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TECHNICAL RESEARCH REPORT
INVESTIGATION OF AgO PRIMARY BATTERIES

XEROX

SEVENTH QUARTERLY PROGRESS REPORT

REPORT NO. E-11-61

1 JANUARY 1961 to 31 MARCH 1961

SIGNAL CORPS CONTRACT NO. DA-36-039-SC-78319

PROJECT NO. 3G18-03-001

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



MISSILE BATTERY DIVISION
THE ELECTRIC STORAGE BATTERY COMPANY
RALEIGH, NORTH CAROLINA

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SEVENTH QUARTERLY PROGRESS REPORT

Report No. E-11-61

Covering the Period

1 January 1961 to 31 March 1961

dated

May 8, 1961

Missile Battery Division

of

The Electric Storage Battery Company

Signal Corps Contract No. DA-36-039-SC-78319

Project No. 3G18-03-001

Research and Development Work on a Non-Reserve

AgO Primary Cell

Signal Corps Technical Requirement No. SCL-6284

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I. PURPOSE

It is the purpose of this research and development contract to study nonreserve type primary cells utilizing the zinc-silver oxide electrochemical system. The investigation is primarily designed to lead to a better understanding of the basic factors governing the electrochemical performance of this system.

The work will be divided as follows:

Phase A - A determination will be made of cell components to produce optimum power and shelf life.

Phase B - An attempt to improve voltage regulation will be made by reducing or eliminating the initial AgO voltage plateau on low rate discharges.

Phase C - A cell container will be developed that will be leakproof, be light in weight, and contain an activated cell at 160°F.

Phase D - An investigation will be made to determine limiting factors on low temperature discharge and to develop methods of improving cell performance at low temperature.

Phase E - A design will be made in the cell container developed in Phase "C" in an attempt to optimize all variables and a series of tests will be made to determine the cell discharge characteristics and shelf life capabilities.

II. ABSTRACT

Non-reserve primary Zn/KOH/AgO cells were assembled and tested in an ESB type 5-15 size cell to evaluate positive electrodes, negative electrodes, and separator systems.

Using the most efficient positive and negative electrodes tested to date and the separator system giving the best 160°F. charged stand, sealed cells were assembled and tested in an ESB type S-7.5 cell container. New cells gave the following discharge results:

Discharge Rate (Hours)	Watt-Hours Per Pound	Watt-Hours Per in. ³
5	75	6.35
25	78	6.5
100	84	7.0

Experimental type 5-15 cells employing a variety of separator systems were stored at 160°F. for up to six weeks and then discharged at the 1.5 ampere continuous rate. With the best separator system tested, the capacity retention after six weeks storage at 160°F. was 50%.

III. CONFERENCES

On January 17, 1961, Messrs. A. M. Chreitzberg and G. M. Wylie of the Missile Battery Division, and Dr. Paul Ruetschi of the Carl F. Norberg Research Center visited the U. S. Army Signal Research and Development Laboratory, Fort Monmouth, New Jersey, to discuss the progress made during the sixth quarter of the contract and to outline work for the seventh quarter. Representing the Signal Corps were Messrs. C. Clark, J. Hovendon, C. Nordell, and C. Trigg.

On March 29, 1961, Messrs. J. Hovendon and C. Nordell of the U. S. Army Signal Research and Development Laboratory, and R. F. Amlie of the Carl F. Norberg Research Center visited the Missile Battery Division to discuss the progress made during the seventh quarter of the contract. Representing the Missile Battery Division were Messrs. L. C. Moore, A. M. Chreitzberg, G. S. Hartman and G. M. Wylie.

IV. FACTUAL DATA

PHASE A - EVALUATION OF CELL COMPONENTS

1. Evaluation of Positive and Negative Electrodes. - During the seventh quarter, positive and negative plates were manufactured by several techniques and charged on various schedules. The plates were discharged in a 5-15 type cell at the 1.5 ampere continuous rate in test electrolytes. The separator system was composed of one layer of 0.001 inch non-woven fibrous dynel mat, one layer of Polypor WA on 0.005 inch nylon, and two layers of 300 PUD-O cellophane (order listed is moving from positive plate to negative plate). Details of construction were given in Paragraph IV, B of the Fifth Quarter Report.

Table I lists the construction of the positive electrodes tested, the charging schedules used, test electrolyte, and the range of percentage efficiencies observed by discharging a sample of at least three cells containing the electrodes. Table II gives the results of similar tests performed on negative electrodes. Table III gives charging schedules used in charging both the negative and the positive electrodes. Electrodes tested earlier in the contract are included in these tables for comparison.

TABLE I
POSITIVE ELECTRODES

Positive Electrode (Number)	Construction	Charging Schedule Table III (Number)	Electrolyte	*Positive Efficiency (Percent)	Remarks
1	D.P. #1	* 1	40% KOH + ZnO	80-81	Possible choice for final design.
2	AgO + 2%	None	40% KOH + ZnO	80-81	Very fragile plate. Difficult to manufacture.
3	D.P. #1	2	40% KOH + ZnO 40% NaOH + ZnO	83-85 83-86	----- Voltage level and regulation unacceptable.
			40% KOH 30% KOH	83-86 83-86	----- -----
4	D.P. #1	3	40% KOH + ZnO	76-78	-----
5	Pressed Ag	1	40% KOH + ZnO	67-68	Density = 61 gm./cu. in
6	Pressed Ag	1	40% KOH + ZnO	75-77	Density = 78 gm./cu. in.
7	D.P. #1 + Conductive Matrix	1	40% KOH + ZnO	80-81	Possible choice for final design.
8	D.P. #1	7	40% KOH + ZnO	75-78	-----

*Output in A.H. to 1.1 volts X 100
0.4968 AH/gm Ag X weight Ag

TABLE II
NEGATIVE ELECTRODES

Negative Electrode (Number)	Construction	Charging Schedule Table III (Number)	Electrolyte	Negative Efficiency (Percent)	Remarks
1	Pressed Ray-O-Vac Material	None-Primary	40% KOH + ZnO	54-56	Very fragile plate. Difficult to manufacture.
2	Pressed Ray-O-Vac material + 1% Solka-Floc	None-Primary	40% KOH + ZnO	67	Very fragile plate. Difficult to manufacture.
3	Pressed ESB Mix C	4	40% KOH + ZnO	66-69	-----
			40% NaOH + ZnO	67-68	-----
			40% KOH	66-69	-----
			30% KOH	67-68	-----
4	D.P. Negative	5	40% KOH + ZnO	47-49	Difficult to Manufacture.
5	Pressed ESB Mix C	5	40% KOH + ZnO	60-63	-----
6	Pressed ESB Mix C	6	40% KOH + ZnO	66-70	-----
7	ESB Mix C + 1% Graphite	6	40% KOH + ZnO	62-74	-----
8	Plated Zn + HgO	None-Primary	40% KOH + ZnO	55	Difficult to manufacture. Fragile plate.
9	Pressed Merck Reagent Zn + HgO	None-Primary	40% KOH + ZnO	50-60	Fragile plate.
10	Pressed ESB Mix C	4	40% KOH + ZnO	74-76	These plates were half as thick as others tested.
11	Pressed ESB Mix C + 0.5 Solka-Floc	6	40% KOH + ZnO	73-78	Choice for final design.

TABLE III
CHARGING SCHEDULE

Schedule (No.)	Charging Rate ma/in ²	Input Capacity Theoretical Capacity %	Electrolyte
1	35	175	40% KOH
2	35 -35 35 -35 18 -18 18 -18 9 -9 4 -9	52 1/2 -2 1/2 52 1/2 -2 1/2 26 1/4 -1 1/4 26 1/4 -1 1/4 13 1/8 - 5/8 13 1/8 - 5/8 Total 175	40% KOH
3	35 to 2 volts vs Zn/ZnO 17 to 2 volts vs Zn/ZnO		40% KOH
4	32	200	5% KOH
5	155 77	100 50	5% KOH
6	155 77 39	100 50 25	5% KOH
7	Same as Schedule Number 1 except at 130°F.		

*3 approximately equally spaced one hour current reversals.

To date, positive electrode Number 3 is the most efficient tested; however, the charging schedule for this electrode is long and expensive, and it is felt electrodes Numbers 1 and 7 are the best choice for use in the final design. The final decision between these two electrodes will be delayed until tests are completed to compare their capacity loss rates during 160°F. storage.

Several attempts were made to test D.P. Number 2, but manufacturing problems resulted in the decision to terminate work on this electrode because the probable gain in efficiency was not large enough to justify the necessary work to solve the manufacturing problems.

To date, negative electrode Number 11 is the most efficient tested and will be used in the final design. It will be noted that the efficiency given for negative electrode Number 7 differs from that previously reported. After additional testing a large variation in efficiency was found from lot-to-lot of this electrode.

2. Evaluation of Separator Systems. - During the seventh quarter, 5-15 type cells employing a variety of separator systems were stored at 160°F. from two to six weeks. Table Number IV lists the separator systems tested and gives the results. Separator systems tested earlier in the contract are included for comparison.

It can be seen from Table IV that only cells containing both Synpor and cellophane were capable of standing reliably at 160°F., and that two layers of Synpor appear to give no better stand than one layer. It can also be seen that the addition of PMA 100-0 to a separator system of Synpor and cellophane does not appear to give better stand. It was found that separator systems containing Synpor did not need the dynel mat.

Figure 1 Shows a piece of Polypor WA on Saran after 10 days storage in a cell at 160°F.

TABLE IV
SEPARATOR SYSTEMS

Separator System (Listed from Positive to Negative)	Time at 160°F. (Days)	No. of Cells	Capacity	Retention
			Positive	Negative
1 layer .008" Synpor	14	3	75-77	70-83
2 layers 300 PUD-O Cellophane	29	3	50-53	50-56
2 layers .008" Synpor	3	9	Shorted	-----
2 layers .008" Synpor	14	3	69-71	53-70
2 layers 300 PUD-O Cellophane	28	3	52	60
	42	2	50	50
	28	1	Shorted	-----
1 layer .008" Synpor	10	9	Shorted	-----
1 layer 100-0 PMA				
1 layer .008" Synpor	14	3	60-68	65-68
2 layers 100-0 PMA	24	3	64-65	57
2 layers 300 PUD-O Cellophane	42	2	46-48	0-45*
	4	1	Shorted	
Polypor WA on Saran	8-10	2	Shorted	-----
2 layers 300 PUD-O Cellophane				
1 layer EM 309 Dynel	3-7	12	Shorted	-----
1 layer Polypor WA on .005" Nylon				
2 layers 300 PUD-O Cellophane				
1 layer EM 309 Dynel	6-8	3	Shorted	-----
1 layer 0.009" Porothene			78	
2 layers 300 PUD-O Cellophane				
1 layer 309 Dynel	7-10	3	Shorted	
1 layer 0.007" PMA 100-0	14	1		83
2 layers 300 PUD-O Cellophane				
1 layer EM 309 Dynel	14	3	77	81
1 layer 0.008" Synpor	29	3	52	58
2 layers 300 PUD-O Cellophane				

*One of these cells dried out on storage and the negative plates may have burned up.

PHASE C - LEAKPROOF CONSTRUCTION

Techniques already in use sealing uncharged cells were adapted to sealing charged cells during the seventh quarter. The method used to seal uncharged cells is to raise the electrolyte level above the top of the plates and cast an epoxy seal on top of the electrolyte. When this technique was tried on charged cells, it was found that the gas evolved from the plates formed bubble paths through the epoxy as it was setting up, resulting in a leaking cell. It was found that if the cell was allowed to stand for about two days, cell gassing decreased. It was also found that if the casting operation was delayed until just before the epoxy set up the length of time for bubbles to form in the epoxy is minimized. Using this technique, charged cells were successfully sealed. Figure 2 shows a curve of cell internal pressure vs time at various temperatures. The cell leaked pressure at 160°F. but did not leak electrolyte or form potassium carbonate at the leak. It was found that the leak was around the pressure gauge which was sealed into the cell. The cell used to obtain the pressure data is shown in Figure 3.

It was necessary to support the large sides of the cell during storage at temperatures above 104°F. to prevent the polystyrene cell case from rupturing.

PHASE D - LOW TEMPERATURE INVESTIGATION

The following additives were tried in negative plates in an attempt to irrigate the plates and retard their polarization:

1. One percent Ceylon Graphite
2. Two percent Ceylon Graphite
3. Three percent Ceylon Graphite
4. One-quarter percent Solka-Floc
5. One-half percent Solka-Floc
6. Three Quarters percent Solka-Floc

None of the additives tested improved the low temperature discharge performance.

Dirkse (1) reported that concentrations of KOH saturated with ZnO between 24% to 36% do not freeze at -40°F. Electrolyte concentrations from 26 to 34% KOH + ZnO have been tested and no significant variation in low temperature performance was noted.

At the present time the only way known to improve low temperature performance is to decrease plate thickness and increase plate area; however, this solution is inconsistent with the requirements for good charged stand characteristics and high watt-hours per pound.

PHASE E - CELL EVALUATION

A cell design was made including the best cell components tested to date. The Ag₂O positives employed in this test were positive electrode Number I (Table I) and the zinc negatives were negative electrode Number 11 (Table II).

The positive and negative plates measured 1 13/16 inches wide by 1 23/32 inches high (6.2) in²/plate, or 41 cm²/plate counting both surfaces). The positive plates were wrapped with one layer of 0.008 inch Synpor and two layers of 300 PUD-O cellophane (order listed is moving from positive plate to negative plate) by placing two positive plates end-to-end on the two separator sheets and rolling them together. This cell design contained four positive plates and five negative plates, with each of the two outside negative plates containing one-half (1/8 of the total) the quantity of negative mix contained in the inner negative plates. The cell design contained 48-52 grams of sintered silver (26 ampere hours theoretical capacity) and 44 to 46 grams of negative mix (30 ampere hours theoretical capacity). A volume of 15 to 16 cc of 40% KOH saturated with zinc oxide was added to bring the electrolyte level to about half way between the top of the plates and the top of the separators. The cell was then sealed by casting the top of the cell with epoxy. Figure 4 shows a completed cell.

Three cells of this design were discharged at each of the following rates: 5 hour rate, 13 hour rate and 60 hour rate. A family of discharge curves for these cells is shown in Figure 5. The weight of a completed cell of this design was 163 grams. A curve of energy densities vs discharge rate is given in Figure 6.

V. CONCLUSIONS

1. The most satisfactory separator system for 160°F. charged storage tested to date is:

1 layer 0.008" Synpor

2 layers 300 PUD-O Cellophane

2. Experimental multi-plate sealed cells in an ESB S-7.5 size container gave 19.6 ampere-hours at the five to 100 hour discharge rates. The energy output for these cells was 75-watt-hours per pound and 6.25 watt-hours per cubic inch at the five hour discharge rate to 84 watt-hours per pound and seven watt-hours per cubic inch at the 100 hour rate.

3. The capacity retention of cells employing the separator system from 1 above after four weeks storage at 160°F. was 52%.

VI. TENTATIVE PROGRAM FOR EIGHTH QUARTER

PHASE A

During the eighth quarter 160°F. storage test will be conducted with positive electrodes made from D.P. Number 1 with conductive matrix.

PHASE B

The positive plates for test cells will be treated in 40% KOH at 150°F. for varying length of time. It is believed this treatment will form a layer of Ag_2O on the surface of AgO in the positive electrodes and eliminate the initial voltage plateau with little loss in capacity.

PHASE C

A final selection of cell components will be made and three cells will be assembled and tested at the five hour and 30 hour rates at each of the following temperatures:

130°F.

113°F

70°F.

32°F

0°F

40°F.

Three cells will be subjected to Shock I and Vibration Test I requirements of MIL-B-18 and subsequently discharged at 70°F. at the five hour rate.

VII. DISTRIBUTION OF HOURS DURING SEVENTH QUARTER

NAME	TITLE	HOURS
A. M. Chreitzberg	Manager, Advanced Design	21
G. M. Wylie	Project Engineer	157
A. Alexander	Engineering Technician	<u>308.5</u>
		Total 486.5
		Hours

VIII. REFERENCES

1. T. P. Dirkse, J. ElectroChem. Soc., 106, 154 (1959)

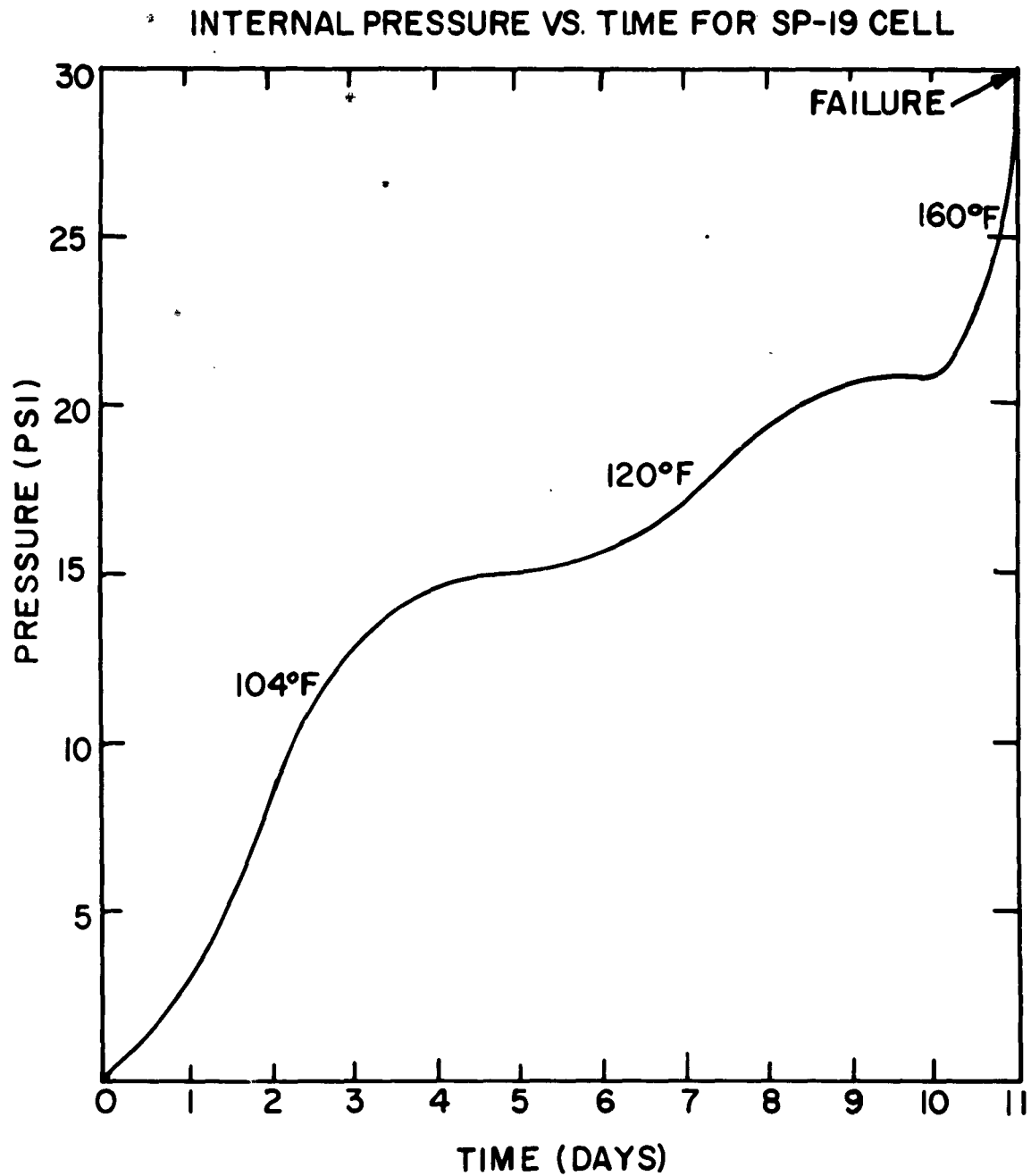


Figure 2

SP-19 CELL USED TO
OBTAIN PRESSURE DATA

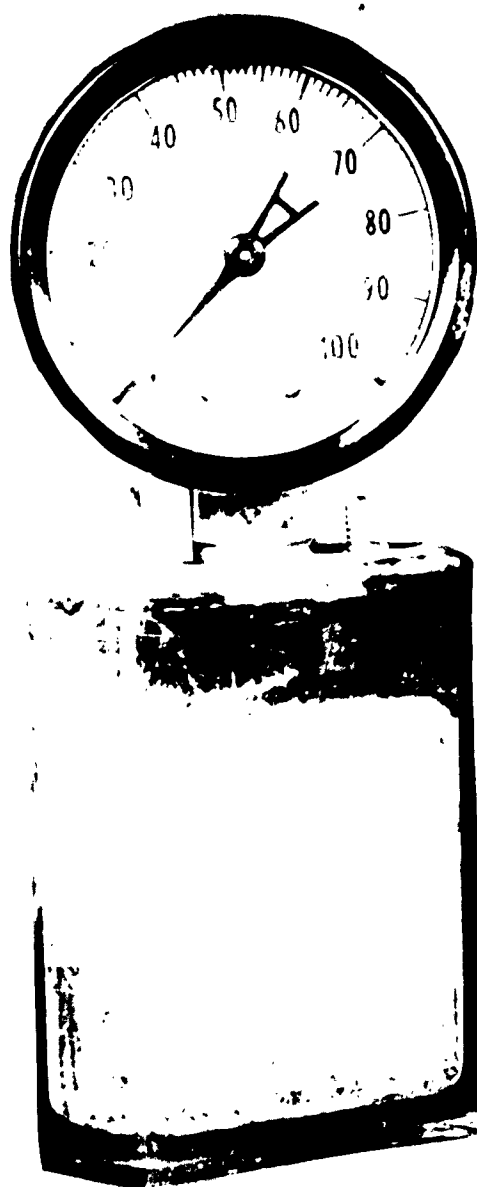


FIGURE 3

SP-19 CELL

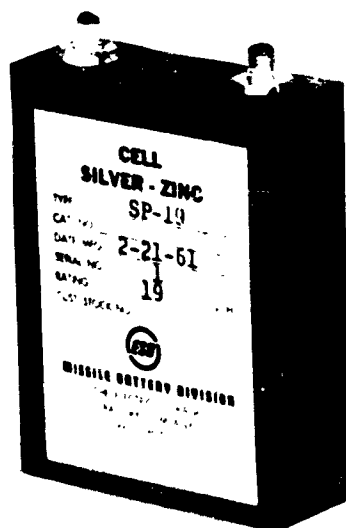


FIGURE 4

TYPICAL DISCHARGE CURVES
FOR SP-19 CELL

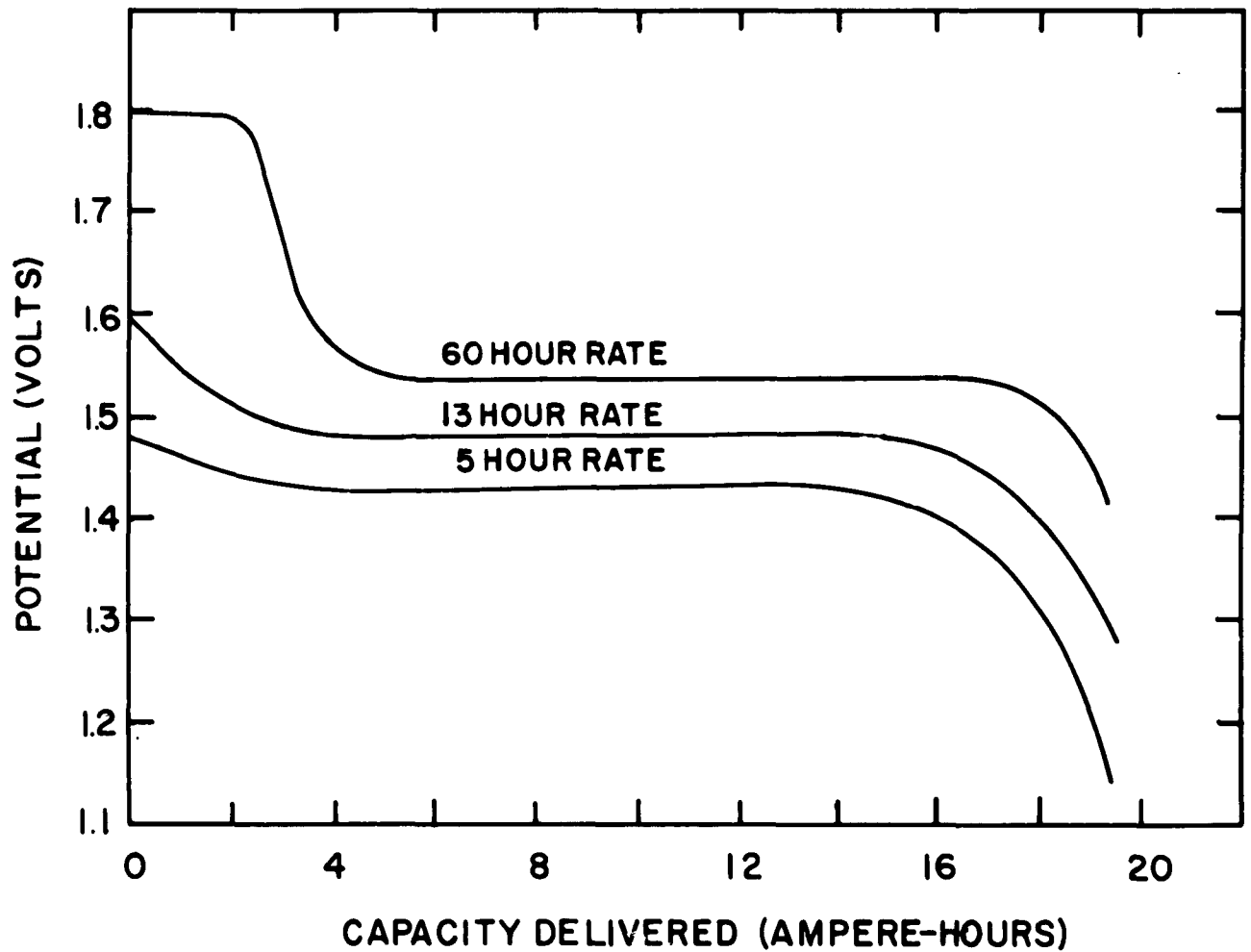


Figure 5

ENERGY DENSITY VS. DISCHARGE
RATE FOR SP-19 CELL

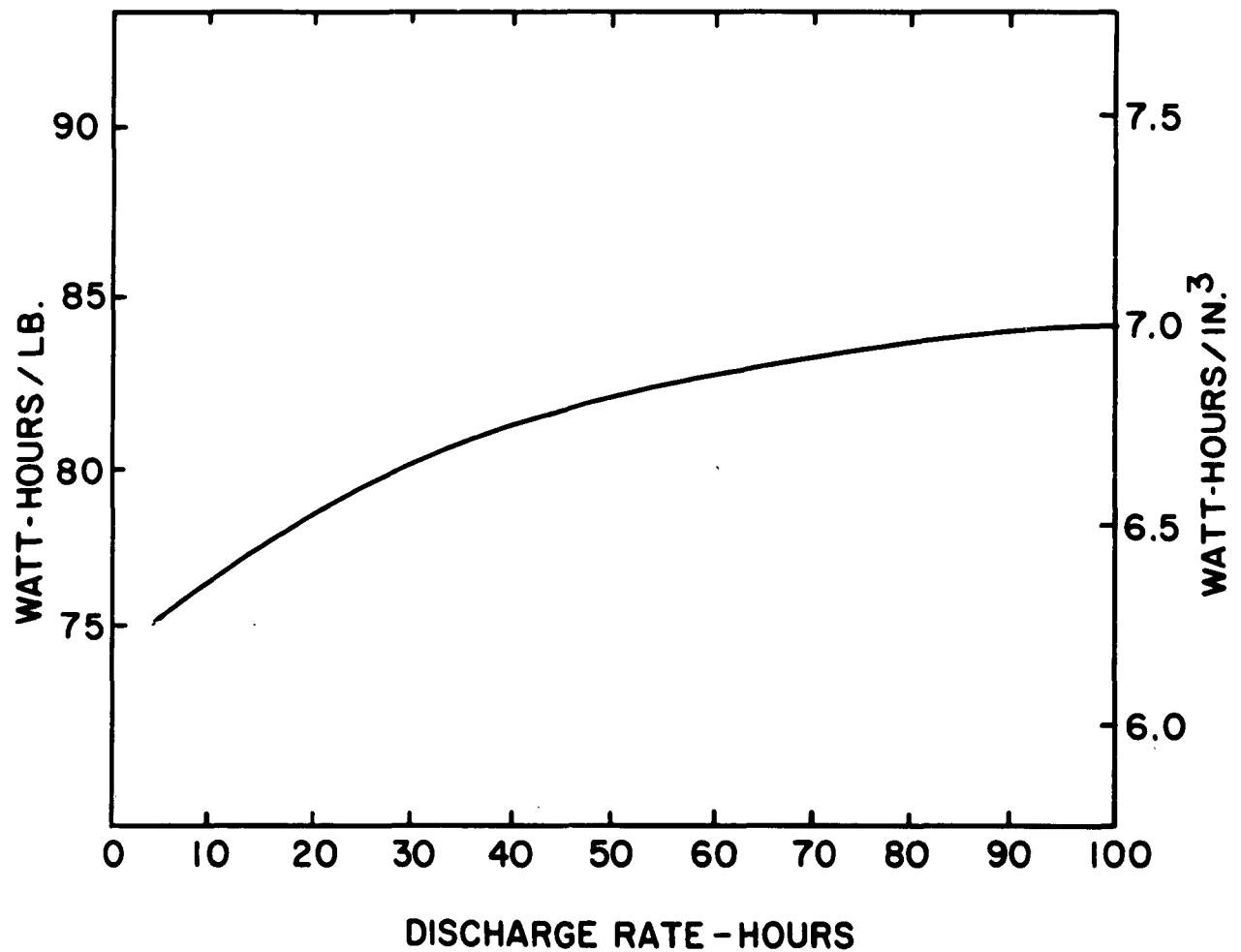


FIGURE 6

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DA Proj. No. 3018-03-001, Unclassified Report

Experimental Zn/KOH/AgO sealed flat plate cells delivered 75 watt-hours per pound and 6.25 watt-hours per cubic inch at the five hour rate to 84 watt-hours per pound and 7 watt-hours per cubic inch at the 100 hour rate. Experimental unsealed cells retained 46 to 50% of their new capacity after six weeks storage at 160°F. No improvement in cell performance was obtained at -30°F.

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