubtful that any mathematical description or prediction can be formulated to estimate accurately the processes which occur in a cell. This is true for several reasons:

1. Only under the most exacting conditions can compression of expanding or "swelling" materials be held at a constant level. This implies variation in the mean pore size, microscopic surface area, and rate of chemical attack, as well as electrical resistance.

2. Silver transport undoubtedly varies with the "age" of a reactive membrane. This age would be reflected by the state of degradation and quantity of silver already held by the membrane.

3. Zincate concentration in the electrolyte probably also affects silver transport.

4. Certain membranes, notably irradiated inerts, appear susceptible to wrinkling, or local overlap.

5. If considerable quantities of "open" separators are employed in a cell, bulk movement of the electrolyte, as well as ionic transport, may be a considerable factor in the movement of silver and zinc throughout the cell.

The apparent conclusion is that at the present state-of-the-art, screening tests only allow the rejection of poor materials on the basis of comparison to acceptable products. For example, it may be decided that a product having an electrical resistance three times that of cellophane does not justify further evaluation. However, it may not be possible to assert that a material which passes a single screening test will perform desirably in a cell. It is likely that factors such as pore size and distribution or other characteristics not easily measured are important. Until testing procedures are further refined, the final step in testing a separator system will involve employing it in a cell.

C. Utilization of the Zinc Plate

Studies reported earlier indicated the beneficial effect of lower apparent densities upon discharge efficiencies of the zinc plate. Since a density of 40 grams per cubic inch, the lowest investigated, proved to give the greatest efficiency, data have been extended over a range of 36-58 grams per cubic inch. As the less dense material requires greater volume, it is apparent that for optimization it is also necessary to examine the volume factor. Table No. I contains data relevant to this study.

TABLE NO. I

PLATE THICKNESS (inches)	APPARENT DENSITY (gm/in^3)	PERCENT EFFICIENCY	AMPERE-HOURS PER MILLI-INCH
0.030	36	52.1	0.00796
0.028	40	48.4	0.0079
0.024	46	42.4	0.00796
0.022	51	33.9	0.0069
0.020	58	28.8	0.0065

These data were obtained specifically for a zinc plate weight of 1.01 grams per square inch, and for a current density of 0.27 ampere per square inch of surface area. Plates were discharged against a considerable excess of positive material and these data represent the average of five "runs". It is apparent that at the lower densities, the increase in plate efficiency is offset by the plate thickness, so that no advantage is gained on a volume basis. As such data are dependent upon current densities, it is planned to attain sufficient data to allow optimization over the practical range of discharge rates.

D. Failure Modes of the Zinc-Silver Oxide System

There are several modes of failure or loss of capacity associated with the zinc-silver oxide system. These include the following:

1. Loss of oxygen from the positive plate: Divalent silver oxide is thermodynamically unstable in caustic solutions. Aside from other reactions the eventual decomposition of the higher oxide of silver will cause considerable loss of capacity from a positive plate which contains little or no metallic silver.

2. Loss of silver to the electrolyte and separator material: Cellulosic membranes effectively remove ionic and colloidal silver from the electrolyte. Less reactive materials also impede the transport of silver to some extent and accumulate silver in the process. A small amount of silver also remains in solution as governed by the solubility product of the species. Silver may be removed from the solution by the separator or zinc plate depending upon the efficiency of the separator system in use. As the silver content of the electrolyte tends to be renewed so long as the oxidized form lasts, it is obvious that a separator system must represent optimum conditions of electrical and physical performance.

3. Contamination of the electrolyte by zincate and carbonate: The effects of such contaminations apparently have not been fully evaluated. It would seem, however, that such additions would affect the impedance of the cell, resulting in the reduced availability of electrochemical energy at the higher voltage levels. This would cause imparied voltage control and reduced acceptable capacity. Figure No. 4 indicates the effect of stand upon voltage control and capacity. While the capacity for the subject cell to an end voltage of 1.30 volts decreased by nearly 20%, the capacity to a 1.10 volt cutoff decreased by less than 13%. One phase of study will involve the determination of cell impedance as a function of activated life, as well as the resistance of separator materials having undergone silver accumulation and physical degradation.

4. Internal shorting: Penetration of the separator system by either metallic silver or dendritic growths may provide conductive paths and therefore, self-discharge of the cell.

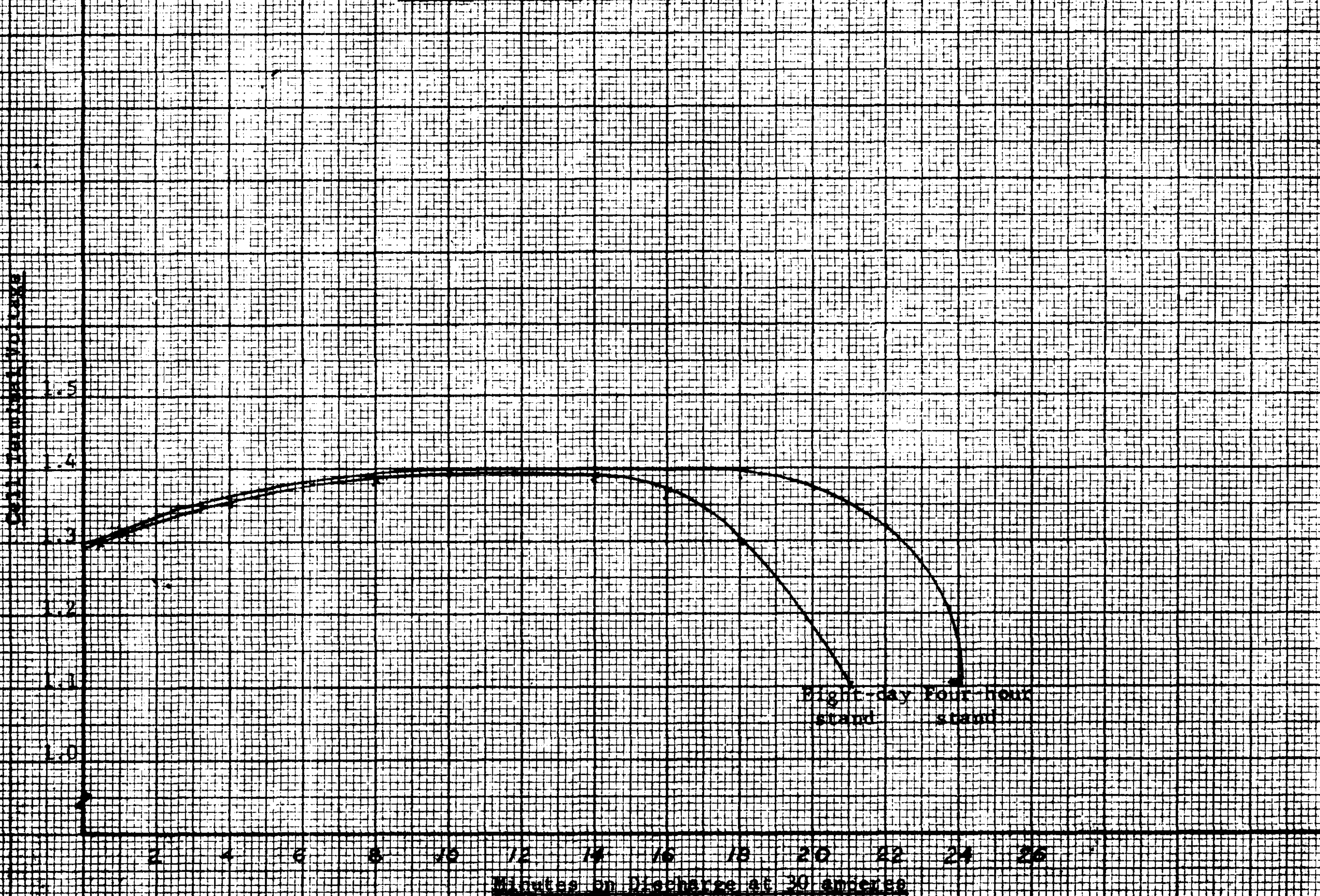
E. Preliminary Design Study

A set of cells has been constructed for the purpose of estimating the capabilities of a design similar to the prototype cell design presented at the conclusion of the original contract period. The only major change was the use of silver grid in both the positive and negative plates.

Data are not yet sufficiently complete to compare to the design objective as stated. Cells have been discharged to 1.30 volts to determine average cell capacity, followed by discharges of 25% of this capacity after intervals of one and six days. Stand temperature is 90° F. The remaining discharges will be conducted and the data presented in the next progress report.

FIGURE NO. 4

EFFECT OF ACTIVATED LIFE ON DISCHARGE VOLTAGE



IV. SUMMARY AND CONCLUSIONS

A number of references have been compiled and most examined for relevance to this contract. It is apparent that appropriate cell designs are capable of numerous cycles, at the expense of the high energy density characteristics of the primary zinc-silver oxide system. It is hoped to arrive at a design which will allow limited cycle life at +90° F without compromising the high rate capabilities and energy density of the primary system.

Tests have been begun to estimate the cycle ability of cells similar in design to those constructed at the end of the original contract period. At this time three cycles have been conducted over a period of twelve days.

Utilization of the active materials is not currently being improved appreciably. It is indicated, however, that zinc material density may be regulated to allow the determination of the optimum quantity of zinc for a specific condition of service. As confirmed earlier (see Final Report, this contract), density of the negative material is a significant design factor with respect to charge retention on activated stand. Data presented in this report reveal that on initial discharges, at least, the benefits of zinc densities lower than 45 grams/inch^3 may be offset by the greater volume required by the anode active materials. These data were obtained at a specific current density and tests are being initiated to extend these data to cover the range of useful current densities.

The selection of the optimum combination of separator materials for a specific application is important in view of the effects of multiple layers upon voltage magnitude and regulation as well as cell capacity. Several researchers have concluded that a reactive membrane, such as cellophane, is necessary to prevent silver transport. The literature includes preparation of membranes and treatments of membranes too numerous to itemize.

Certain treatments which are indicated to be beneficial, such as the isocyanate treatment of cellophane, will be evaluated with respect to attaining the design goal.

V. PROGRAM FOR THE NEXT INTERVAL

Effort will be made to regain some of the slippage indicated in the Master Schedule included in the Appendix. This will include effort in the following specific areas:

1. Survey of the current literature will be continued.

2. Attempts will be made to locate thinner suitable separator materials, both membranous and "open" types.

3. Optimization studies relating to zinc efficiency as affected by apparent density of the material and discharge current density will continue.

4. A program will be outlined which will allow estimation of the operting and reliability characteristics of the final cell design.

5. Studies relating to the effects of various electrolyte additives will be initiated.

6. The preliminary design study will be completed.

VI. PERSONNEL

The following totals of man-hours have been expended during the contract period:

Engineering -----------	2381	hours
Technical -------------	2894	hours
TOTAL	5275	hours

A P P E N D I X

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MASTER SCHEDULE

PROGRAM TO EXTEND THE CAPABILITIES OF THE PRIMARY ZINC-SILVER OXIDE SYSTEM

CONTRACT Extension & Modification of Contr. NAS 8-5493

REFERENCE Technical Approach

PREPARED Bill R Hawkins
APPROVED [signature]
APPROVED EMM
APPROVED

← P.O. DATE

ACTIVITIES

LITERATURE SURVEY

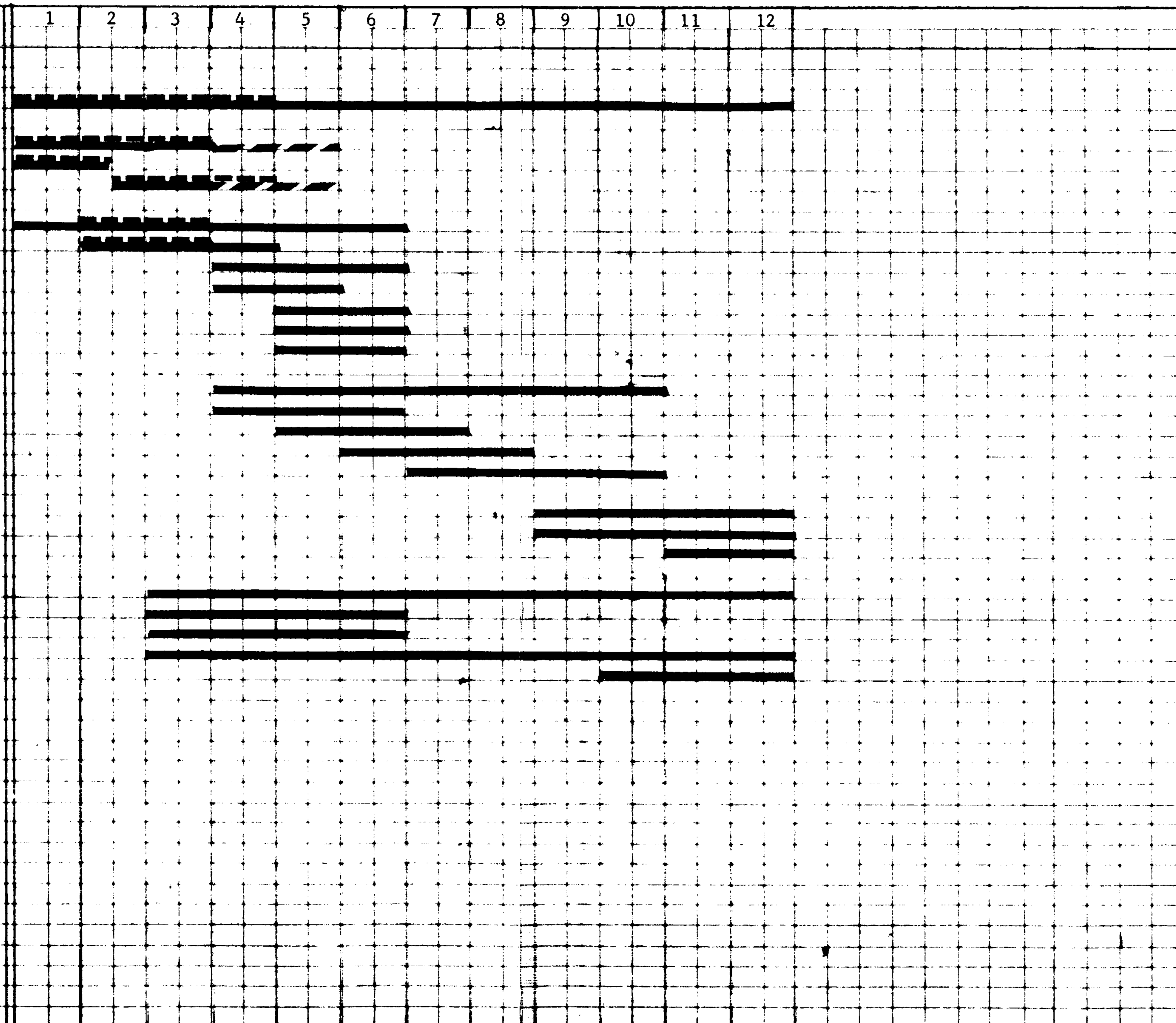
ANALYSIS AND PLANNING
- Fundamental Design Review
- Preliminary Cell Design

DEVELOPMENT STUDIES
- Separator Evaluation
- Special Plate Treatments
- Zinc Density Studies
- Plate Construction
- Electrolyte Studies
- Gassing Studies

CELL AND BATTERY CONSTRUCTION
- Element Design
- Hardware Design
- Prototype Cells
- Prototype Batteries

TESTING
- Prototype Cells
- Prototype Batteries

RELIABILITY EVALUATION
- Drift Analysis
- Stress Analysis
- Modes & Rates of Failure on Prototype Battery



AFTER GO-AHEAD

CALENDAR

MILESTONES

Schedule
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