NASA CR-72416

TOPICAL REPORT

DEVELOPMENT OF HIGH ENERGY DENSITY PRIMARY BATTERIES

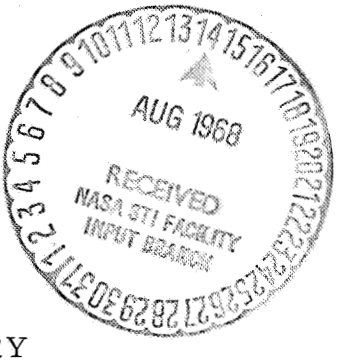
BY

S. G. ABENS

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-10613



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DEVELOPMENT OF HIGH ENERGY DENSITY PRIMARY BATTERIES

by

S. G. Abens

ABSTRACT

Studies of discharge properties of thin-plate CuF_2-Li cells with MF-$LiClO_4$ electrolyte were undertaken. Seven-plate, 4 - 5 AH (theoretical) cells were discharged at -5°C and +35°C at five rates giving current densities in the range of 1.2 to 34.5 mA/cm^2. At -5°C, 63.3% CuF_2 reduction efficiency and 106 wh/lb of net cell was obtained at 8.6 mA/cm^2 (ca. 3-hour rate); at 35°C, the corresponding figures were 61.9% and 106 wh/lb at 3.0 mA/cm^2 (ca. 8-hour rate).

TABLE OF CONTENTS

1. INTRODUCTION

This report describes construction and testing of 30 experimental CuF_2-Li cells using methyl formate (MF)-$LiClO_4$ electrolyte.

The methyl formate electrolyte system was studied in CuF_2-Li cells at this laboratory in two previous high energy density battery development programs under contracts to NASA Lewis Research Center (NAS 3-6004[1] and NAS 3-7632[2]). This work showed that relatively high discharge rates (1 hour and higher) were possible with the MF electrolyte with good CuF_2 electrode utilization (60%). The wet shelf life properties of these cells were not studied, but stability tests indicated some reactivity between the electrolyte and the lithium negative electrode material.

The purpose of the tests described in this report was to establish the performance capability of the system as developed under the above contracts. The present work was performed for NASA Lewis Research Center as Task I. A. of contract NAS 3-10613.

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[1] Abens, S. G., et. al; Livingston Electronic Corporation, "Development of High Energy Density Batteries, 200 Watt Hours per Pound of Total Battery Weight Minimum", NAS 3-6004, Final Report, NASA CR-54803.

[2] Abens, S. G., et. al; HONEYWELL INC., Livingston Electronic Laboratory, "Development of High Energy Density Primary Batteries", NAS 3-7632, Final Report, NASA CR-72331.

2. MATERIALS AND EQUIPMENT

2. 1. Cell Components

2. 1. 1. Positive Electrodes

The following materials were employed for constructing the pasted positive plates:

CuF_2, Lot A - Ozark-Mahoning Co., 53% Lot KW4-12B, 47% Lot KW3-44, 1.3% H_2O.

CuF_2, Lot B - Ozark-Mahoning Co., Lot KW4-105, 0.3% H_2O.

$CuF_2 \cdot 2H_2O$ - Ozark-Mahoning Co., Lot KW3-48, 98% purity.

Powder diffraction charts for the above materials are shown in Figures 1 to 3, pages 3 - 5.

Graphite - Dixon Airspun.

Expanded Silver - Exmet Corp., "high purity" silver, mesh designation 1/0 - 5Ag14.

Pasting Solution - 2% cellulose acetate (Eastman Organic Chemicals) dissolved in a 9:1 volume mixture of ethyl acetate (J. T. Baker, Reagent Grade) and ethanol (Publicker Industries, U.S.P. 200 proof).

2. 1. 2. Negative Electrodes

The solid lithium electrodes were prepared from the following materials:

Lithium - Foote Mineral Co., 2 x 0.015 in. ribbon (supplied packed in argon). Typical assay[3]:

Li	99.97 %
Na	0.0069 %
K	0.0056 %

[3] Manufacturer's data (June 10, 1967).

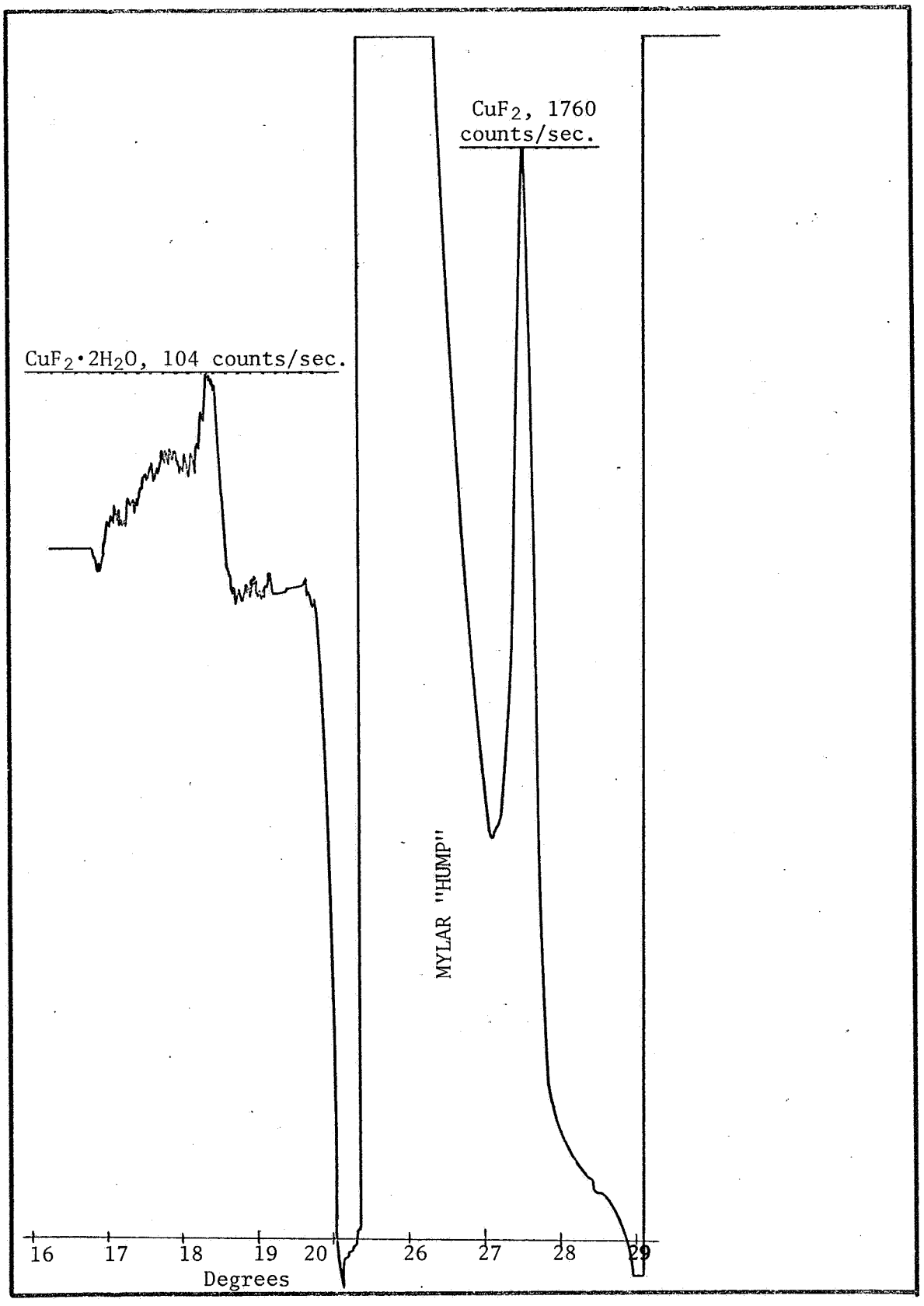

FIGURE 1: DIFFRACTION PATTERN OF CuF_2 USED IN MIX A (5.1% $CuF_2 \cdot 2H_2O$)

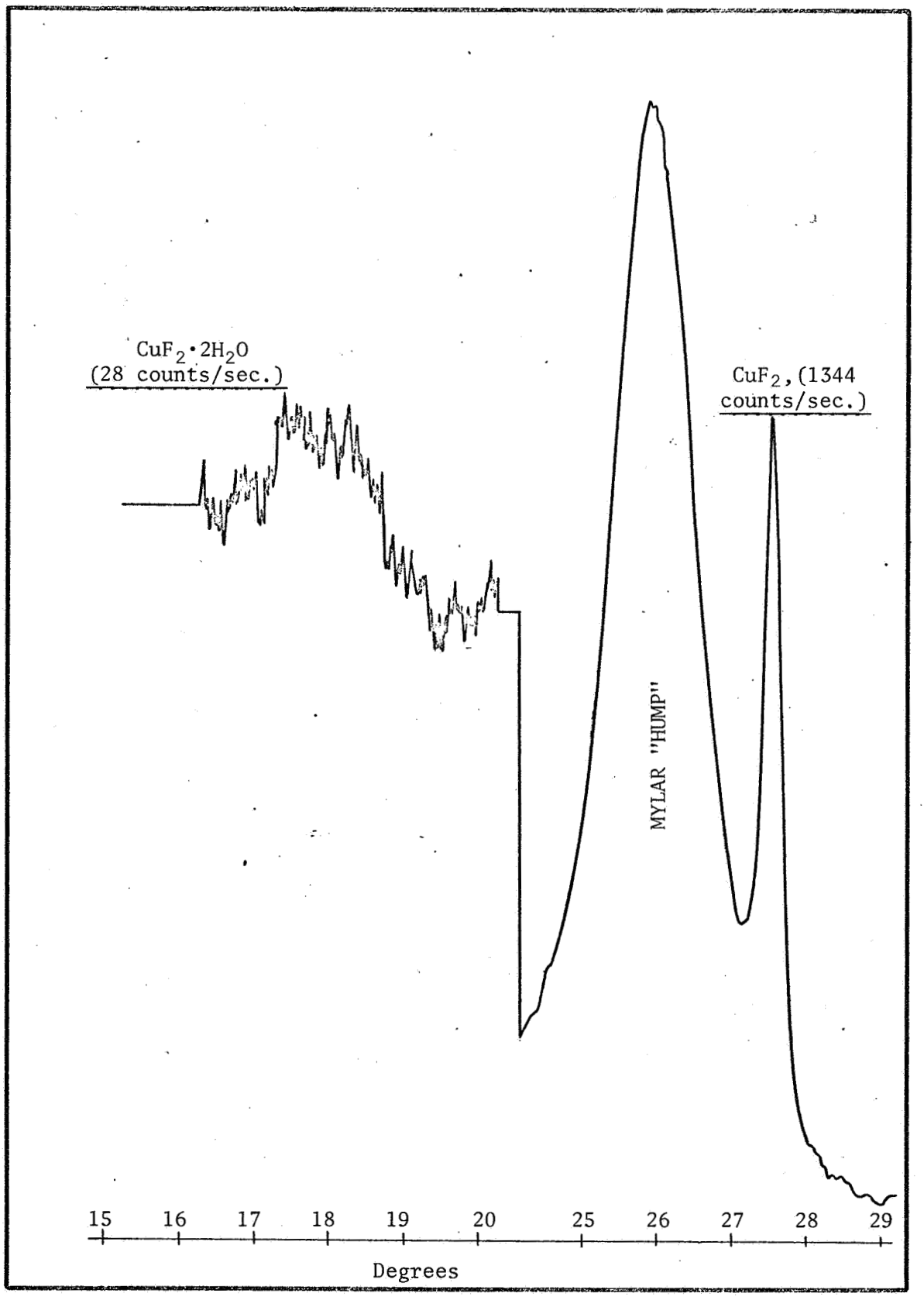

FIGURE 2: DIFFRACTION PATTERN OF CuF_2 USED IN MIX B (1% $CuF_2 \cdot 2H_2O$)

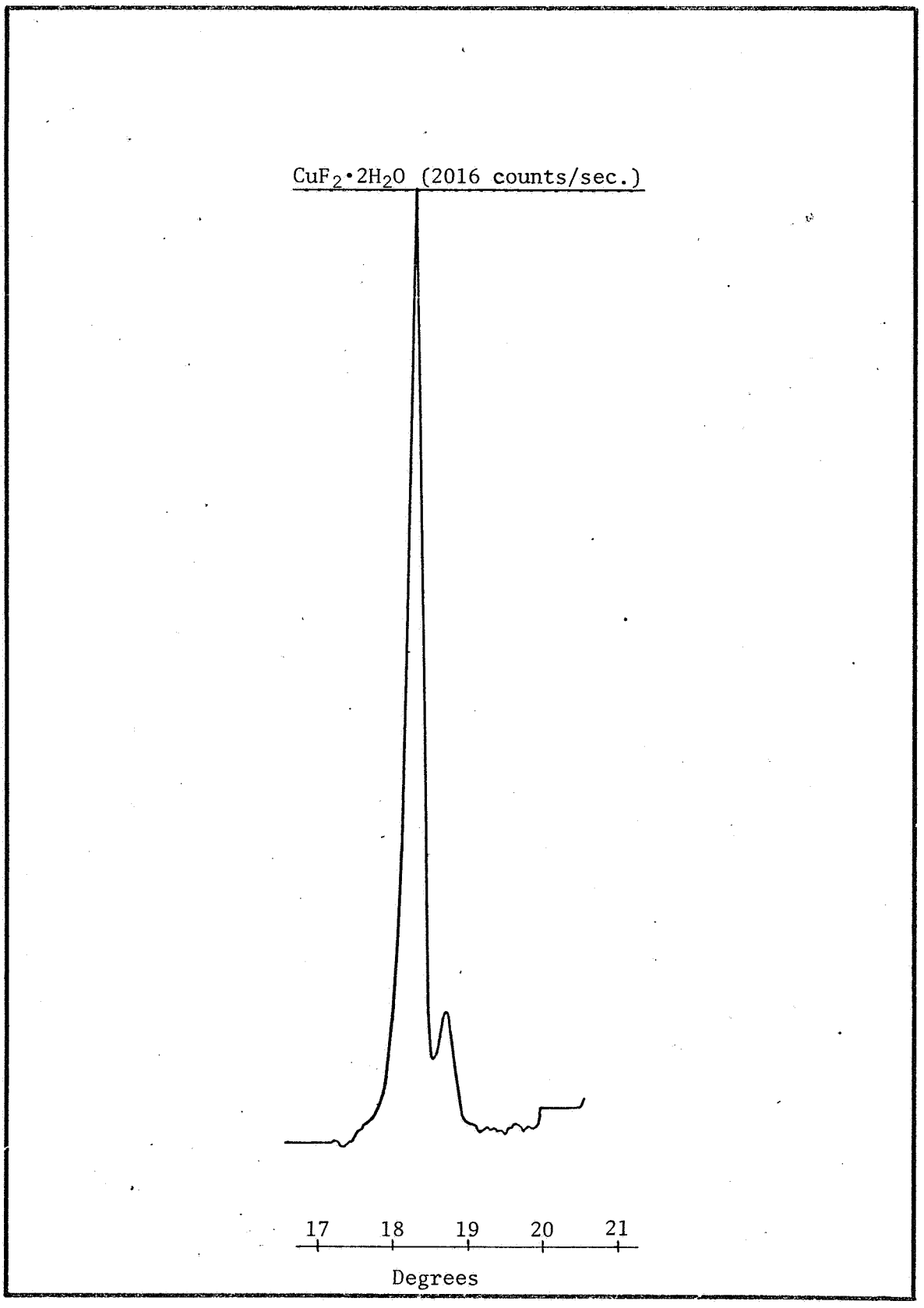

FIGURE 3: DIFFRACTION PATTERN OF $CuF_2 \cdot 2H_2O$

Expanded Silver - same as used for positive electrodes.

2. 1. 3. Electrolyte

The electrolyte used in all cells was 50 grams $LiClO_4$ in 100 ml of methyl formate (MF). The following materials were employed:

$LiClO_4$ - G. M. Smith, Grade Reagent, Lot B-7, Vacuum dried 96 hours at 110°C.

Methyl Formate - Matheson, Coleman & Bell, Spectroquality grade, Lot 13.

Container A - 760 ppm H_2O

Container B - 560 ppm H_2O.

2. 1. 4. Separation

In all cells, two layers of Reeve Angel 934-AH glass mat was used; this gave a separation thickness of 0.020 in.

2. 2. Cell Container

The elements were contained in 2 in. wide polyethylene envelopes (0.004 in. sheet thickness). The cells were placed singly in hermetically sealed glass tubes as shown in Figure 4, page 7.

2. 3. Discharge Equipment

The 4.0 ampere discharges were conducted with a NJE Corp., Model EG-22RM power supply and lamp bank in series with the cells. The remaining discharges were performed with a Hewlett-Packard Model 6200B constant current power supply in series with the cells and the required load resistance. Cell potentials were recorded on a Brown "Elektronik" recorder having a 60 second record cycle for each cell and a precision of 50 mV.

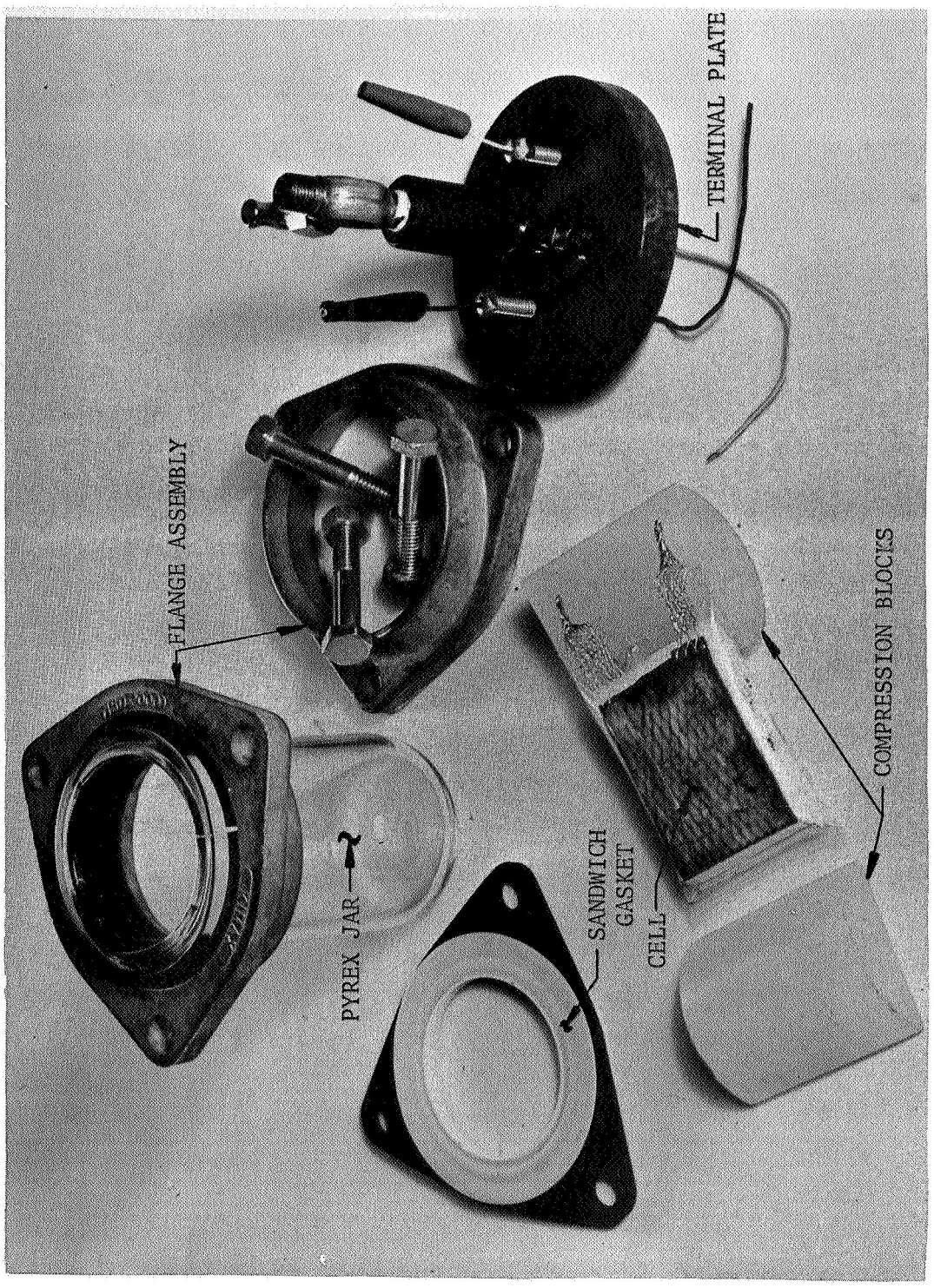

FIGURE 4: EXPLODED VIEW OF CELL DISCHARGE ASSEMBLY

3. PROCEDURE

3. 1. Construction of Cell Components

3. 1. 1. Positive Electrodes

In an argon glove box, the following ingredients were weighed into a Hi-Speed[4] blender:

Mix A:	CuF_2, 53% Lot KW4-12B, 47% Lot KW3-44 (Lot A)	41.8 g
	$CuF_2 \cdot 2H_2O$	2.2*
	Graphite	4.4
Mix B:	CuF_2, Lot KW4-105, (Lot B)	40.0 g
	$CuF_2 \cdot 2H_2O$	4.0*
	Graphite	4.4

*Weight adjusted to give 5% H_2O in both mixes.

The dry mix was blended (with blender baffle in position) for 30 seconds and was transferred to a small beaker. Twenty-four ml of 2% cellulose acetate pasting solution were added, and a workable stiff paste was formed by mixing with a steel spatula (additional solvent was added as required to maintain the desired paste consistency). The paste was immediately applied to the silver support in a 2.0 x 1.5 x 0.03 in. cavity. The resulting electrodes were dried and stored under vacuum.

3. 1. 2. Negative Electrodes

The ribbon was cut to 2 x 1.5 in. strips and placed on both sides of the expanded silver between polyethylene sheets; this assembly was compressed at 1100 psi between steel platens and the electrodes were stored under argon between the polyethylene sheets.

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[4] Hi-Speed Mixing and Blending Co., Hillside, New Jersey.

3. 2. Construction of Cells

In the argon glove box, the lithium electrodes were removed from the polyethylene sheets and trimmed to size if necessary[5]. Seven-plate, outside-negative elements were then immediately assembled using 1/4 in. oversize separators (2-1/4 x 1-3/4 in). The elements were heat-sealed into 2 in. wide polyethylene envelopes (0.004 in. thick) and assembled into the discharge chambers between polyethylene retainer blocks as shown in Figure 5, page 10.

The weights of the cell components were as follows:

Lithium	3.2 grams
CuF_2 electrodes (excluding grids)	$9.5\ ^{+1.2}_{-2.1}$
Silver grids	3.2
Glass mat separation	2.0
Total	$17.9\ ^{+1.2}_{-2.1}$ grams.

The polyethylene envelope weighed 1.0g, and the 12.0 ml of electrolyte added 15.0g for a total activated cell weight of $33.9\ ^{+1.2}_{-2.1}$ g.

The geometric discharge area for these cells was calculated to be 18 sq. in. (116 sq. cm).

3. 3. Preparation of Electrolyte

The solvent was cooled to -40°, transferred to the argon glove box, and added to $LiClO_4$ to give a concentration of 50g of salt/100 ml of solvent. All subsequent handling of the electrolyte was done thru serum stoppers with hypodermic syringes to avoid contamination by atmospheric moisture. The water content of the electrolyte solutions was:

[5] A slight increase in dimensions may occur during pressing; the lithium trimmed off was no more than 5% of the total lithium weight.

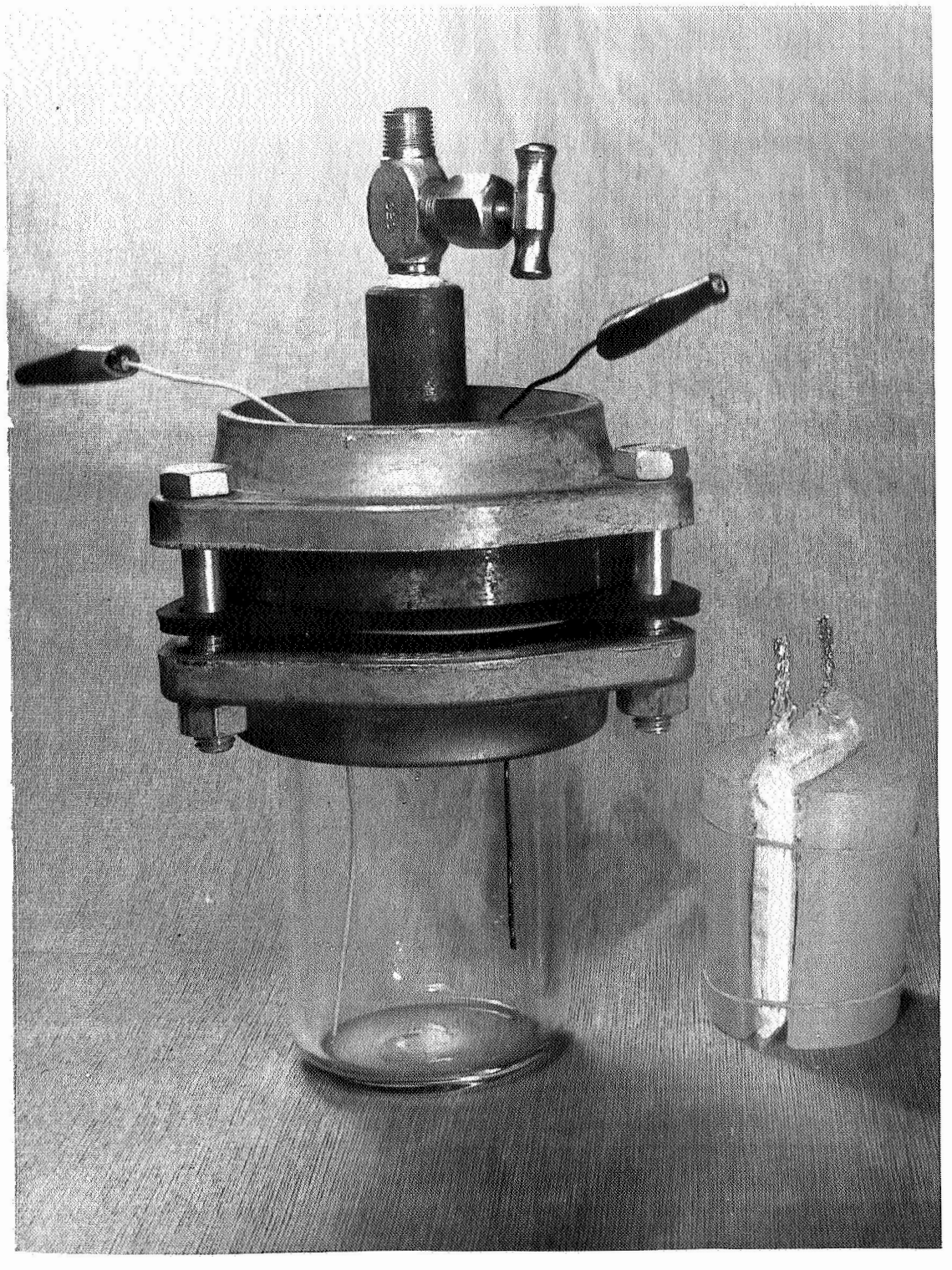

FIGURE 5: CELL DISCHARGE ASSEMBLY

Batch A: 1000 ppm

Batch B: 600 ppm.

The electrolyte was stored at room temperature until required for test.

3. 4. Activation and Discharge

Prior to activation, the cells were allowed to stand in the sealed test chambers for at least two hours at the test temperature. The electrolyte was likewise allowed to equilibrate at this temperature for the same period of time. Activation was accomplished by rapidly adding 12 ml of the electrolyte via a hypodermic needle to each cell; the total activation time for a group of three cells was about 5 minutes. A mercury manometer was attached to every third cell container, and the cells were given a wet stand of 15 minutes before constant current discharge was commenced.

Three cells were tested simultaneously at each discharge rate recording cell potential, current, and chamber pressure for the cell equipped with a manometer. After all cells had fallen below the selected cut-off voltage (2.0V), discharge was terminated and the cells were opened for visual inspection.

4. RESULTS AND DISCUSSION

Results of the discharge tests are shown in Table I, page 13, and the voltage-time data for the best cell in each group have been plotted in Figures 6 to 11, pages 15 - 20. The effect of discharge rate and temperature on the "net"[6] wh/lb obtained from these cells can be seen from the plot of energy/weight ratio vs. discharge rate shown in Figure 8.

The best performance at -5°C (about 100 wh/lb) is obtained at about the 3-hour rate (8.6 mA/cm^2), but either increasing or decreasing the current causes a sharp drop in energy output. At the 35°C discharge temperature, the best performance is likewise approached at the 3-hour rate. However, while the performance expectedly drops off at higher discharge rates, lowering the current to the 20-hour rate does not significantly alter the energy output at this temperature. This output trend seems anomalous, since one would expect the decrease in current to be more beneficial to the low temperature cells than to those at the higher temperature[7].

An explanation might be attempted in terms of some abrupt physical change (such as precipitation of the solute at the anode surface) at the lower temperature, which is eliminated by the temperature rise from I^2R heating inside the cell at the higher currents. This phenomenon should be researched in future work with electrode polarization studies at the temperatures and current densities of interest.

The highest container pressure observed during discharge was 10 psig at 2.5A and 35°C. For all other discharges, the pressure remained below this value.

The scope of this test did not allow comparison of the two lots of CuF_2 and electrolyte used. However, the data does not suggest any major effect of these variables on cell performance.

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[6] Based on the weights of electrodes, electrolyte, separation and polyethylene envelope.

[7] In commercial batteries, increasing the discharge rate or decreasing the temperature generally have similar effect on discharge performance, i.e., the voltage and energy output are reduced in both cases.

TABLE I

DISCHARGE PERFORMANCE OF CuF_2-Li CELLS

Cell No.	Pos. Mix	Elec'yte Batch	Theo. Cath. AH	Temp., °C	Current, A	(mA/cm^2)	Initial C.C.V.	Average Dischg. Voltage	Time to 2.0 VF, Hours	Cathodic Eff., %
1	B	A	3.38	- 5	0.133	1.2	3.17	2.68	9.18	36.1
2	B	A	3.19	- 5	0.133	1.2	3.18	2.32	8.53	35.5
3	B	A	3.51	- 5	0.133	1.2	3.17	2.66	9.64	36.5
4	A	B	4.40	+35	0.133	1.2	3.24	2.97	15.00	45.4
5	A	B	4.56	+35	0.133	1.2	3.26	2.96	19.58	57.0
6	A	B	4.30	+35	0.133	1.2	3.31	2.95	16.78	51.9
7	A	A	4.15	- 5	0.350	3.0	3.15	2.75	5.30	44.8
8	A	A	4.26	- 5	0.350	3.0	3.13	2.76	6.06	49.8
9	A	A	4.17	- 5	0.350	3.0	3.08	2.78	5.95	50.0
10	A	A	4.37	+35	0.350	3.0	3.15	2.66	6.30	50.4
11	A	A	4.47	+35	0.350	3.0	3.13	3.00	7.75	60.8
12	A	A	4.34	+35	0.350	3.0	3.15	2.93	7.67	61.9
13	A	A	4.17	- 5	1.00	8.6	2.73	2.72	2.60	62.5
14	A	A	4.59	- 5	1.00	8.6	2.75	2.75	2.90	63.3
15	A	A	4.26	- 5	1.00	8.6	1.29	1.29	2.50	58.8
16	A	A	4.36	+35	1.00	8.6	2.99	2.78	1.87	42.8
17	A	A	4.32	+35	1.00	8.6	2,92	2.72	1.92	44.4
18	A	A	4.23	+35	1.00	8.6	2.98	2.83	2.60	61.6

TABLE I (Continued)

DISCHARGE PERFORMANCE OF CuF_2-Li CELLS

Cell No.	Pos. Mix	Elec'yte Batch	Theo. Cath. AH	Temp., °C	Current, A (mA/cm^2)		Initial C.C.V.	Average Dischg. Voltage	Time to 2.0 VF, Hours	Cathodic Eff., %
19	B	A	3.65	- 5	2.50	21.6	2.34	2.44	0.50	34.2
20	B	A	3.66	- 5	2.50	21.6	2.42	2.59	0.86	58.8
21	B	A	3.48	- 5	2.50	21.6	2.34	2.37	0.86	61.9
22	B	B	3.81	+35	2.50	21.6	2.60	2.58	0.45	30.2
23	B	B	4.63	+35	2.50	21.6	2.58	2.59	0.47	25.4
24	B	B	4.46	+35	2.50	21.6	2.64	2.58	0.35	19.6
25	B	A	3.85	- 5	4.00	34.5	2.27	2.36	0.38	39.5
26	B	A	3.81	- 5	4.00	34.5	1.67	2.38	0.55	57.7
27	B	A	3.97	- 5	4.00	34.5	2.14	2.37	0.43	43.3
28	B	B	4.35	+35	4.00	34.5	2.42	2.42	0.21	19.7
29	B	B	4.44	+35	4.00	34.5	2.28	2.42	0.30	27.0
30	B	B	4.29	+35	4.00	34.5	2.28	2.41	0.20	18.6

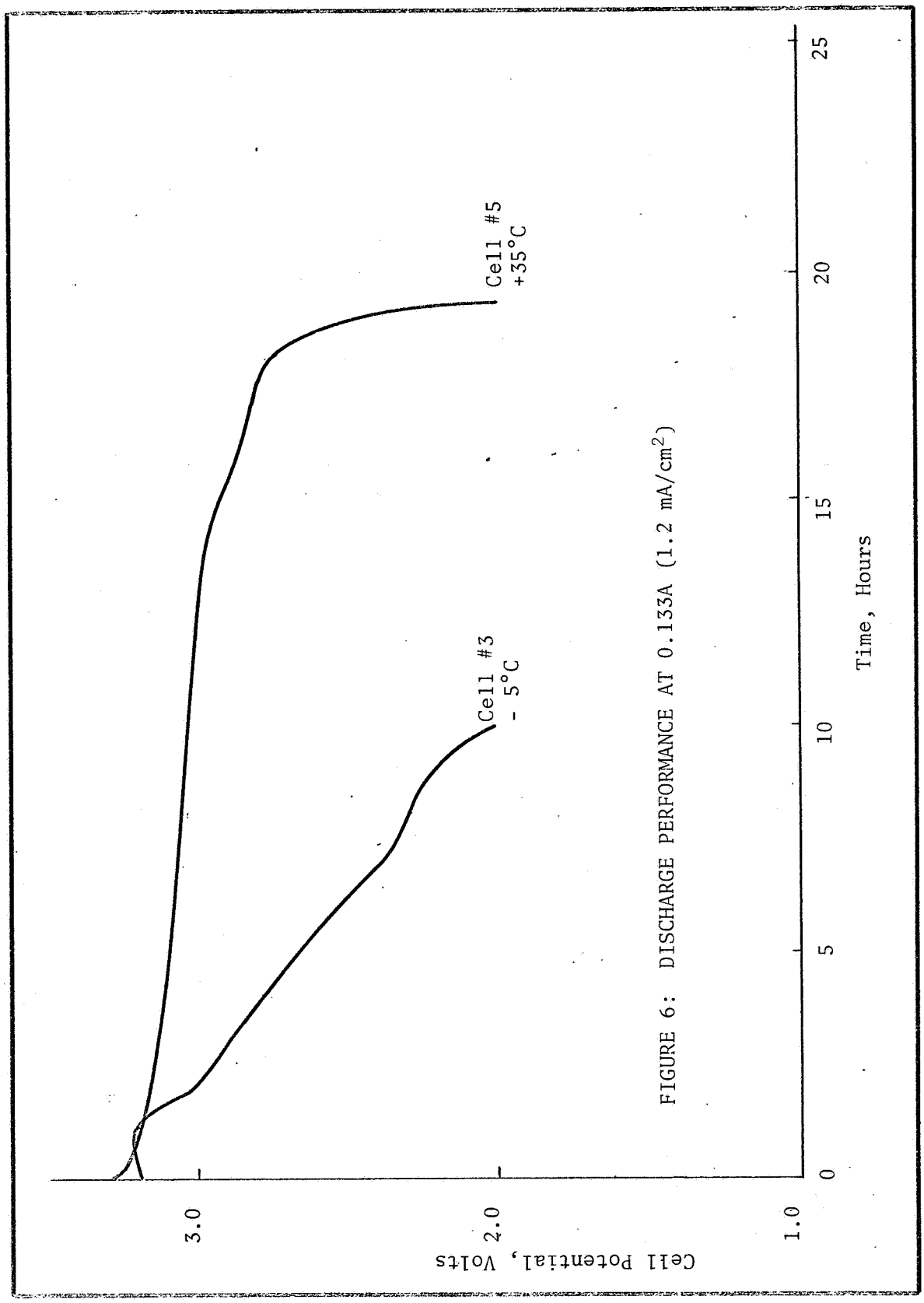

FIGURE 6: DISCHARGE PERFORMANCE AT 0.133A (1.2 mA/cm^2)

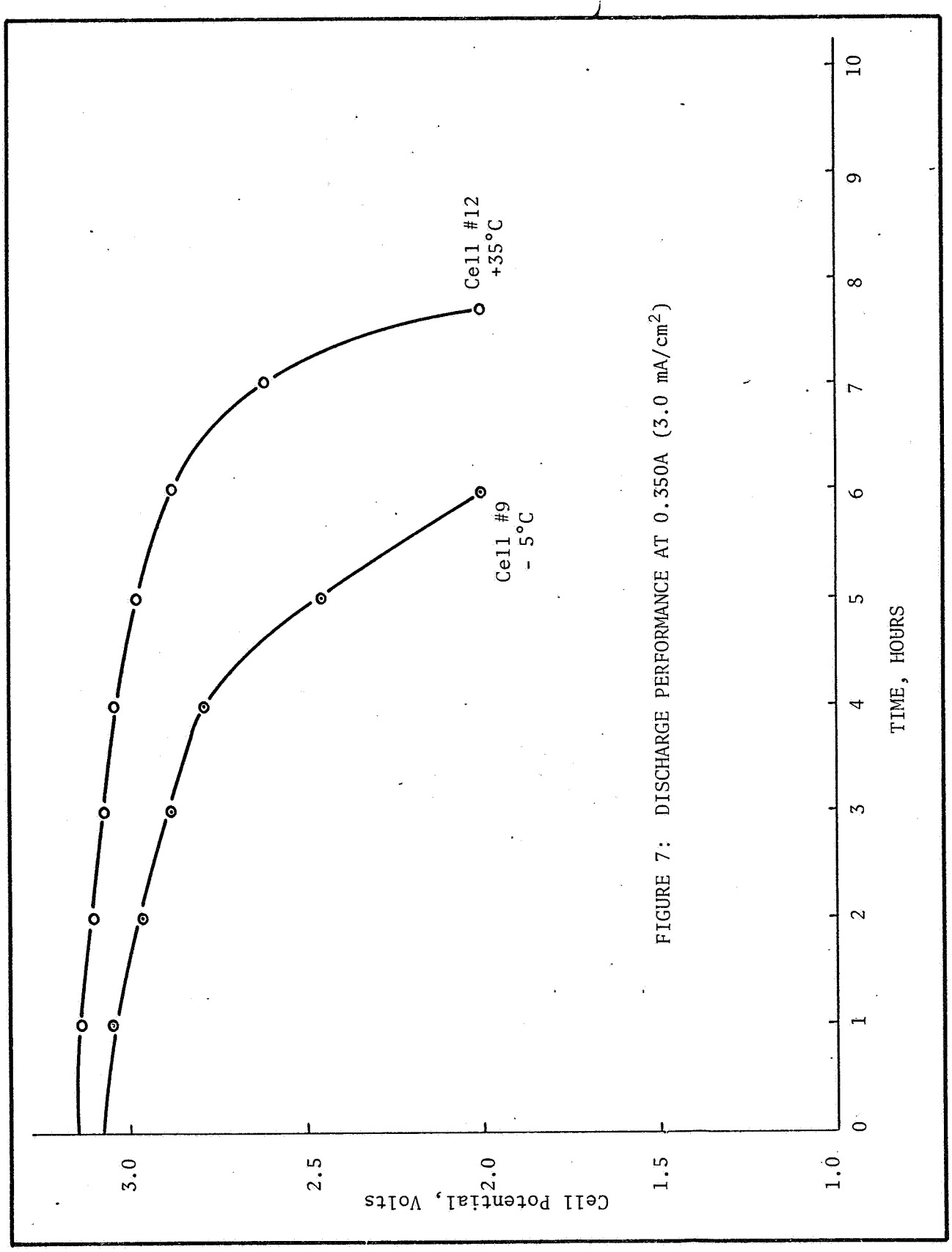

FIGURE 7: DISCHARGE PERFORMANCE AT 0.350A (3.0 mA/cm^2)

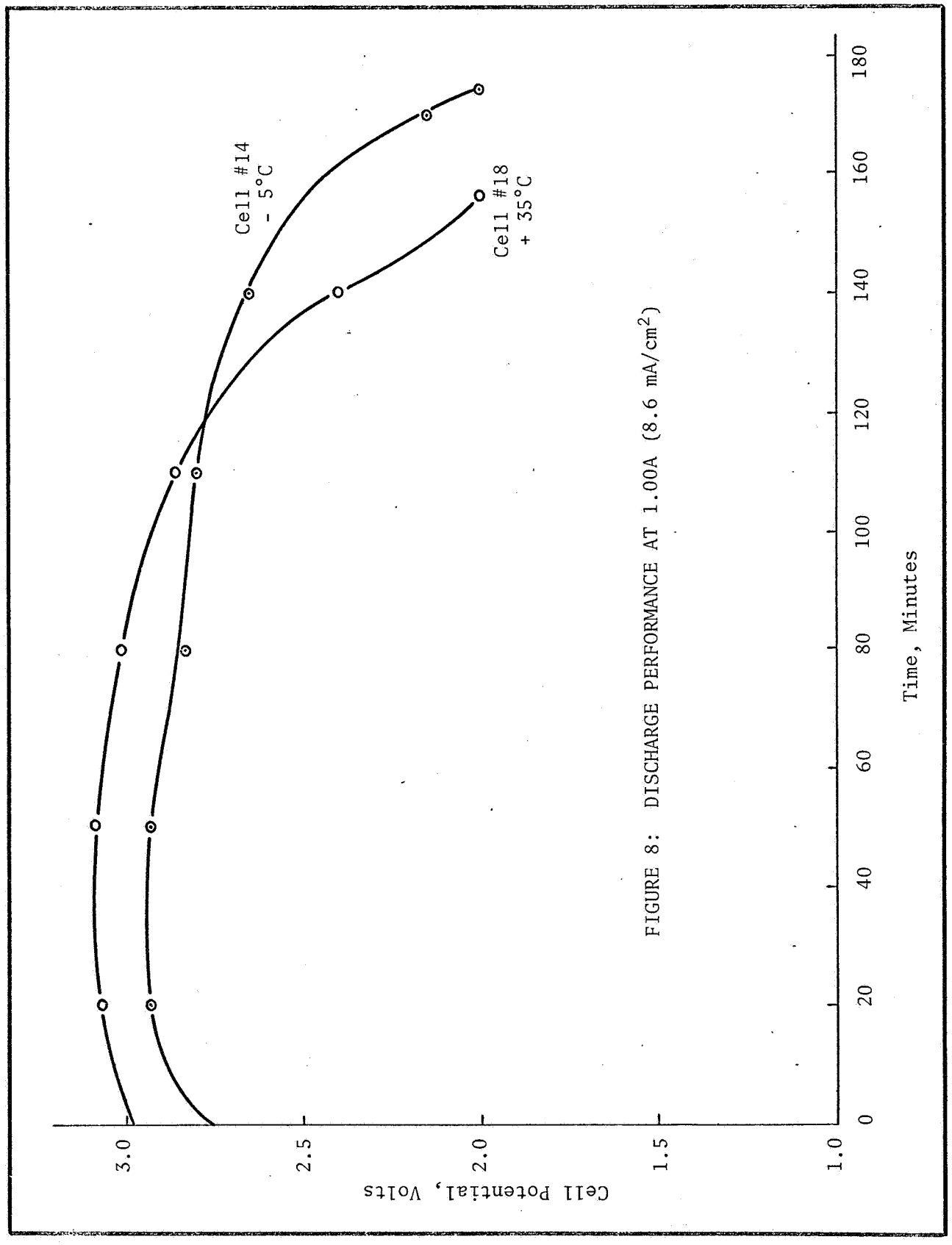

FIGURE 8: DISCHARGE PERFORMANCE AT 1.00A (8.6 mA/cm^2)

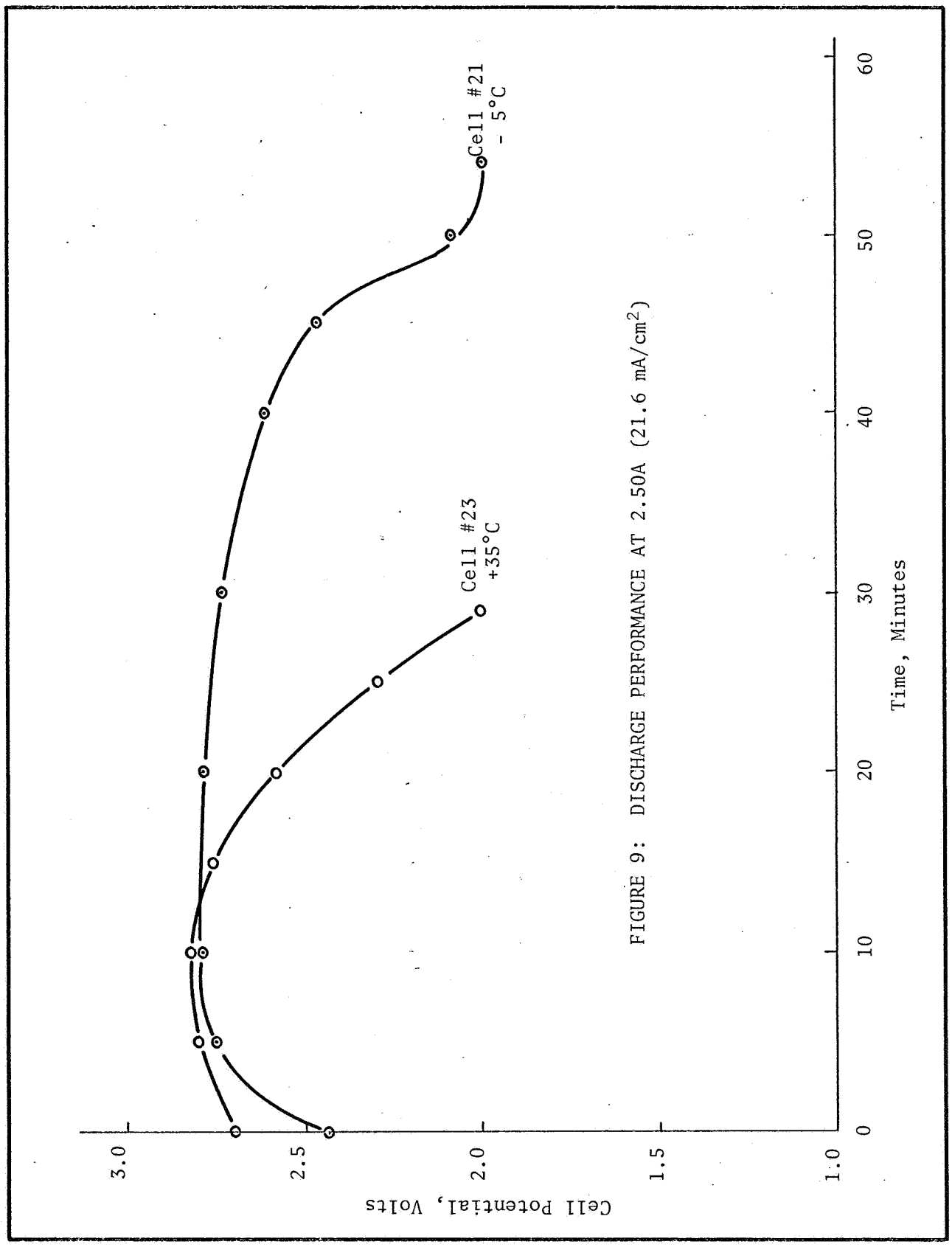

FIGURE 9: DISCHARGE PERFORMANCE AT 2.50A (21.6 mA/cm^2)

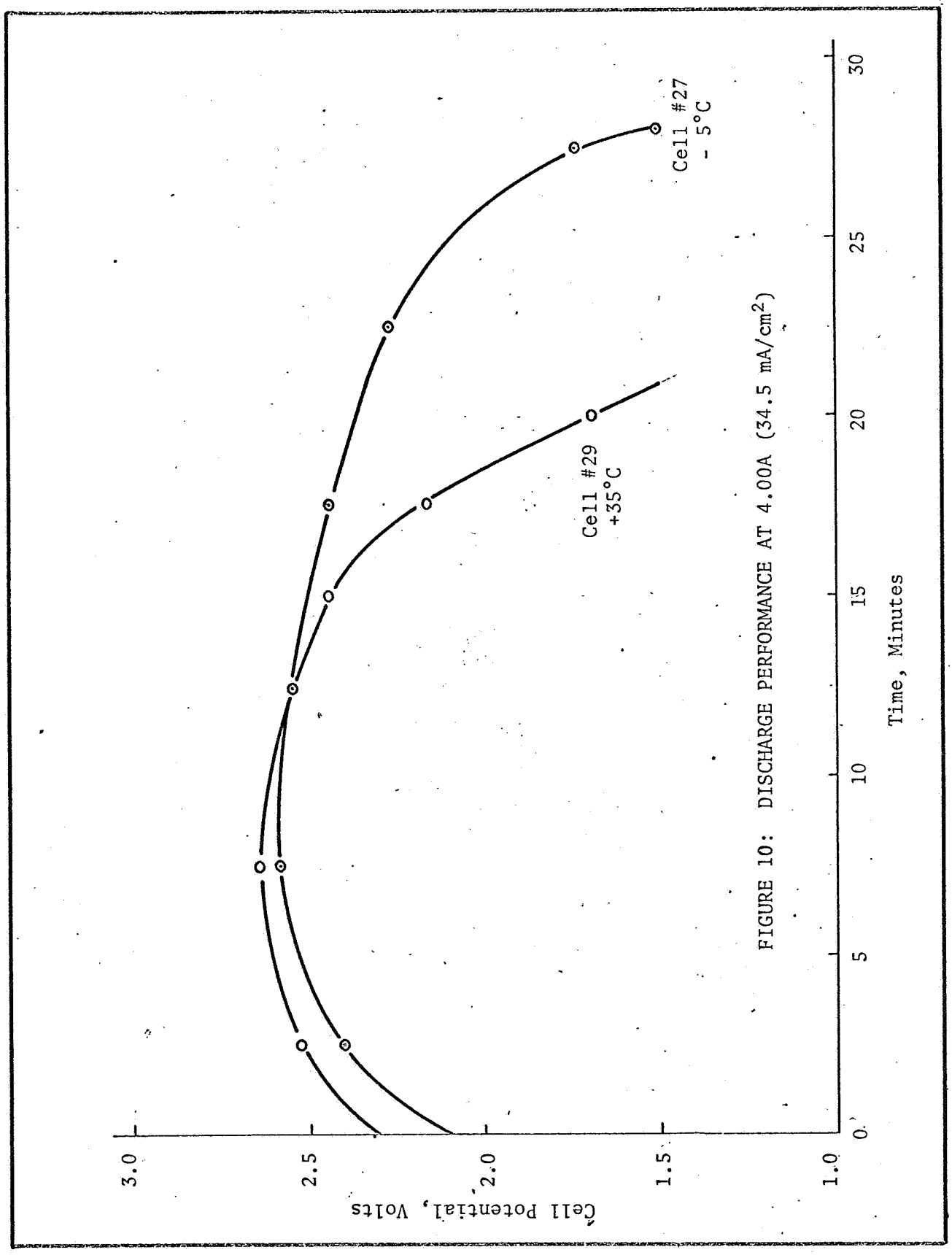

FIGURE 10: DISCHARGE PERFORMANCE AT 4.00A (34.5 mA/cm^2)

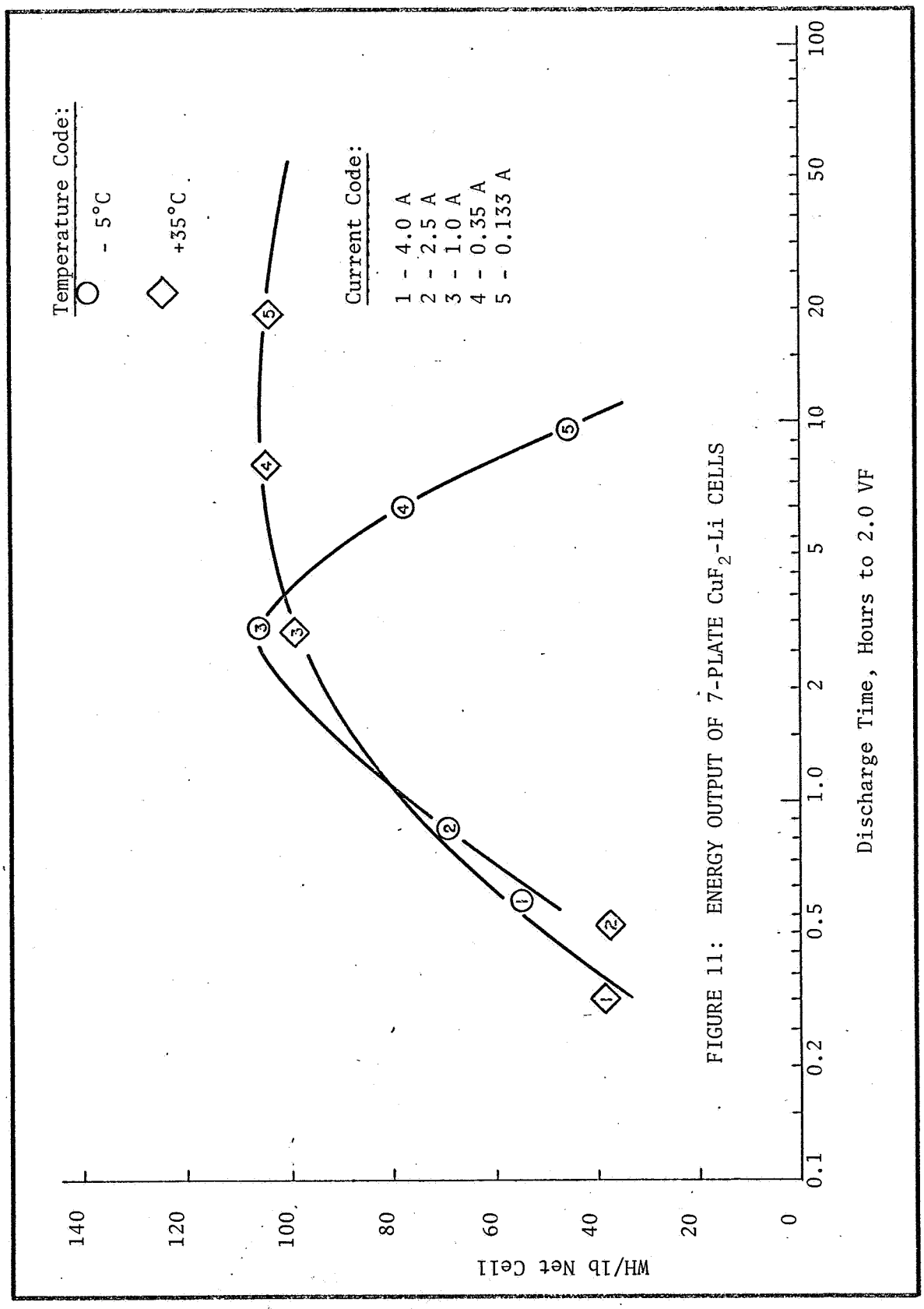

FIGURE 11: ENERGY OUTPUT OF 7-PLATE CuF_2-Li CELLS

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