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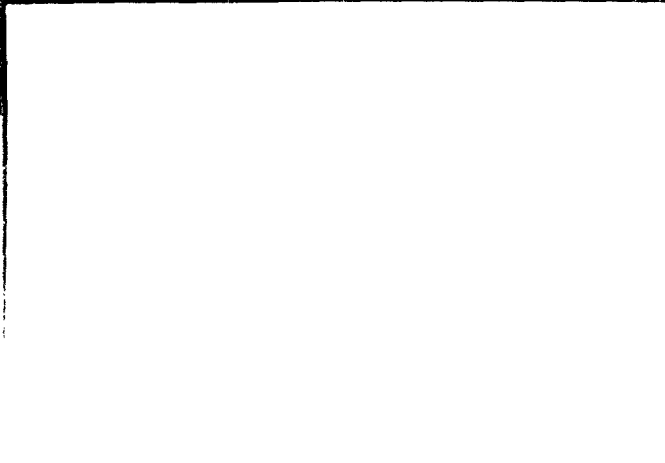
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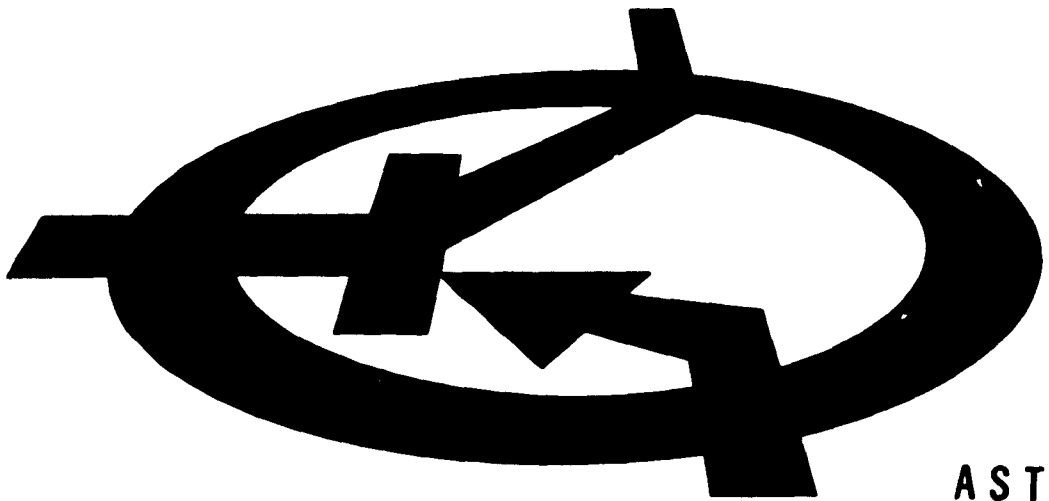
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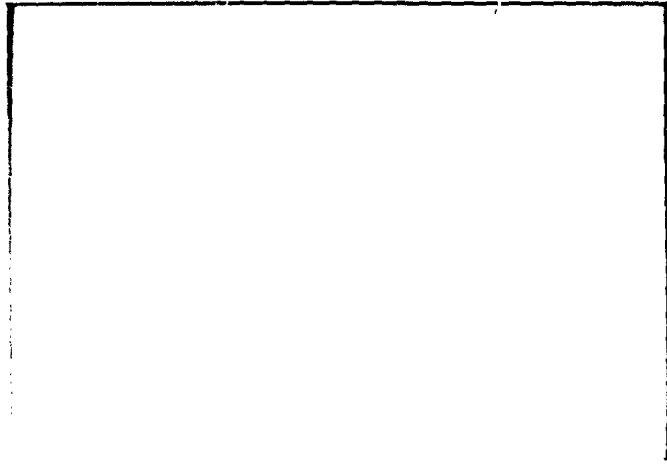
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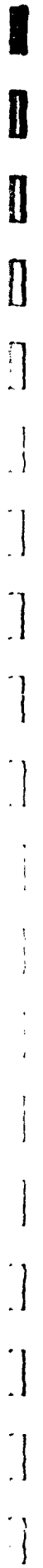


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**HIGH-CAPACITY MAGNESIUM BATTERIES
REPORT NO. 8**

**SIGNAL CORPS CONTRACT NO.
DA-36-039-SC-85340**

**DEPARTMENT OF THE ARMY
PROJECT NO. 3A99-09-002.**

EIGHTH QUARTERLY PROGRESS REPORT

1 MARCH 1962 to 31 MAY 1962

**U.S. ARMY SIGNAL RESEARCH
and
DEVELOPMENT LABORATORY
FORT MONMOUTH, NEW JERSEY**

**Prepared By:
RADIO CORPORATION OF AMERICA
Semiconductor and Materials Division
Somerville, New Jersey**

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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(ClO₄)₂/MnO₂ and Mg/Mg(ClO₄)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 eight quarterly period from March 1, 1962 to May 31, 1962.

Data are presented which show the low-temperature characteristics of magnesium-magnesium perchlorate-mercuric oxide and cupric oxide reserve cells at ambient temperatures down to -58°F .

Capacity data for three cell batteries are also included.

Data from mercuric oxide cell studies conducted to evaluate the use of a titanium cathode grid are summarized.

Low-temperature capacity data for magnesium-magnesium perchlorate-manganese dioxide A-cells and 6-cell batteries discharged at -20°F are presented.

Results from magnesium anode efficiency studies are summarized for pure magnesium and AZ-21X1 alloy over a current density range of 0.09 to 10.9 ma/cm^2 . A correlation of the efficiency data with magnesium corrosion film studies is also presented.

3. CONFERENCES

On March 22, 1962, 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 A. Alverini, J. Hovendon, C. Trigg and D. Wood of the U. S. Army Signal Research and Development Laboratories.

On May 8, 1962, Mr. C. Trigg of the U. S. Army Signal Research and Development Laboratories visited RCA in Somerville, New Jersey, to discuss progress under this contract. Present at the meeting were Dr. G. S. Lozier, and Mr. R.J. Ryan of RCA.

4. EXPERIMENTAL AND FACTUAL DATA

4.1 MAGNESIUM PERCHLORATE RESERVE CELLS

Data were presented in the Seventh Quarterly Report of this contract showing the characteristics of mercuric oxide and cupric oxide reserve cells at discharge rates from one to eight hours. These studies were continued during this report period to determine the characteristics of these systems during low-temperature operation.

4.1.1 Magnesium-Mercuric Oxide Cells

4.1.1.1 Low-Temperature Studies

Mercuric oxide cells of 277 ampere-minutes theoretical cathode capacity were used for all characterization studies; the construction of these cells was described in the Seventh Quarterly Report. Two types of cell cases were used for the low-temperature studies, a non-insulating rigid plastic case and a similar case with a one-half inch thick polyurethane foam jacket added. The cells were activated with 5N magnesium-perchlorate electrolyte at room temperature and allowed to stand at the test ambient temperature for a period of five minutes prior to starting the discharge. This type of activation procedure is similar to that used in meteorological applications.

The capacity data for single cells discharged at constant-current drains of 0.5 to 4.0 amperes and at temperatures as low as -58°F are presented in Table 4-1. The data show that room temperature capacity can be approached at low temperatures by insulating the cell case to conserve the heat evolved from the magnesium reactions. With the 4.0-ampere discharge, a sufficient internal cell temperature is maintained to give room-temperature capacity. The capacity of the non-insulated cell at an ambient temperature of -40°F drops to 60 percent of the room temperature capacity.

Discharge curves for both cells at an ambient temperature of -40°F are presented in Figure 4-1. Data for the cells at an ambient temperature of -58°F are presented in Figure 4-2.

Three cell batteries were constructed of cells similar to those described above to determine the low-temperature characteristics of a multiple-cell unit. The three-cell batteries were activated with room-temperature 5N magnesium-perchlorate electrolyte as in the previous single-cell tests and discharged at a one-ampere rate. Discharge curves for these batteries, both with and without polyurethane insulation, at an ambient temperature of -40°F are presented in Figure 4-3. The data show that full room-temperature capacity was obtained with the insulated battery. For the non-insulated battery, 50-percent capacity was obtained to a 20-percent voltage-drop end point. The gradual fall off in voltage is caused by heat loss from the cells. It should be noted that magnesium-mercuric oxide cells have an operating voltage of 1.20 volts at -40°F where no heat is conserved.

Capacity data for a three-cell battery discharge at 1.0 ampere at an ambient temperature of -58°F are presented in Figure 4-4. A one-half inch thick polyurethane foam jacket was used for insulation. The data shows that 85 percent of the room temperature capacity was obtained to a 10-percent voltage-drop end point.

In the above studies cells were activated with electrolyte at 70°F . For cells activated at low ambient temperatures the initial operating voltage is equal to the operating voltage for that temperature as given in Figure 4 of the Fifth Quarterly Report of this contract. Cell voltage then rises during the discharge as heat is generated in the cell reactions. It is apparent from the above studies that magnesium-mercuric oxide batteries can be designed with excellent low-temperature characteristics.

4.1.1.2 Evaluation of Titanium Grids

Mercuric oxide cathode plates require either a pure silver or a silver-plated copper grid to provide good performance. These grids however may be limited in various applications because silver-plated grids breakdown on activated stand tests as previously reported. Pure silver grids also can break down on long discharge rates (24 hours and over) due to amalgamation with the mercury from the cathode reduction. The use of an expanded titanium grid was studied for use with mercuric oxide cathodes. Four cells were constructed using 2-inch square cathode plates made with 10Ti1C-1/0 expanded titanium grids. Theoretical cathode capacity was 277 ampere-minutes. Two cells made with pure magnesium anode plates

were discharged at four-hour and eight-hour rates to a 20-percent voltage-drop end point at an ambient temperature of 70°F.

The results of the tests showed that these cells had a capacity equivalent to cells with a silver-plated copper grid. The initial cell voltage was 0.20 volt lower than comparable silver-grid cells; the voltage then increased to the silver-grid cell value after 25 percent of the discharge. This effect is probably associated with a surface film on the titanium grid. Two cells were also made with AZ-31 magnesium anode plates and discharged at the 24-hour rate both immediately after activation with 5N magnesium-perchlorate electrolyte and after a week activated stand. The data are plotted in Figure 4-5 and show a nine percent drop in capacity after the one-week stand. Anode efficiency measured 64 percent for the cell discharged immediately and 59 percent for the cell discharged after a one-week stand. It can be concluded from these tests that titanium grids can be used in low-rate mercuric oxide cells where silver grids are limited.

4.1.2 Magnesium-Cupric Oxide Cells

4.1.2.1 Low-Temperature Studies

Magnesium-cupric oxide cells with a theoretical cathode capacity of 590 ampere-minutes were tested at discharge rates from two to eight hours at ambient temperatures as low as -58°F. Cells were activated at room temperature with 5N magnesium-perchlorate electrolyte and allowed to stand for five minutes at the test ambient temperature before

the start of discharge. The capacity data as determined by the tests are presented in Table 4-2. Representative curves for cells discharged at 2.0 amperes and 4.0 amperes at various temperatures are shown in Figures 4-6 and 4-7.

The test data show that room-temperature capacity is obtained at temperatures down to -58°F for cells with a polyurethane jacket. Capacity of noninsulated case cells discharged at -4° and -40°F dropped to less than 20 percent of optimum capacity. These results show that the cupric-oxide cathode is highly temperature dependent and that control of heat within the cell will affect cell capacity. Also, the cupric-oxide cathode has a high temperature coefficient and wide variation of cell voltage can be obtained. Data were presented in Figures 10 and 11 of the First Quarterly Report of this contract which showed the variation of magnesium-cupric oxide cell voltage with temperature over a temperature range of 0 to 80°C . This variation is evidenced in the present study by the increase in cell voltage of cells enclosed in a one-half inch polyurethane case as shown in Figures 4-6 and 4-7. The high operating voltage indicates an internal cell temperature above 40°C .

The low temperature studies of the cupric oxide system described above show that good capacity can be obtained down to at least -58°F . Cell operating temperature however, must be controlled by the battery design to insure proper operating voltage.

4.2 MAGNESIUM PERCHLORATE DRY CELLS

The results of initial studies of the low-temperature characteristics of $Mg/Mg(ClO_4)_2/MnO_2$ dry cells were summarized in the Seventh Quarterly Report of this contract. Data were presented for cells with a 3.6N $Mg(ClO_4)_2$ electrolyte. These studies were continued during this quarter to determine the characteristics of cells with a 5N $Mg(ClO_4)_2$ electrolyte.

Capacity data for A-cells discharged through a 16- Ω ohm load at 70°F and at -10°F are presented in Figure 4-8. The data shows that the cell capacity is sharply reduced at the lower temperature with the cell having only 26.5 percent of its room-temperature capacity.

The low-temperature tests described above were conducted with no covering or insulation. In an effort to study the effects of insulation on cell capacity, two six-A-Cell battery packs were constructed, one with a cardboard case and one with a polyurethane-foam-potted case. The cardboard-case battery measured 2.05 x 1.425 x 2.175 inches and weighed 123.6 grams; the foam-case battery measured 2.675 x 1.85 x 3.075 inches and weighed 136 grams. The six-cell batteries were tested by storing at -20°F for 16 hours and subsequently discharging them through a 100-ohm resistor. The capacity data are presented in Figure 4-9. The data show that a discharge time of 3 hours and 45 minutes was obtained with the foam insulated battery to a 5.4-volt end point with an average voltage of 7.55 volts compared to a discharge time of 2 hours and 10 minutes for the cardboard case battery with a 6.2-volt average voltage. Although an improvement was realized with the foam insulation, only one-third of room-temperature capacity was obtained.

The batteries from the above tests were then placed back on discharge at room temperature and full capacity was obtained from both batteries for the combinations of low-temperature and room-temperature discharge.

A study was made of the differences in cell components at temperatures of 70°F and -20°F to determine possible causes of loss of capacity at low temperatures. Cell anodes, cathode bobbin, separators, and carbon rods were measured and examined at the two temperatures. No change was noted in dimensions of the anodes or carbon rods. The separator and the cathode mix appeared wet and similar to room temperature appearance at the -20°F temperature. The only significant variation noted was a slight loosening of the carbon rod in the cathode mix. Cell structure and fabrication studies are continuing for the purpose of improving low-temperature performance.

4.3 MAGNESIUM ANODE EFFICIENCY STUDIES

Data were presented in previous quarterly reports of this contract which showed the variation of magnesium anode efficiency with alloy type, temperature, and type of discharge rate. Figure 8 of the Fourth Quarterly report showed the variation of AZ-10 magnesium anode efficiency as a function of current density. Anode efficiency studies were continued during this report period to determine the efficiency of pure magnesium and AZ-21 alloy, at various current densities and to correlate the efficiencies with data from impedance studies.

4.3.1 Anode Efficiency

The cell geometry used was similar to that described for the impedance study described in section 4.3.5 of the Seventh Quarterly Report. Magnesium

anodes 0.125 inch in diameter with an 0.5 inch exposed length were used with a platinized platinum cathode in 2N magnesium-perchlorate electrolyte. The cells were discharged at various constant-current drains. No inhibitors were used in the electrolyte. The anodes were cleaned in acetic acid pickle solution prior to the discharge and in a chromic-acid solution immediately following the discharge. The anode efficiencies were then determined based on the resultant weight losses.

Figure 4-10 shows the anode efficiency of the two alloys as a function of current density. The data show a nearly constant efficiency for the AZ-21X1 alloy. This, together with its excellent low-discharge rate efficiency, makes this alloy desirable for use in magnesium dry cells and low-rate reserve cells. The efficiency measured for the AZ-21X1 alloy anode was somewhat lower than expected. The reason for this reduced efficiency is not known at present. In practice, efficiencies as high as 80 percent have been obtained for this material used in reserve cells. The pure magnesium anode has a much higher efficiency than the AZ-21X1 alloy anode at high-current densities which results in optimum performance in high rate reserve cells. At current densities below 3 ma/cm² the efficiency falls off sharply, limiting the use of pure magnesium at low discharge rates.

Cell polarization data is presented in Table 4-3 together with the efficiency data and the anode composition.

4.3.2 Correlation of Anode Efficiency with Magnesium Corrosion

Film Studies

A study method, based on impedance measurements, for magnesium dissolution

processes was described in the Seventh Quarterly Report of this contract. Measured values of the capacitance and resistance of the magnesium corrosion film were related to film area and thickness and predictions were made of the anode efficiency of AZ-21X1 and pure magnesium. Figures 4-11 and 4-12 show the relationship between exposed anode area and anode efficiency as a function of current density. The data show that the exposed area ($A_T - A_f$) decreases with decreasing current density to a point where the total anode surface is covered with corrosion film. This occurs at 1.7 ma/cm^2 for pure magnesium and 0.3 ma/cm^2 for the AZ-21X1 alloy.

The rate of corrosion film formation is much greater for the pure magnesium as indicated by the higher current density required to have exposed magnesium present. Below this current density the corrosion film formation rate increases as the load-current density decreases, resulting in a decrease in anode efficiency. Good agreement is shown between the anode efficiency curves and the data predicted in the Seventh Quarterly Report.

5. CONCLUSION

Low-temperature characterization of magnesium-magnesium perchlorate-mercuric oxide reserve cells show that room-temperature capacity can be obtained down to -58°F with the use of a polyurethane insulated cell case. Fifty percent of room-temperature capacity is obtained at -40°F for noninsulated cells.

Characterization of magnesium-magnesium perchlorate-cupric oxide reserve cells show the capacity drop to below twenty percent of optimum at -40°F . Good capacity can be obtained however with the use of an insulated cell case.

A titanium cathode grid was shown to perform satisfactorily in the mercuric oxide reserve cell. Activated stand tests showed a nine percent loss in capacity after a one week stand

Capacity data for magnesium-magnesium perchlorate-manganese dioxide A-cells and six cell batteries show only 25 percent of room temperature capacity is obtained at -20°F .

Anode efficiency studies showed efficiencies over 80 percent are obtained for pure magnesium at high current densities. Efficiency of pure magnesium falls off sharply at current densities below 3 ma/cm^2 .

Data for AZ-21X1 magnesium alloy show nearly constant efficiency with good efficiency at the low-current densities. Good correlation was obtained between the anode efficiency data and magnesium corrosion film studies presented in the Seventh Quarterly Report.

6. PROGRAM FOR THE NEXT QUARTER

1. Low-temperature studies of magnesium-magnesium perchlorate-manganese dioxide dry cells to improve performance will be continued.
2. The performance of manganese dioxide reserve type cells will be determined.
3. The magnesium corrosion film studies will be continued.

7. DISTRIBUTION OF HOURS

		<u>HOURS</u>
G. S. Lozier	Project Director	96
R. J. Ryan	Physical Chemist	200
E. F. Uhler	Physical Chemist	39
J. B. Eisen	Physical Chemist	58
J. Vossen	Physicist	78
M. Kuettel	Technician	237
A. Lindabery	Technician	56
P. Nichol	Technician	384
L. Trager	Technician	12
Miscellaneous Technical Personnel		89

DISCHARGE CURRENT (Amperes)	AMBIENT TEMPERATURE (°F)	CAPACITY TO 20-PER- CENT VOLTAGE DROP			CELL CASE
		DISCHARGE TIME		AVERAGE VOLTAGE (Volts)	
		HOURS	MIN.		
0.5	+70°	8	45	1.87	noninsulated plastic
0.5	-4°	5		1.53	noninsulated plastic
0.5	-40°	6	15	1.18	noninsulated plastic
0.5	-4°	6	30	1.62	1/2-inch polyfoam
0.5	-40°	4	10	1.51	1/2-inch polyfoam
0.5	-58°	2		1.55	1/2-inch polyfoam
1.0	+70°	4	22	1.81	noninsulated plastic
1.0	-4°	2	10	1.57	noninsulated plastic
1.0	-40°	2	30	1.09	noninsulated plastic
1.0	-4°	3	50	1.67	1/2-inch polyfoam
1.0	-40°	3	11	1.69	1/2-inch polyfoam
1.0	-58°	3		1.64	1/2-inch polyfoam
2.0	+70°	2	5	1.79	noninsulated plastic
2.0	-4°	1	15	1.56	noninsulated plastic
2.0	-40°	1	14	1.36	noninsulated plastic
2.0	-4°	2	12	1.67	1/2-inch polyfoam
2.0	-40°	1	49	1.63	1/2-inch polyfoam
2.0	-58°	1	41	1.66	1/2-inch polyfoam
2.0	-58°	1	6	1.74	1/2-inch polyfoam

Table 4-1. Capacity Data for Mg/Mg(ClO₄)₂/H₂O Reserve Cells at Various Discharge Rates and Temperatures.

DISCHARGE CURRENT (Amperes)	AMBIENT TEMPERATURE (°F)	CAPACITY TO 20-PER-CENT VOLTAGE DROP		AVERAGE VOLTAGE (Volts)	CELL CASE
		DISCHARGE TIME			
		HOURS	MIN.		
1.0	70°	8	30	1.06	noninsulated plastic plus heat sink
2.0	70°	4	20	1.10	noninsulated plastic plus heat sink
2.0	-4°	4	49 (1)	1.29	1/2-inch polyfoam
2.0	-40°	4	5	1.02	1/2-inch polyfoam
2.0	-58°	4	23	1.06	1/2-inch polyfoam
4.0	70°	2	3	1.06	noninsulated plastic plus heat sink
4.0	-4°	2	2 (1)	1.35	1/2-inch polyfoam
4.0	-40°	2	21 (1)	1.26	1/2-inch polyfoam
4.0	-58°	2	4 (1)	1.33	1/2-inch polyfoam
1.0	-4°	4	10 (2)	.98	1/2-inch polyfoam
1.0	-40°	4	20 (2)	.95	1/2-inch polyfoam
1.0	-58°	4	50 (2)	.96	1/2-inch polyfoam
2.0	-4°		50	1.00	noninsulated plastic
2.0	-40°		48	1.02	noninsulated plastic
2.0	-4°		27	.89	noninsulated plastic

(1) to 1.10 volts

(2) to 0.80 volt

Table 4-2. Capacity Data for Mg/Mg(ClO₄)₂/CuO Reserve Cells at Various Discharge Rates and Temperatures.

APPARENT CURRENT DENSITY (ma/cm ²)	PURE MAGNESIUM (1)		AZ21X1 ALLOY (2)	
	CELL(3) VOLTAGE (volts)	ANODE EFFICIENCY (percent)	CELL(3) VOLTAGE (volts)	ANODE EFFICIENCY (percent)
10.9	0.59	82.3	0.50	66.9
7.8	0.64	81.8	0.54	67.1
3.8	0.73	77.4	0.61	62.2
2.0	0.82	73.6	0.64	67.1
0.46	0.94	49.7	0.64	64.5
0.09	1.20	33.9	0.92	59.0

Notes:

(1) Dow Sublimed Mg

%Al	Ca	Cu	Fe	Mn	Ni	Pb	Si	Sn	Zn
<.003	<.01	<.001	<.001	<.001	<.0005	<.002	<.01	<.01	.003

(2) Dow AZ21X1

1.96	.15	<.001	.0014	.016	<.0005	.004	<.01	<.01	1.08
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(3) Magnesium vs. platinized-platinum.

Table 4-3. Magnesium Anode Efficiency Data in
2N Magnesium Perchlorate Electrolyte.

APPARENT CURRENT DENSITY (ma/cm ²)	PURE MAGNESIUM (1)		AZ21X1 ALLOY (2)	
	CELL(3) VOLTAGE (volts)	ANODE EFFICIENCY (percent)	CELL(3) VOLTAGE (volts)	ANODE EFFICIENCY (percent)
10.9	0.59	82.3	0.50	66.9
7.8	0.64	81.8	0.54	67.1
3.8	0.73	77.4	0.61	62.2
2.0	0.82	73.6	0.64	67.1
0.46	0.94	49.7	0.64	64.5
0.09	1.20	33.9	0.92	59.0

Notes:

(1) Dow Sublimed Mg

%Al	Ca	Cu	Fe	Mn	Ni	Pb	Si	Sn	Zn
<.003	<.01	<.001	<.001	<.001	<.0005	<.002	<.01	<.01	.003

(2) Dow AZ21X1

1.96	.15	<.001	.0014	.016	<.0005	.004	<.01	<.01	1.08
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(3) Magnesium vs. platinized-platinum.

Table 4-3. Magnesium Anode Efficiency Data in
2N Magnesium Perchlorate Electrolyte.

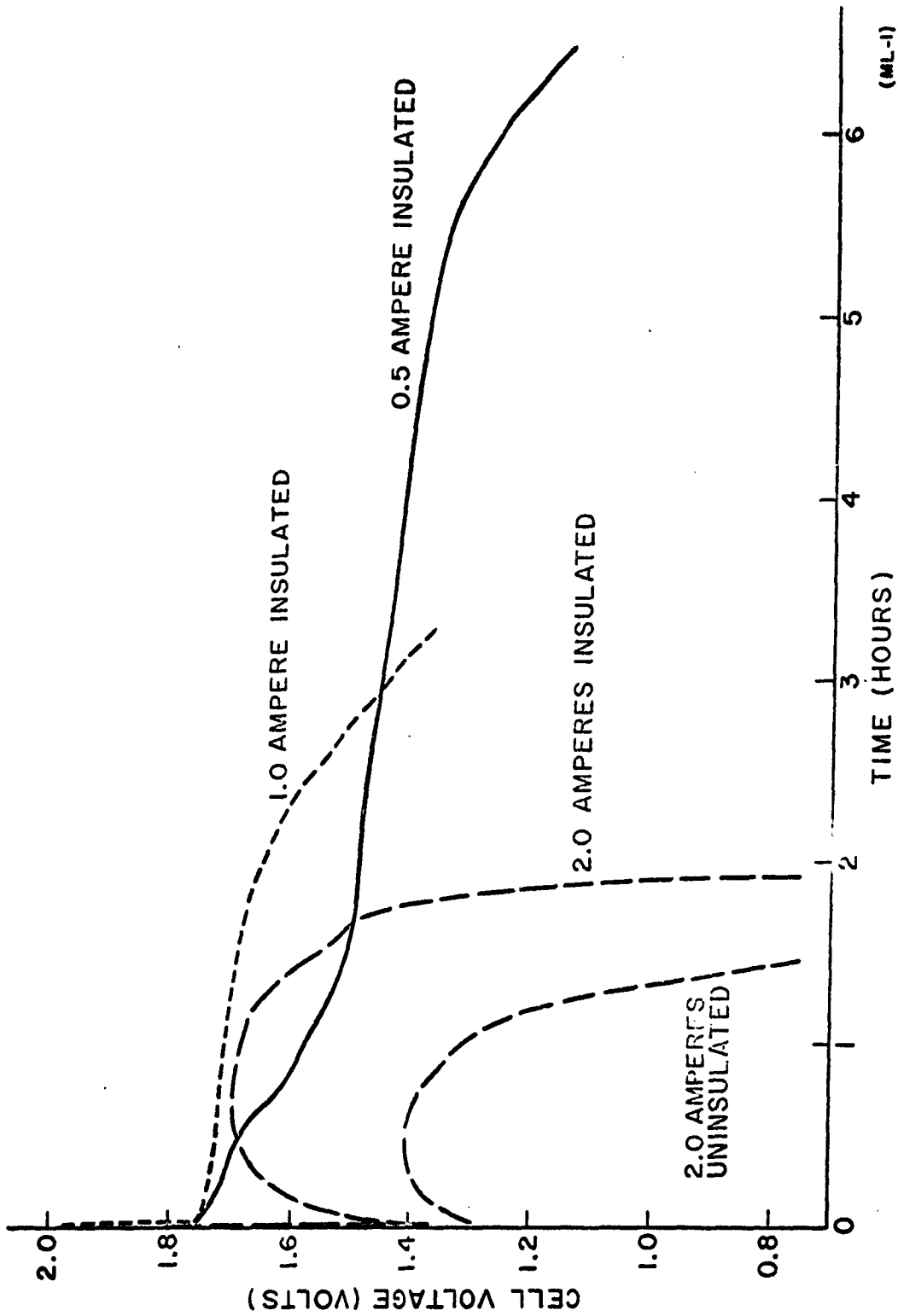


Figure 4.1. Capacity Data for Mg/Hg(ClO₄)₂/HgO Reserve Cells at Various Discharge Rates at -40°F.

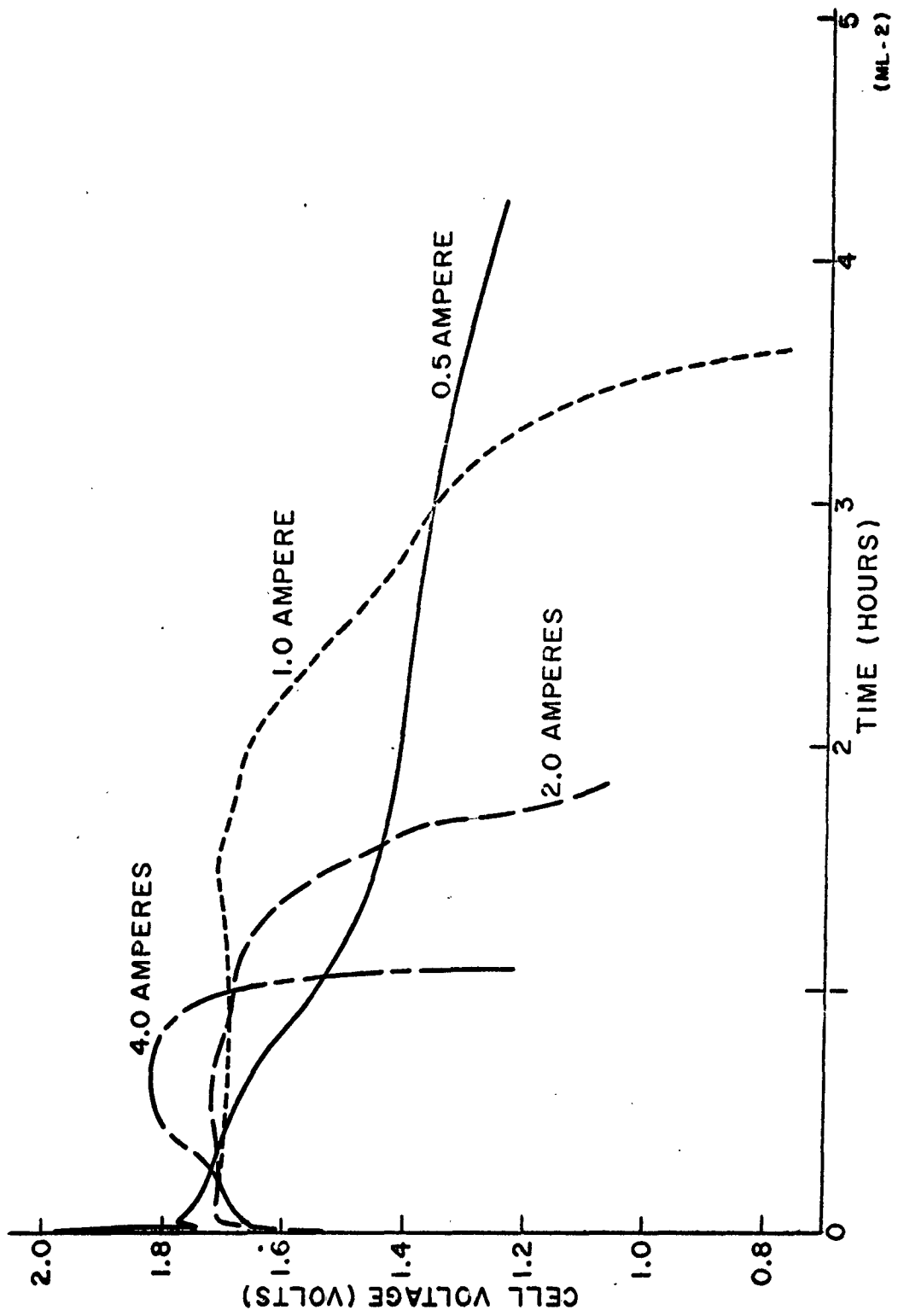


Figure 4-2. Capacity Data for Mg/Ag(ClO₄)₂/HgO Reserve Cells at Various Discharge Drains at -58°F. All Cells Insulated.

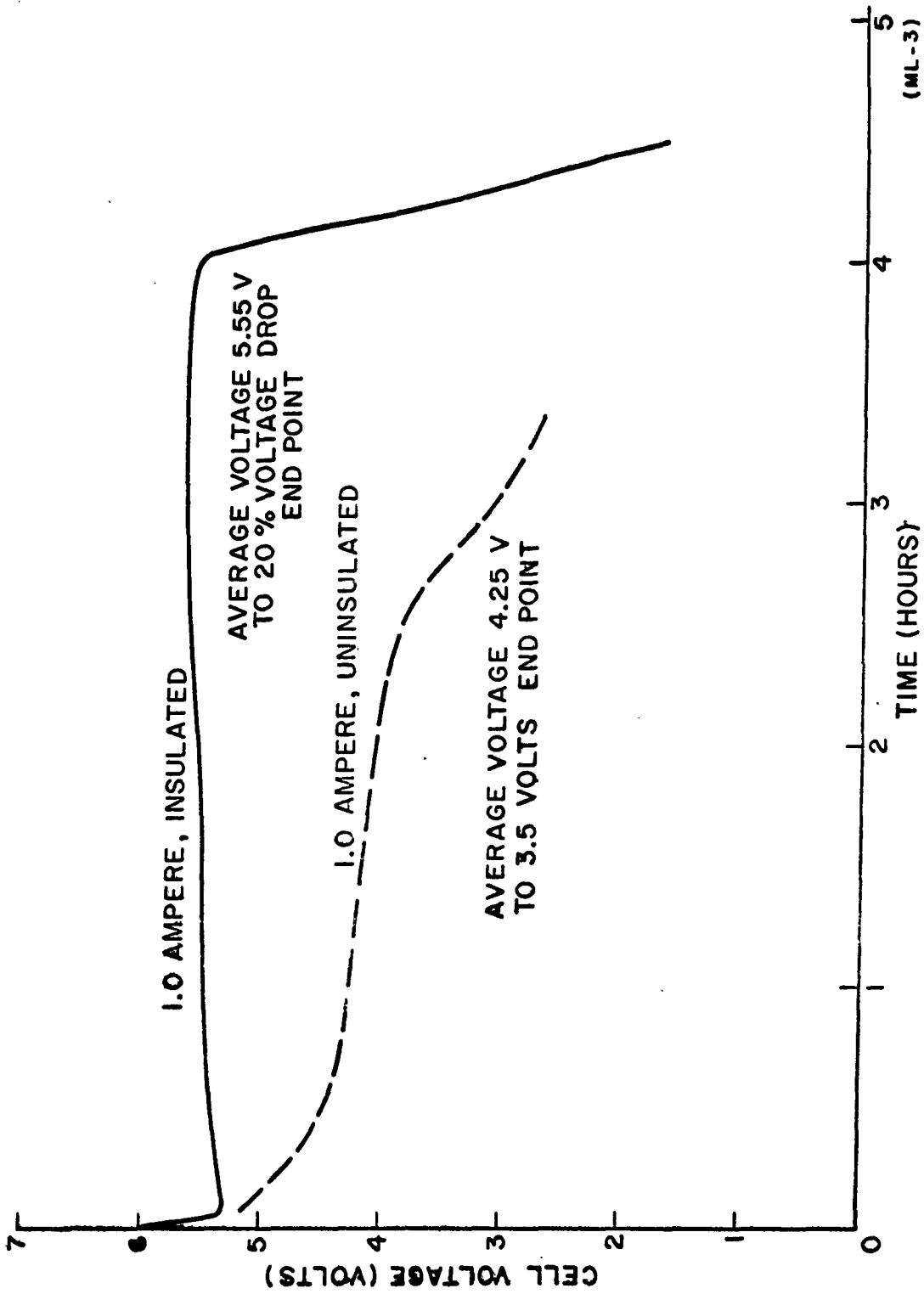


Figure 4.3. Capacity Data for a 3-Cell Mg/Hg(ClO₄)₂/HgO Reserve Battery at 1.0 Ampere at -40°F.

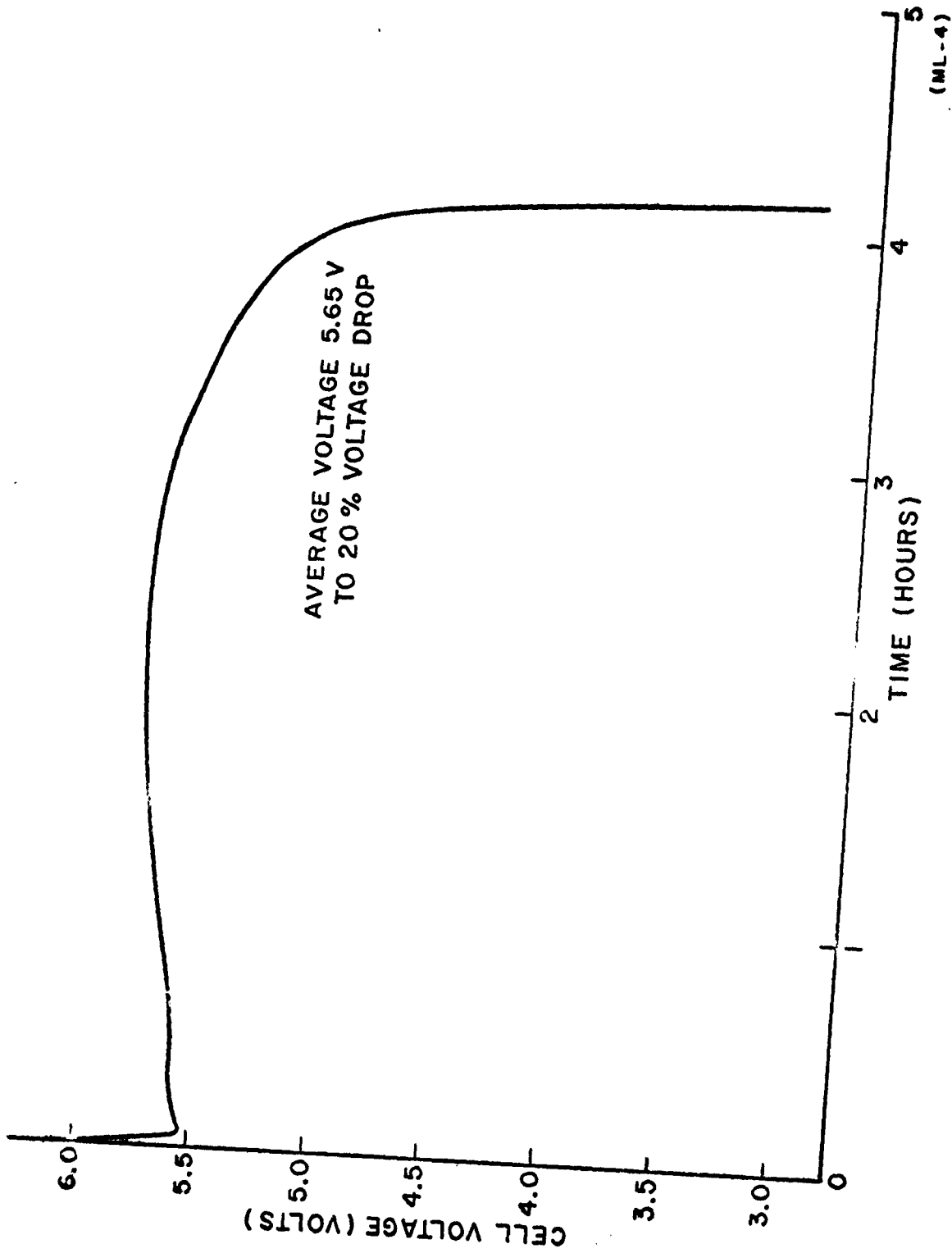


Figure 4-4. Capacity Data for a 3-Cell Mg/Mg(ClO₄)₂/HgO Reserve Battery at 1.0 Ampere at -58°F. Battery Contained in an Insulated Case.

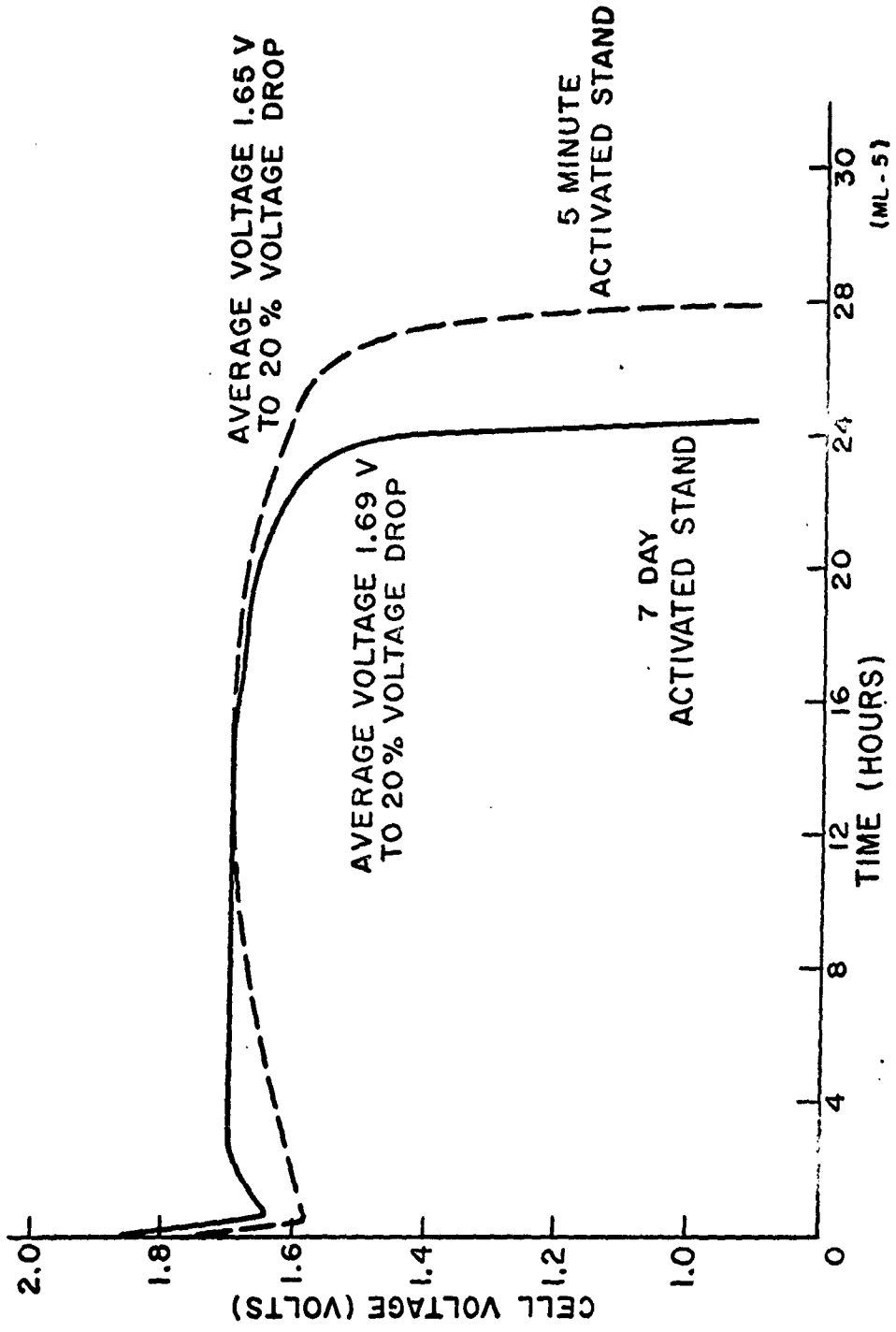


Figure 4-5. Capacity Data for Mg/Mg(ClO₄)₂/HgO Reserve Cells with a Titanium Cathode Grid at Various Activated Stand Times at 164 ma at 70°F.

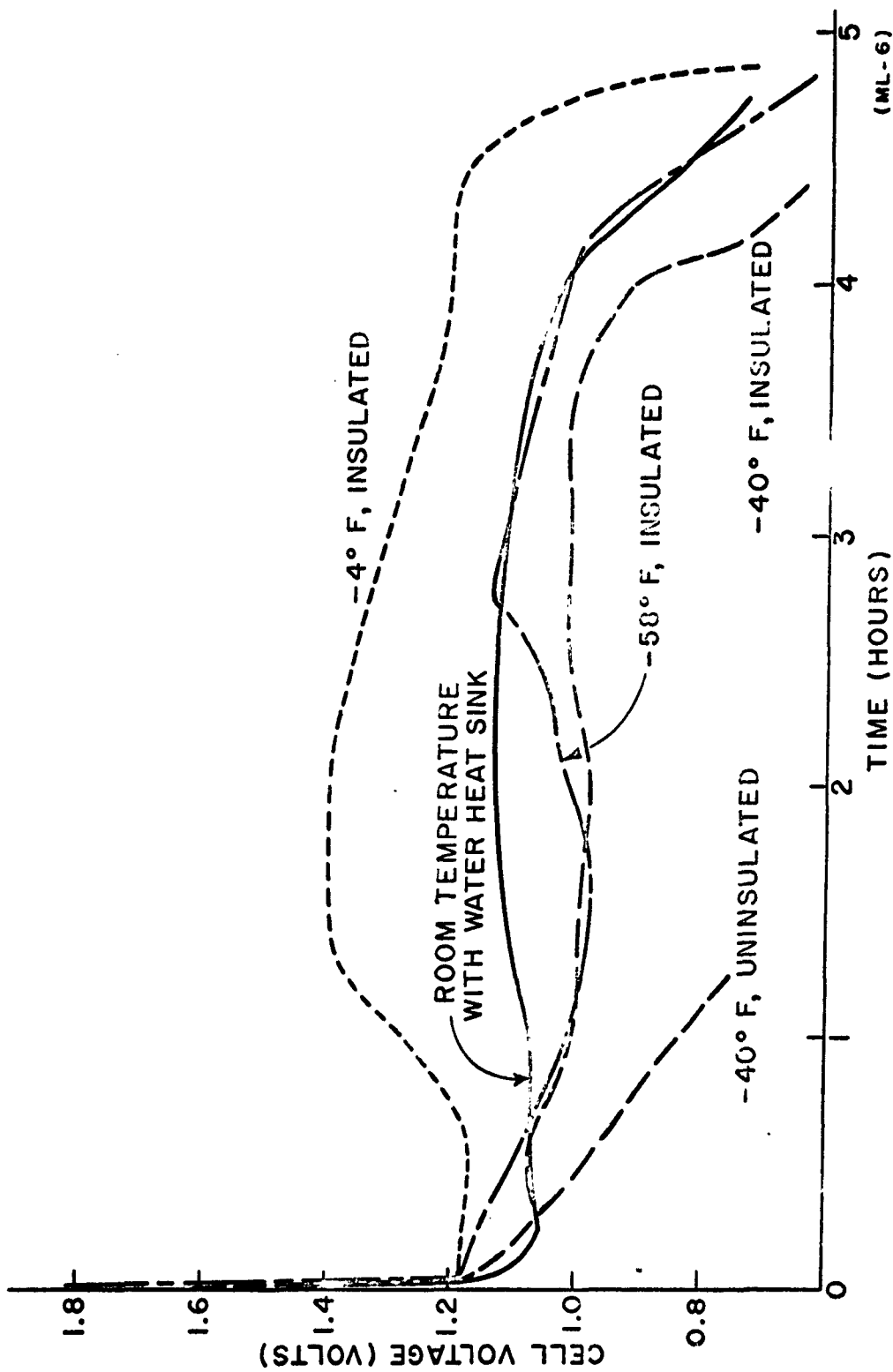


Figure 4-6. Effect of Temperature on the Discharge Characteristics of Mg/Mg(ClO₄)₂/CuO Cells Discharged at a Constant Current of 2.0 Amperes.

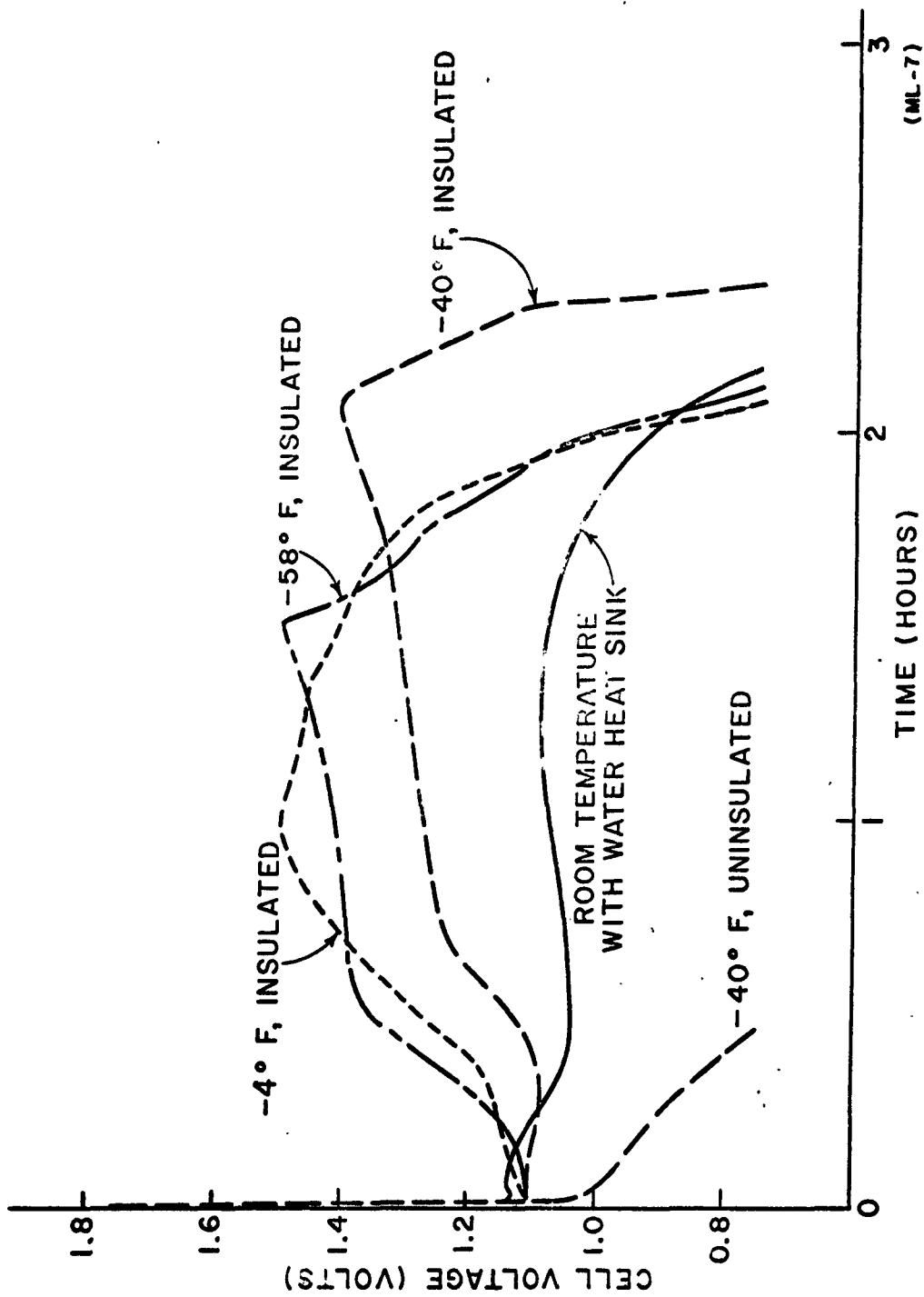


Figure 4-7. Effect of Temperature on the Discharge Characteristics of Mg/Mg(ClO₄)₂/CuO Reserve Cells Discharged at a Constant Current of 4.0 Amperes.

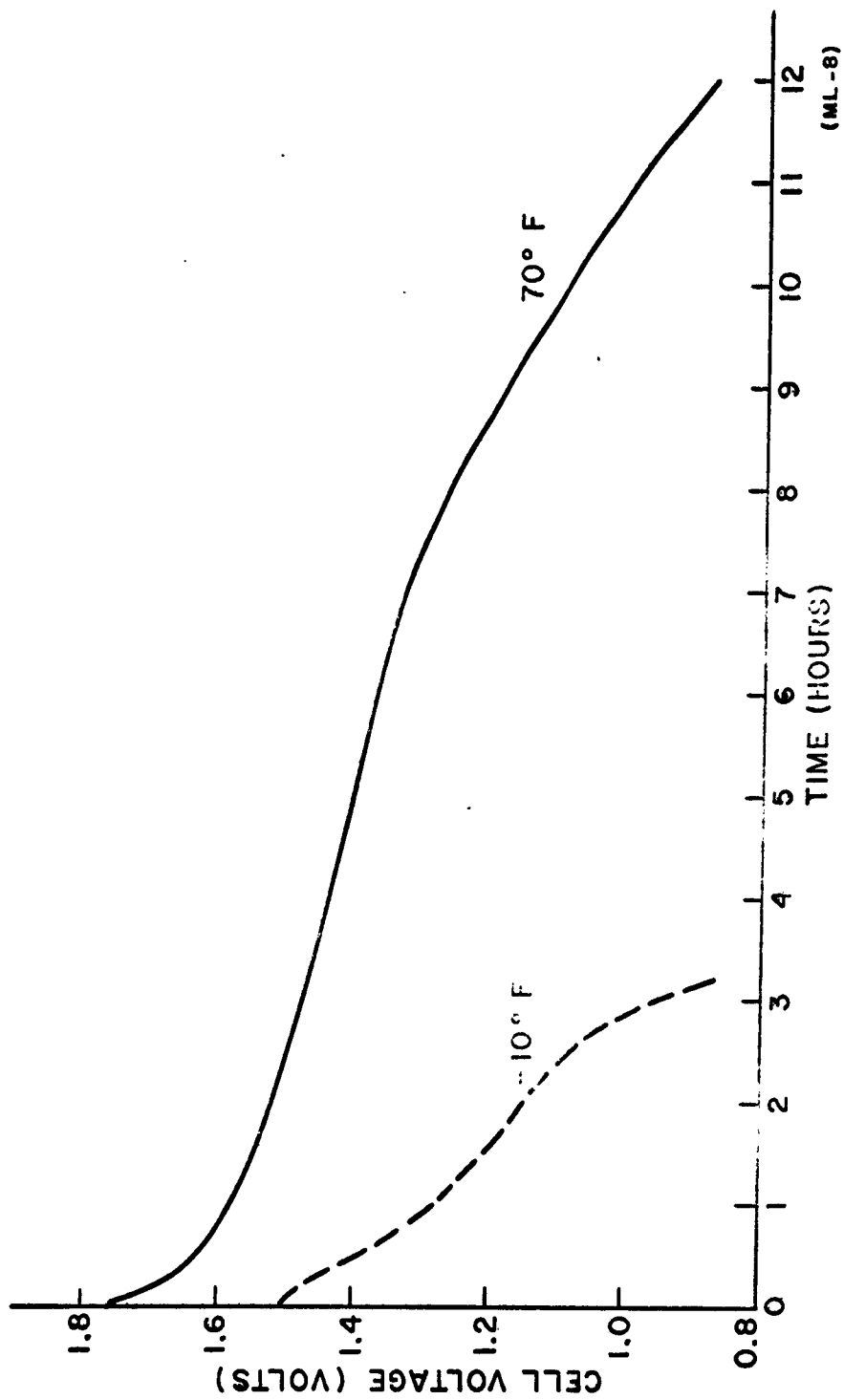


Figure 4-8. Capacity Data for Mg/Mg(ClO₄)₂/H₂O₂ A-Cells Discharged
 Continuous at Various Temperatures.

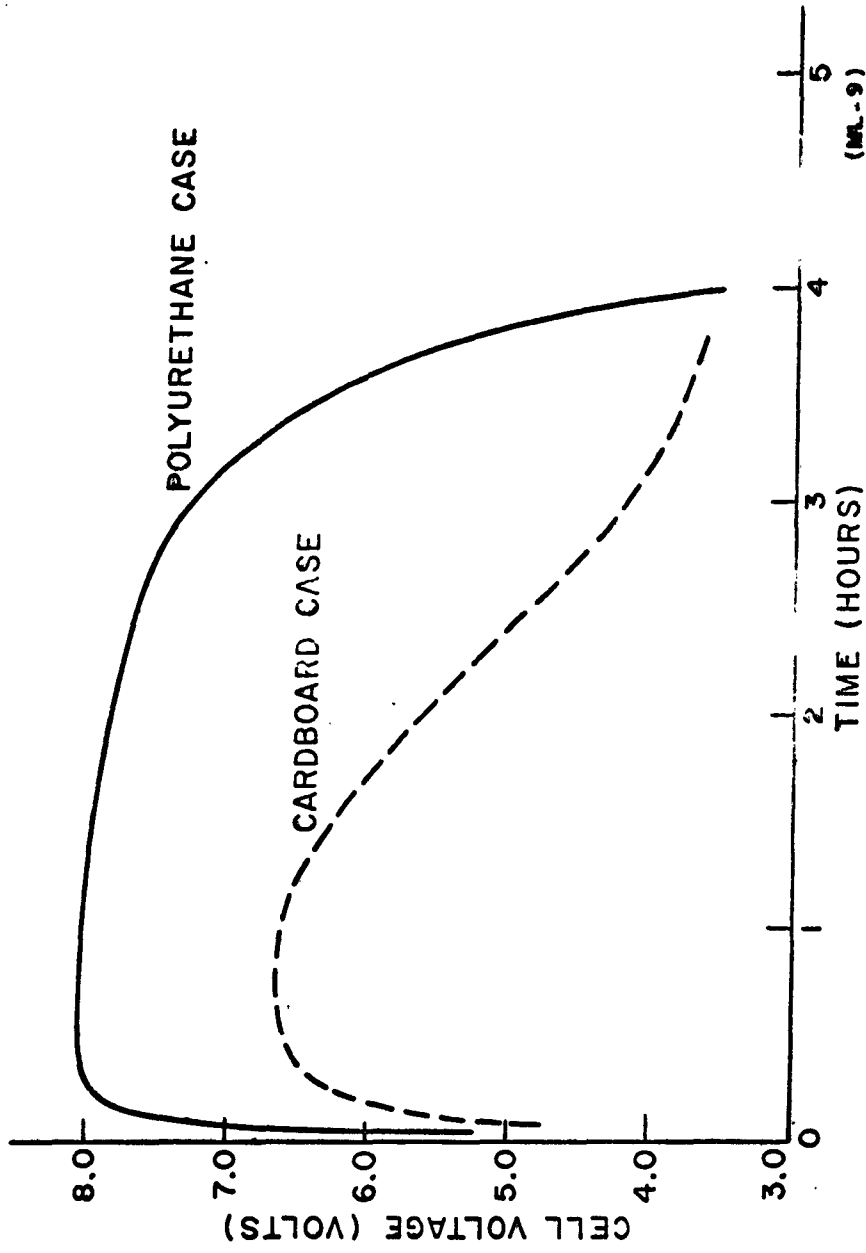


Figure 4-9. Capacity for Mg/Mg(ClO₄)₂/InO₂ Six A-Cell Batteries Discharged Continuously at 100 ohms at -20°F.

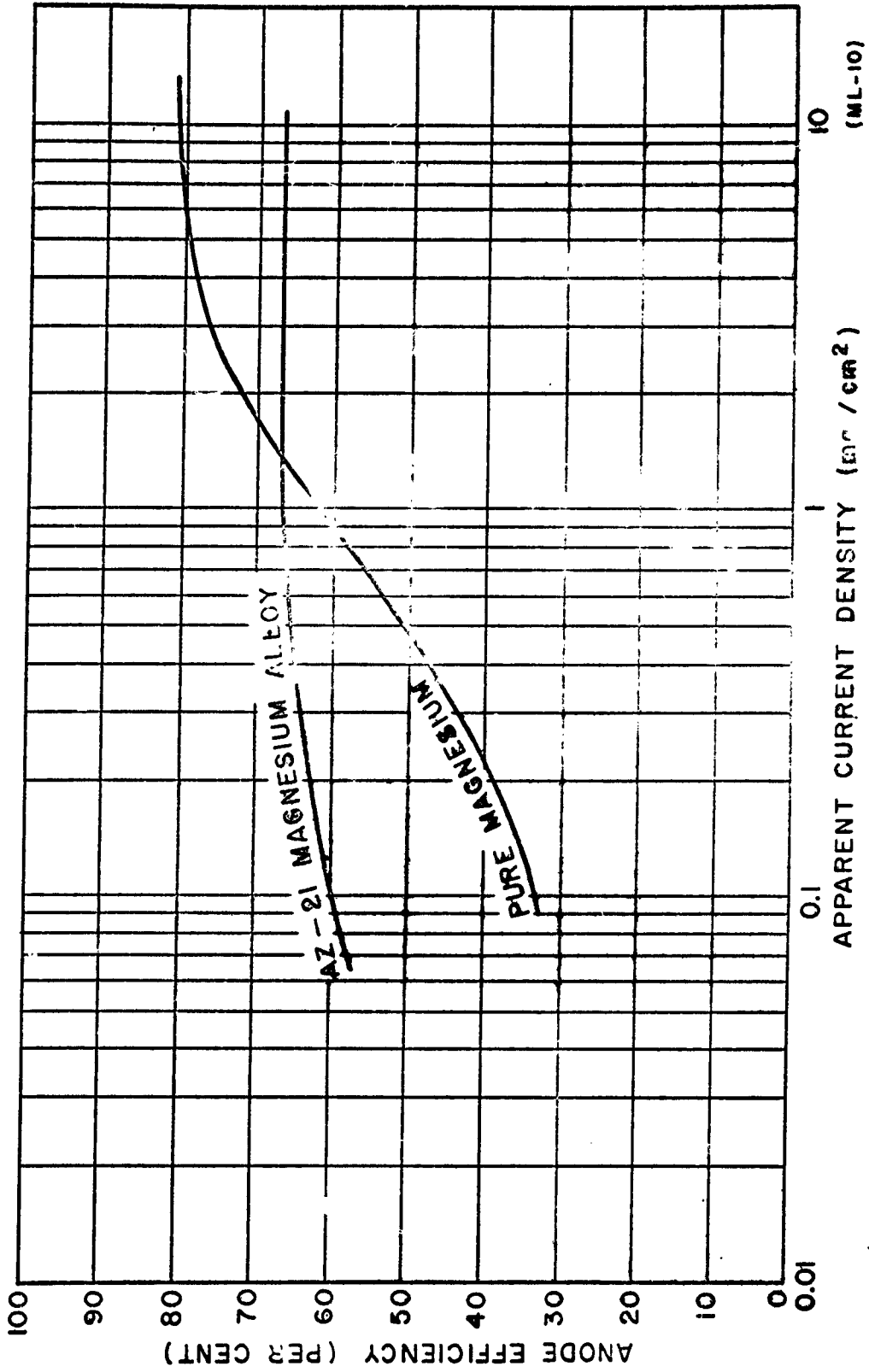


Figure 4-10. Magnesium Anode Efficiency as a Function of Current Density in 2% $(\text{ClO}_4)_2$ electrolyte.

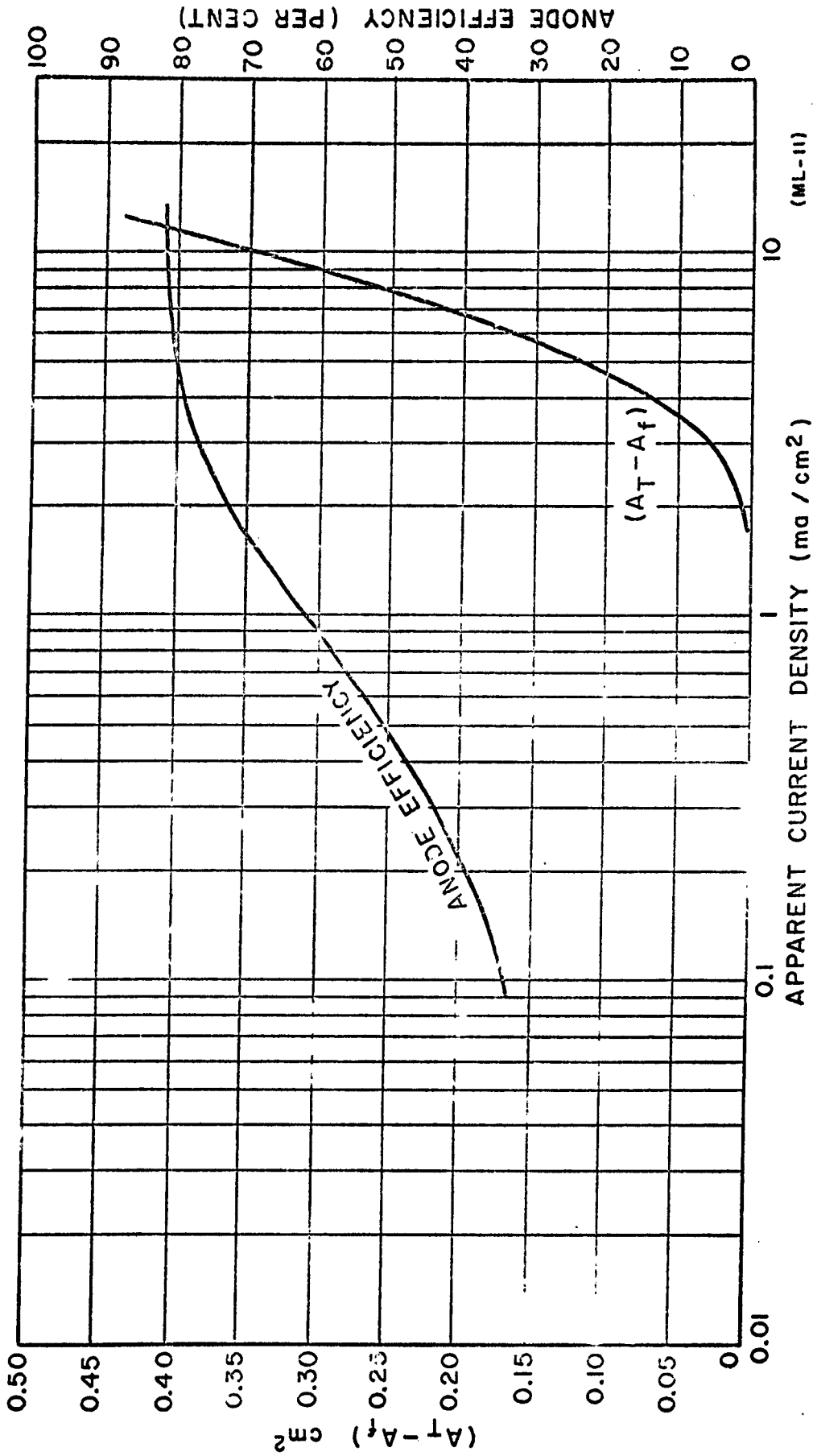


Fig. 4-11. Exposed Anode Area ($A_T - A_f$) And Anode Efficiency vs Apparent Current Density of Pure Magnesium in 2N $Mg(ClO_4)_2$ Electrolyte.

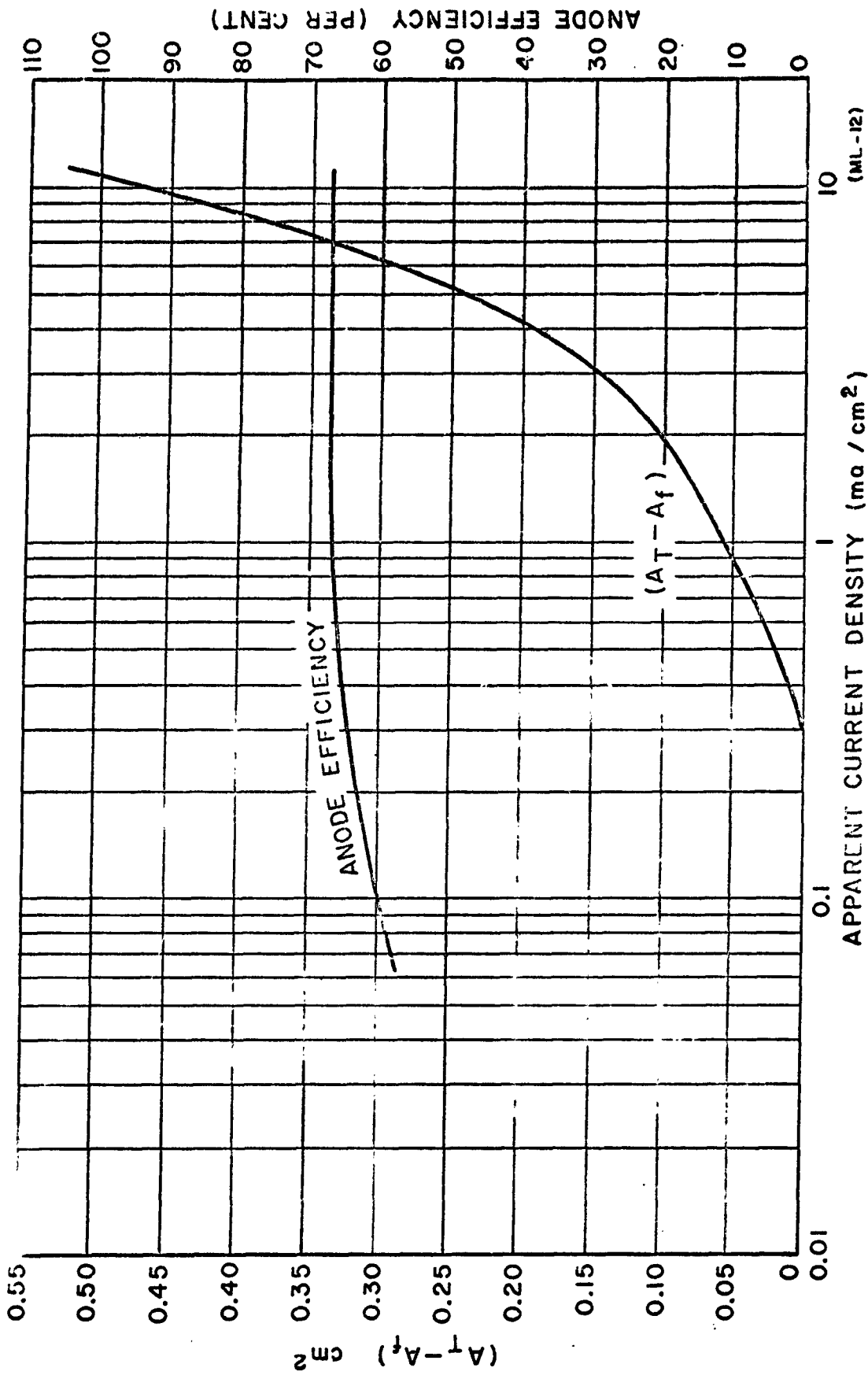


Figure 4-12. Exposed Anode Area ($A_T - A_f$) And Anode Efficiency vs Apparent Current Density (AZ-21 Magnesium Alloy) in $2N \text{ Mg}(\text{ClO}_4)_2$ Electrolyte.

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