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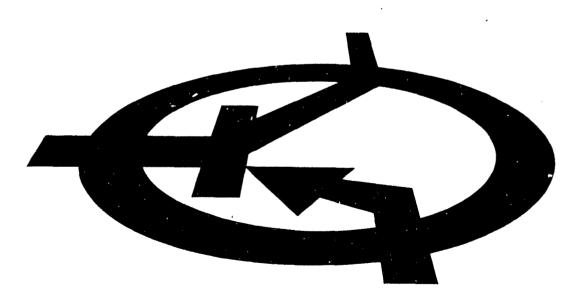
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HIGH CAPACITY MAGNESIUM BATTERIES REPORT NO. 6 SIGNAL CORPS CONTRACT NO. DA-36-039-SC-85340 DEPARTMENT OF THE ARMY PROJECT NO. 3A99-09-002

SIXTH QUARTERLY PROGRESS REPORT 1 September 1961 to 30 November 1961

> U.S. ARMY SIGNAL RESEARCH AND DEVELOPMENT LABORATORY FORT MONMOUTH, NEW JERSEY





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NO. 3A99-09-002

SIXTH QUARTERLY PROGRESS REPORT

1 September 1961 to 31 November 1961

The objective of this research and development project is the development of high-capacity magnes-ium primary batteries using the perchlorate electrolyte systems.

This report prepared by:

C. S. Ingler

R G Ryan

Approved by:

B.Schwartz, Manager

Component Development

Microelectronics Department Semiconductor and Materials

Division

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1. PURPOSE

The purposes of this research and development contract are to

- 1. Develop practical Mg-CuO and Mg-HgO reserve cells suitable for utilization in high-rate batteries.
- 2. Characterize the $Mg/Mg(ClO_{l_1})_2/MnO_2$ and $Mg/Mg(ClO_{l_1})_2/CuO$ dry cells.
- 3. Perform research studies to determine the factors and mechanisms controlling anode efficiency, inhibitor function, and delayed action of the magnesium anode with emphasis on the perchlorate electrolyte.

2. ABSTRACT

This abstract describes briefly the significant accomplishments and progress made on the U. S. Army Signal Research and Development Laboratories High Capacity Magnesium Battery Program by the Radio Corporation of America, Semiconductor and Materials Division, during the sixth quarterly period from September 1, 1961 to November 30, 1961. The subject matter in this abstract is divided into the major areas of endeavor for convenience of review.

An analysis of the heat evolved from magnesium cells is presented. Emphasis is placed on performance of mercuric oxide reserve cells at high discharge rates. Further development of $Mg/Mg(ClO_{l_1})_2/HgO$ high-rate reserve cells is summarized.

Delayed action and impedance data are presented for Mg/Mg(ClO₁)₂/MnO₂ "A"-size cells made with AZ-21 magnesium alloy and compared with that of other alloys. Data are also included for peak voltages observed in switching from high-to-low discharge drains. Shelf data are summarized for mangarese-dioxide and cupricoxide cells stored for one year.

3. CONFERENCES

On October 30, 1961, Dr. G. S. Lozier and Mr. R. J. Ryan visited the U. S. Army Signal Research and Development Laboratories at Fort Monmouth, New Jersey to discuss progress under the subject contract. Present at the meeting were Messrs. J. Murphy, H. Knapp, A. Almerini and D. Wood of the U. S. Army Signal Research and Development Laboratories.

4. EXPERIMENTAL AND FACTUAL DATA

4.1 MAGNESIUM PERCHLORATE RESERVE CELLS

Data presented in the previous quarterly reports of this contract showed that heat evolved from magnesium reserve cells during high-rate discharge was a major problem affecting efficient cell performance. A thorough analysis of heat evolved was made, and development studies continued on methods of heat control.

4.1.1 Analysis of Heat Evolution in Magnesium Reserve Cells

In a magnesium cell, two reactions occur at the anode

- 2. The corrosion side reaction

$$Mg + 2H_2O \longrightarrow Mg (OH)_2 + H_2$$

Both of these reactions produce heat. Reaction No. 1 gives off heat because of the irreversibility of the magnesium anode in aqueous electrolytes. Reaction No. 2 is exothermic. The heat given off by these reactions may be calculated with sufficient accuracy for present purposes.

The heat produced by reaction No. 1 may be calculated for any magnesium cell from the following equation:

 $W = A \cdot \nabla \cdot T$

Where W is watt-hours, A is current drain in amperes, V is the voltage difference between the reversible potential of the magnesium anode and the operating potential of the magnesium anode, and T is the time in hours. V is approximately 1.1 because, under most conditions, the magnesium anode operates at 1.2 to 1.3 volts vs. S.H.E., whereas the reversible potential is 2.34 to 2.40 volts vs. S.H.E. Heat in calories is obtained by multiplying watt hours by 860.

The heat due to the corrosion side-reaction is calculated by first determining the ampere-minutes of magnesium utilized in the corrosion reaction. This is given by the following equation:

$A.M. = A \cdot T \cdot E$

Where A is current drain, T is time and E is an efficiency factor determined by the ratio of percent of anode corrosion to percent of anode efficiency. The heat produced in calories is determined by relating ampere minutes to moles of magnesium multiplied by
H for the corrosion reaction which is 82.6 K cal/mole.

The total heat evolved from a magnesium cell per hour is due to the two-heat producing reactions, and is a function of current drain. Heat evolved under different current-drain conditions is shown in Figures 4-1 and 4-2. The 100 percent line represents heat evolved only by the irreversibility of the magnesium. The heat evolved from the corrosion reaction is presented in Figures 4-3 and 4-4.

The significance of the thermal data can be realized from the analysis of a mercuric oxide reserve cell at a constant current discharge of 10 amperes. Figure 4-1 shows that 13.5 kilocalories are evolved per hour at 10 amperes at 80 percent anode efficiency. For the 5-ampere-hour mercuric-oxide cell, 6.75 kilocalories are evolved in the half-hour discharge. This quantity of heat is sufficient to vaporize up to 11.5 grams of water.

Capacity data for a magnesium (AZ-21 alloy) - mercuric-oxide reserve cell discharged at a constant current of 10-amperes in 2N $Mg(ClO_{l_1})_2$ is presented in Figure 5. A heat sink of 150 ml of water was used. The cell was discharged for a total time of $36\frac{1}{4}$ minutes. The magnesium anode efficiency was 80.7 percent, as determined by measurement of weight loss. The total heat evolved during the discharge time was calculated from the data of Figure 1 at 80 percent efficiency and is 8.15 kilocalories.

Measurement of the temperature rise in the water heat sink and electrolyte showed a heat output of 4.15 kilocalories. The difference in the measured heat and that calculated from the data of Figure 4-1 may be accounted for by these factors:

- a) Reduced anode polorization at high temperatures.
- b) Inhibition of the anode by mercuric ion.
- c) Heat absorbed by the cell.
- d) Heat loss to boiling in center of cell.

The above analysis shows that an efficient heat sink is a necessary requirement in the design of high-rate magnesium cells. Without this heat sink, close voltage tolerances cannot be attained and cell capacity will be limited because of water loss. It should be noted that the resultant heat evolved is independent of cell size; most cathode materials used with magnesium operate close to their reversible potential, and their contribution to the total heat evolved is omitted in this analysis.

The data given in Figures 4-1 to 4-4 can be extended for use in magnesium battery design at any discharge rate. Previous studies of 6-volt magnesium batteries at a 12-hour rate showed excessive heat build-up after three hours discharge at room temperature. These studies indicate that heat evolved from magnesium dry-cell pack batteries can present a problem in confined systems. The data are extremely useful in low temperature battery design where conservation of heat is important.

4.1.2 <u>High-Rate Mercuric-Oxide Design Studies</u>

Previous experiments which used water as a heat sink showed that cells could be operated at the half-hour rate at 10 amperes within a voltage tolerance of ± 5%. Battery design necessitates the use of heat-sink methods other than water. Studies employing metal-jacket techniques were carried out; the results are summarized in the following sections.

4.1.2.1 Effect of Copper-Foil Wrap on Heat Dissipation

A three-cell five-ampere mercuric-oxide reserve battery using single cell plastic cases wrapped with copper foil was constructed. The three cells were wrapped as a unit and placed in contact with 0.125-inch-thick aluminum plate.

Discharge of the battery at 10 amperes constant current showed that this method of heat sinking was inadequate for high-rate discharge. Battery voltage could not be maintained at a close tolerance level, and additional water had to be added to the cells throughout the discharge to prevent cells from drying out. The discharge data are presented in Figure 4-6. Although this method of heat control was unsatisfactory at high discharge rates, it is sufficient for lighter drains.

4.1.2.2 Effect of Aluminum Cell Case with Air-Space on Heat Dissipation

A study was made of the use of an aluminum cell case in place of the rigid plastic case employed in previous tests. Cells constructed of three cathode plates measuring $3^n \times 3$ -1/8", and four commercial pure magnesium anodes had a theoretical cathode capacity of 300 ampere-minutes. The cell elements were placed in thin polyethylene bags and installed in an 1/16-inch-thick aluminum case with a 3/8-inch air space between cells. The cells were activated

with 19 ml of 2N magnesium perchlorate, and discharged at 10 amperes in contact with an aluminum base plate.

Discharge data showed poor voltage regulation because of excessive heat build-up within the cells. Figure 4-7 shows the discharge characteristics of two 3-cell constructions. The upper curve is for the 3-cell battery described above. The lower curve is based on data from a single cell having a water heat sink. Average voltage for the 3-cell battery was 5.74 volts to a 20 percent voltage drop. Computed data for the other battery shows an average voltage of 4.56 volts to the 20 percent voltage-drop point.

4.1.2.3 Cell-Design Parameters

Studies were continued to improve high-rate performance of the mercuric-oxide system. Test cells of thin-plate construction to increase surface area of the electrodes were assembled. A comparison of the characteristics of a cell of this construction discharged at 20 amperes constant current with a cell having thicker plates is presented in Figure 4-8. Cell data are as follows:

Cell A

Cathode

10 plate, 10 to 1 Hg0 to Shawfnigan plus 5% Ag₂0,2g.mix per .003" grid

Cell B

5 plates, 10 to 1 HgO to Shawinigan plus 5% Ag₂0,4g.mix per .005" grid

	Cell A	Cell B
Theoretical capacity	179 Amp-minutes HgO	179 Amp-minutes HgO
Anode	11 plates, .005" commercial pure Mg	6 plates, .005" commercial pure Mg
Total active Surface area	362 cm ²	181 cm ²
Element volume	0.975 in ³	0.822 in ³
Element weight (dry)	34.5 g	29.5 g
Element weight (wet)	51.6 g	46.6 g
Average voltage to 20% voltage drop	1.68	1.46
Cell capacity to 20% voltage drop	157 amp-minutes	90 amp-minutes
Watt hours	4.4, watt hours	2.2 watt hours
Cathode Efficiency	88%	50%

The elements were placed in a plastic case with plastic spacers, activated with $2N \, \text{Mg}(\text{ClO}_4)_2$ for two minutes, and discharged in a beaker with $140 \, \text{ml}$ water as a heat sink.

Figure 4-8 shows that a cell made of thin plates of increased surface area gives improved performance. The current density of cell B is 110 ma/cm², which is twice that of cell A. Data presented in Figure 7 of the Third Quarterly Report on this contract showed extreme anodic and cathodic polarization of the mercuric oxide reserve cell at this high-current density.

Performance is dependent on the use of an efficient heat sink.

The feasibility of using alternate techniques for temperature control in high-rate magnesium cells is being determined.

4.2 MAGNESIUM PERCHLORATE DRY CELLS

4.2.1 Delayed-Action Studies

Tests were conducted on AZ-21 and AZ-10 magnesium/magnesium perchlorate/manganese dioxide (Type M) "A"-size cells to compare delayed-action characteristics. Freshly made cells were tested in
groups of three on an 8.9-ohm 2-minute intermittent drain, and on
150-ohm 18-minute intermittent drain. Cell voltage variation recorded from the 18-minute to the 2-minute cycle with a Sanborn recorder. Voltage was also recorded from the 2-minute to the 18minute cycle to determine peak voltage characteristics from heavy
to light drains. This factor is of equal importance with the
minimum delay Voltage for many applications, particularly in transistor apparatus.

Data are presented in Figure 4-9 and 4-10 and in Table 4-1 for the delayed action of the two magnesium alloys measured at various times throughout the discharge. The results show that magnesium AZ-21 behaves similarly to AZ-31. Data for AZ-31 "AA" cells were presented in Figure 9 of the Second Quarterly Report. The voltage level of the AZ-21 is slightly lower than the AZ-10, as indicated by continuous-drain tests. Cell capacity was equivalent for both alloys, giving 50 hours to a 1.20-volt cut off.

Peak-voltage data are presented in Table 4-1. Data are taken from measurements made throughout the first minute of the 18-minute cycle. The results show that both alloys operate with voltage in excess of

1.80 volts in the initial 15 seconds of the cycle during the first eight hours of discharge. The AZ-21 voltage levels off faster than the AZ-10, and operates about 0.1 volt lower on the light drain. Thus, AZ-21 would be the more desirable alloy in applications where peak voltages are a problem.

The wide voltage range noted in this type of intermittent service must be considered carefully in the design of magnesium dry-cell packs. Raising the cut-off voltage per cell would minimize the peak-voltage, but would increase the lower delay voltage on the high-drain cycle.

4.2.2 AZ-21 Impedance

The impedance of "A"-size magnesium/magnesium-perchlorate/manganese dioxide (Type M) dry cells with AZ-21 and AZ-10 magnesium alloy was measured at a 50-ohm continuous discharge rate over a frequency range of 60 to 10,000 cps. Test results showed that the AZ-21 alloy impedance was slightly less than that of the AZ-10 alloy. The data for the three freshly made cells are summarized in Table 4-2.

4.2.3 Shelf Studies

4.2.3.1 Delayed Action and Impedance

Capacity, delayed-action, and impedance tests were run on $Mg/Mg(ClO_4)_2/MnO_2(Type M)$ and $Mg/Mg(ClO_4)_2/CuO$ "AA" size cells stored for 13 months. All cells were made with AZ-10 alloy. Delayed-action measurements showed no significant differences

from initial fresh-cell results as reported in the Second Quarterly Report of this contract.

Impedance tests showed a slight increase in impedance at the 25-ohm drain for cupric-oxide cells and the 50-ohm drain for the manganese-dioxide cells. The cupric-oxide cell impedance was 3.4 ohms at 60 cps and 1.9 ohms at 400 cps as compared to the initial values of 2.8 ohms and 1.6 ohms. The manganese-dioxide cell impedance was 4.2 ohms at 60 cps and 2.1 ohms at 400 cps as compared to the initial values of 4.0 and 2.1 ohms.

Cell capacities averaged 90 per-cent retention. Data for the various lots are summarized in Table 4-3.

4.2.3.2 AZ-21 Magnesium Storage Tests

Six-months storage of "A"-size magnesium (AZ-21)-magnesium perchlorate-manganese dioxide (type M) dry cells at 113°F and 95 per-cent relative humidity showed a capacity retention of 95 per-cent. Eight-cell perforations were found in the air-space region. The perforations were probably caused by electrolyte leakage resulting from poor seals. Other cells in the lot show evidence of electrolyte leakage around the seal. Additional cells were placed on storage for seal evaluation.

Six-months storage of "A"-size magnesium (AZ-21)-magnesium perchlorate-cupric oxide dry cells at 113°F and 95 per-cent relative humidity showed a capacity retention of 65 per-cent.

This capacity is lower than indicated previously in cupricoxide cell storage, and cannot be explained at this time.

Water content is a critical factor in cupric-oxide cells. Hence,
loss of water through the seal could be a factor in limiting
cell capacity. Three cell perforations were found in the lot.

4.3 RESEARCH STUDIES

4.3.1 Magnesium Impedance Studies

It has been shown theoretically that the corrosion film on magnesium anodes can be studied directly by means of cell impedance measurements. The resistance-ratio bridge used for impedance measurements was shown to have more than sufficient accuracy and sensitivity for these studies. From the impedance data, the following may be determined as a function of load current.

- 1. An approximation of the true anode area
- 2. The portion of the anode area covered by the corrosion film
- 3. The average thickness of the corrosion film

This method is useful for the study of various magnesium alloys, inhibitors, and electrolytes and in demonstrating their effects on anode performance.

Because only two independent measurements are made (capacitance and resistance), only two independent values can be calculated. The true anode area must be estimated on the basis of an equation

emperically derived from the test data or measured independently.

A BET apparatus is being constructed to make accurate measurements of the total surface area.

Tests were run using flat AZ-21 magnesium and commercial pure magnesium anodes in 2N Mg(ClO₄)₂ over a current density range of 0.1 to 10 ma/cm² based on the plane area of the anode. Analysis of the data is not complete. Conclusions of the initial tests will be withheld until further analysis, and testing are completed.

5. CONCLUSIONS

An analysis of the heat evolved during magnitum—cell discharge shows the heat is due to the irreversibility of the magnitum anode and to the corrosion.

Weat evalution involls is problem which must be controlled for efficient operation, particularly light who harge rates.

An efficient heat sink must be used at high discharge rates to control voltage tolerance. The use of a water sink permits good operation at rates as low as the seminate rate. Netal cell association is inadequate as a heat sink for high rate reserve cells.

A cathode efficiency of 88 per cent was obtained with a mercuric oxide reserve cell at a neight minute discharge rate by using a thin plate who like construction. Capacity based on element wight and volume was 39 watt-hours who low in,

Delayed-action studies of AZ-21 magnesium agnosium perchlorate magnesez dimide cells on a trancei er-type discharge showed this alloy to have have characteristics similar to AZ-31 alloy. Voltage rise observed in switching from high to low drain in this type of discharge must be considered in bottom decimal.

Impedance of AZ-21 magnesium magnesium perchlorate mengences distide "A"-size cells was slightly lower than comparable AZ-10 alloy cells.

Delayed action and impedance measurements of magnetium (AZ-10)-magnetium peruhlerate manganese dioxide and cupric evide cells stored for energy showed no significant difference from initial fresh-cell results. Cell capacities averaged 90 per cent.

6. PROGRAM FOR NEXT QUARTER

- 'a) Mercuric-oxide reserve studies will be continued on design criteria for optimum high-rate performance.
- b) Determine characteristics of mercuric oxide and cupric oxide reserve cells for low-rate applications.
- c) Studies of the magnesium dissolution process will be continued.
- d) The low-temperature properties of magnesium dry cells and reserve cells will be determined.

7. DISTRIBUTION OF HOURS

G. S. Lozier, Project Director	128.5	Hours
R. J. Ryan, Physical Chemist	314	Hours
E. Uhler, Physical Chemist	96	Hours
J. Vossen, Physicist	. 88	Hours
A. Lindabery, Technician	216	Hours
Miscellaneous Technical Personnel	63	Hours

CELL VOLTAGE

	•				<u>(v</u>	(volts)								
	AT END OF					•		;			rn `			
	(150ohms)	586. 15860	15800	ec 30sec.	Linda.	1 1/2min 2min.		1sec. 58ec.	288	0.10800	15880	30sec.	30sec. 45sec. ludn.	lafa.
After 30 minutes discharge		1.37	1.43	1.47	1.50	1.50		1.90	1,88		1.70	1.72		
After 8 hours discharge	1.60	1,32	1.35	1.37	1.39	1.40	1.40	1.80	1.7	1.67	1.57	1.58	1.59	1.60
After 24 hours discharge	1.55	1.40	1.41	1,41	1.41	1,40	1.40							
After 31 hours discharge	1.53	1.35	1.37	1.40	1.40	1.40	1.40	1.70	1.67	1.62	1.58	1,50	1.52	1.53
After 49 hours discharge	1.48	1.20	1,22	1.23	1.23	1.23	1.24	1.62	1.60	1.55	1.50	1.43	1.45	1.46
AZ-10 ALTOY			······································				,							
After 30-minutes discharge	1.73	1.44	1.45	1.46	1.46	1.47	1.48	1,88	1.83	1.78	1.72	1.73	1.74	1.74
After 8 hours discharge	1.66	1.50	1.50	1.49	1.49	1.48	1.48	1,88	1.89	1.88	1.87	1.78	1.72	1.70
After 25 hours discharge	1.66	1.45	1.45	1.44	1.44	1.43	1.42	1.76	1.76	1.75	1.73	1.70	1.68	1,68
After 50 hours discharge	1.53	1.23	1,21	1.20	1,20	1.19	1,19	1.60	1.60 1.63	1.63	1.62	1.61	1.60	1.59

Table 4-1. Voltage Variations of Mg/Mg(GlO₄)₂/MmO₂(Type M) "A" Size Cells with AZ-10 and AZ-21 Megnesium Alloy on Intermittent Service.

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AZ-10 MAGNESIUM

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	CELLS		MPEDA	IMPEDANCE IN OFMS	OHAC		CELIS		PEDAN	MPRDANCE TN OPER	PAG	
į	VOLTAGE		E	FREQUENCY	X		VOLTAGE		FE	FREDITENCY		
N I	(volts)	8	8	87	1,5	10k	(volts)	8	100	71 007	当	, 101
15 minutes	1.69	3.5	3.5 4.5	2.0 1.47 0.96	1.47	96.0	1.71	3.8	3.8 4.6		1.9	1.2
7 hours	1.56	2.6	2.3	2.6 2.3 0.86 0.69 0.58	69.0	0.58	1.59	3.4	3.4 3.2	1.7	1.09 0.69	69.0
24 hours	1.50	2.9	2.8	2.9 2.8 1.20 0.77 0.57	0.77	0.57	1.54	3.6	3.6 3.2	1.7	1.07 0.69	69.0
32 hours	1.46	2.9	8.	2.9 2.8 1.14 0.70 0.57	0.70	0.57	1.50	3.5	3.5 3.2	1.6	1.02 0.70	0.70
49 hours	1.00	4.0	3.4	4.0 3.4 1.34 0.91 0.70	0.91	0.70	0.93	4.0	3.6	1.54	4.0 3.6 1.54 0.99 0.71	0.71
				-						-		

Table 4-2. Impedance Data for $Mg/Mg(GlO_4)_2/MmO_2$ (Type M) "A" Size Cells Discharged at a Constant Resistance of 50 ohms.

TYPE OF CELL	DISCHARGE DRAIN	INITIAL CAPACITY (hours)	STORAGE CONDITIONS	CAPACITY RETENTION (PER CENT)
MnO ₂ (Type M) "A"	16 2/3 ohms continuous	15 1/4	13 months at 113°F	92
MmO ₂ Type M "AA"	50 ohm con- tinuous	35	14 months at 113°F	90
MmO2 Type M "AA"	150 ohm for 18 minutes 8.9 ohm for 2 minutes	28	14 months at 113°F	86
CuO "AA"	25 ohm continuous	58	13 months at 70°F	96
CuO "AA"	91 ohm for 18 minutes 5.4 ohm for 2 minutes	34	13 months at 70°F	90

Table 4-3. Capacity Retention Data for $Mg/Mg(ClO_4)_2/MmO_2$ and $Mg/Mg(ClO_4)_2/CuO$ Dry Cells for Various Discharge Rates.

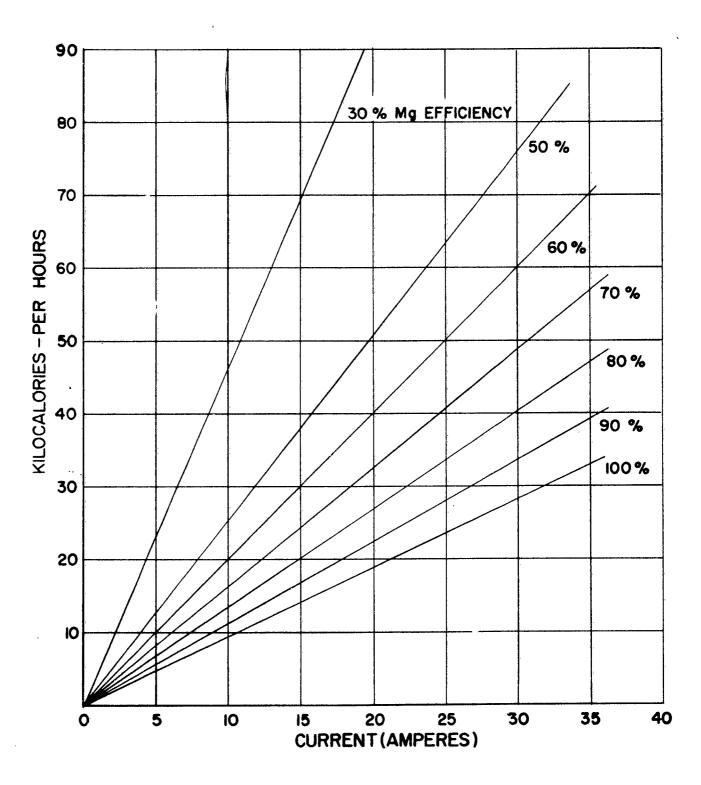


Figure 4-1. Salaries Evalved from Magnesium Electrode per Hour at Various Mg Efficiencies at High Discharge Rates.

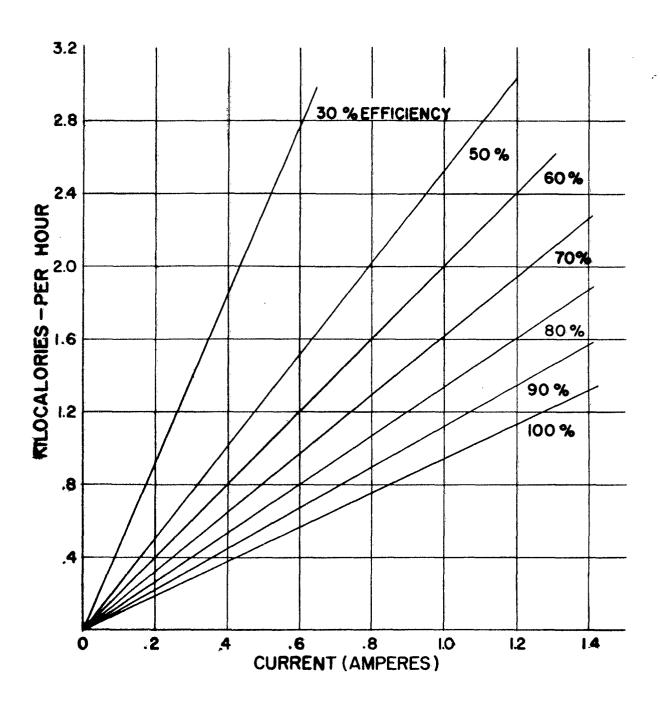


Figure 4-2. Galeries Evelved from a Magnesium Electrode per Hour at Various Amode Efficiencies at Low Discharge Entes.

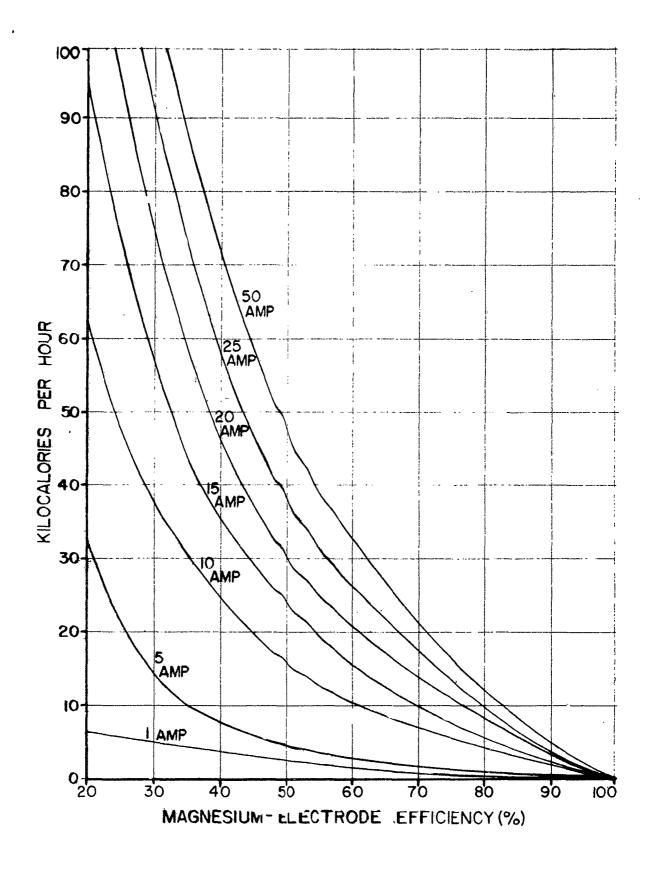


Figure 4-3. Calories Evolved from Magnesium Corrosion Reaction as a Function of Electrode Efficiency at High Discharge Rates.

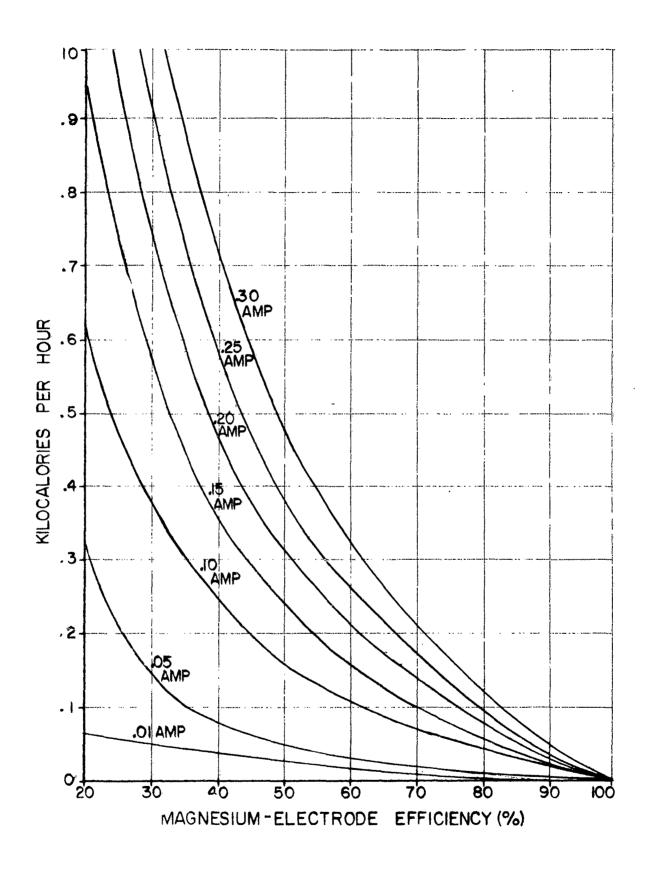


Figure 4-4. Calories Evelved from Magnesium Corresion Reaction as a Function of Electrode Efficiency at Low Discharge Rates.

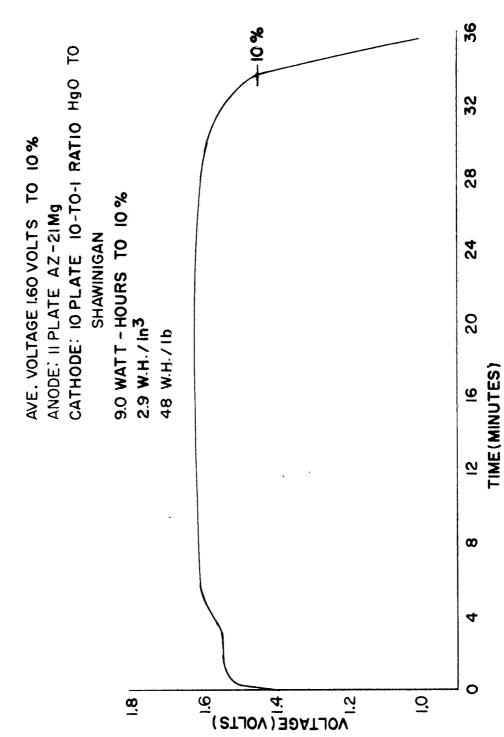


Figure 4-5. Capacity Data for Mg/Mg(ClO4)2/HgO Reserve Cell Discharged at 10 Amperes.

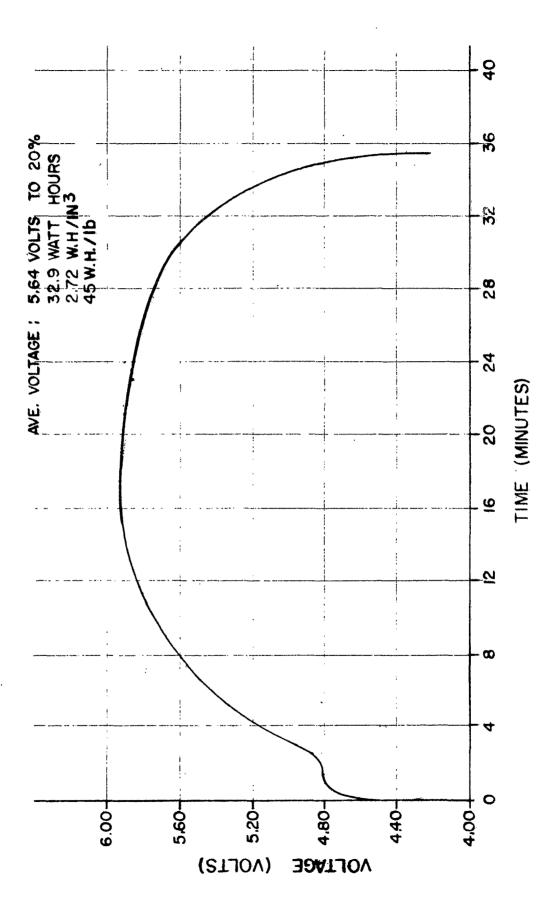


Figure 4-6. Three-Sell Mg/Mg(GlOg)2/HgO Reserve Battery with Copper-Foil Jacket Mischarged at 10 Amperes.

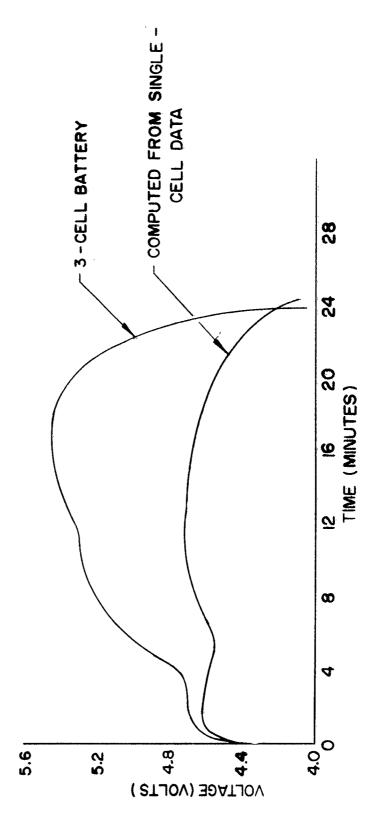


Figure 4-7. Capacity Data for a Three-Cell Mg/Mg(ClO,)2/HgO Reserve Battery with Aluminum Case Construction Discharged at 10 Amperes.

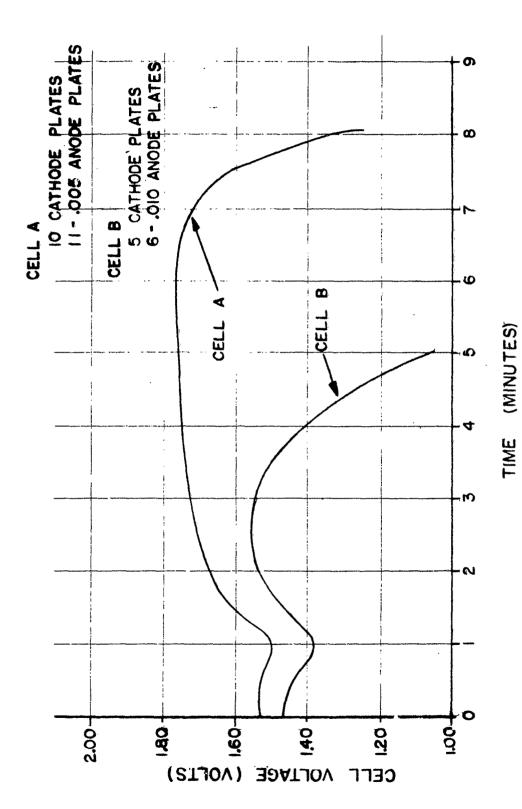


Figure 4-8. Capacity Data for Mg/Mg(ClO₄)₂/HgO Reserve Cells Discharged at a Constant Current of 20 Amperes.

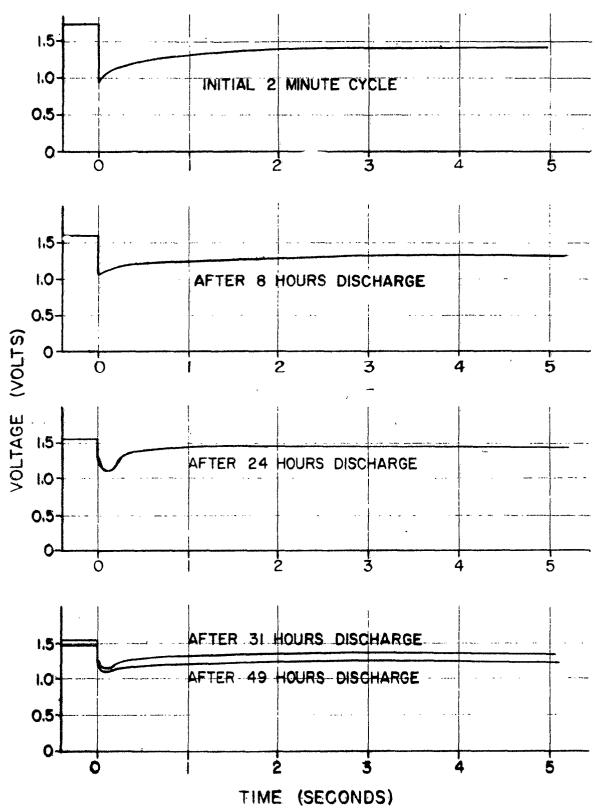


Figure 4-9. Delayed-Action Characteristics of "A"-sime Mg(AE-21)Mg(G10₂)₂/MmO₂ (Type M) Sells at 150-chm to 8.9-4m Brain.

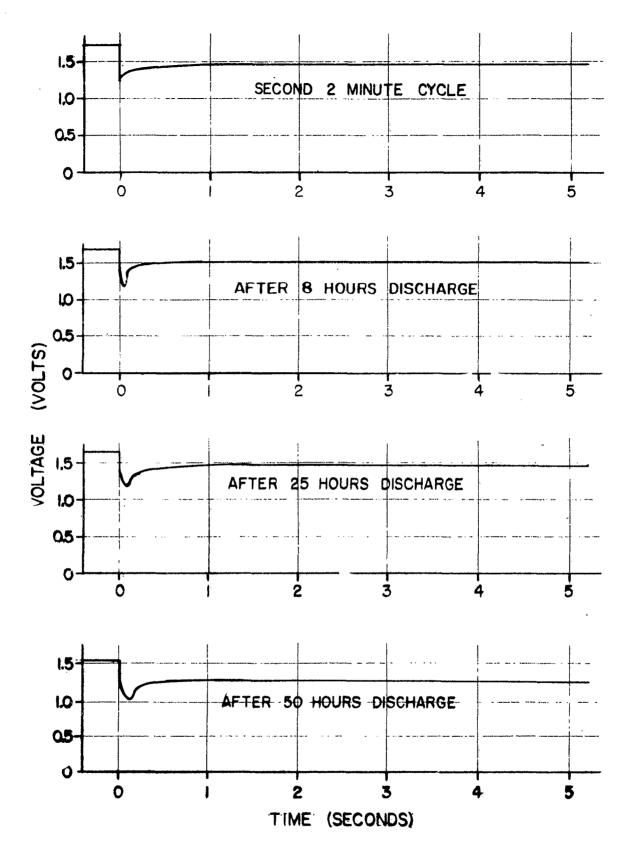


Figure 4-10. Belayed Artism tenting testies of "A" Size Mg(AZ-10)/Mg(6104), Amb2 (Type M) Cells at 150-obs to 8.9-obs brein.

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