

**UNCLASSIFIED**

**AD** 412865

**DEFENSE DOCUMENTATION CENTER**

FOR

**SCIENTIFIC AND TECHNICAL INFORMATION**

CAMERON STATION, ALEXANDRIA, VIRGINIA



**UNCLASSIFIED**

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

CATALOGED BY DDC

AS AD No. \_\_\_\_\_

412865

412865

# A THIN - PLATE BATTERY

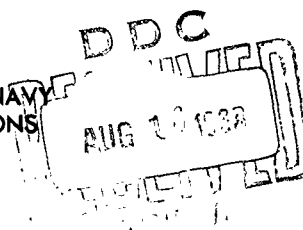
SUPPLEMENT TO

## Thirteenth Quarterly Report on Molecular Circuit Development

Contract NOw60-0362-c

Submitted to

U. S. DEPARTMENT OF THE NAVY  
BUREAU OF NAVAL WEAPONS  
Washington 25, D. C.



### MELPAR INC

3000 ARLINGTON BOULEVARD

FALLS CHURCH, VIRGINIA

A THIN-PLATE BATTERY  
SUPPLEMENT  
TO  
THIRTEENTH QUARTERLY REPORT  
ON  
MOLECULAR CIRCUIT DEVELOPMENT

Period of 15 May to 15 August 1963

Prepared by

R. T. Foley  
J. Smit  
H. H. Titus

Contract No. N0w 60-0362-C

Submitted to

U.S. Department of the Navy  
Bureau of Naval Weapons  
Washington 25, D.C.

Submitted by

Melpar, Inc.  
3000 Arlington Boulevard  
Falls Church, Virginia

## TABLE OF CONTENTS

	<u>Page</u>
1. INTRODUCTION	4
2. TECHNICAL EFFORT	5
2.1 Description of Technical Effort	5
2.2 Results of Technical Effort	5
2.2.1 Multiple Cell Evaluation, Fixture Tested Fractional Factorial Experiments	6
2.2.1.1 Experimental Conditions	6
2.2.1.2 Estimate of Error Variance	7
2.2.1.3 Evaluation of Main Effects and Interactions	14
2.2.1.4 Summary	16
2.2.2 Multiple-Cell Evaluation, Fixture Tested	17
2.2.3 Anode-Cathode Couple Effect	21
2.2.4 Data Handling	24
3. CONCLUSIONS AND RECOMMENDATIONS	25
4. PROGRAM FOR NEXT QUARTER	28

## LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Voltage Curve Battery Run TC 7	26
2	Voltage Curve Battery Run TC 15	27

## LIST OF TABLES

<u>Table</u>		<u>Page</u>
IA	Fractional Factorial Experiment, Voltage Table	8
IB	Fractional Factorial Experiment, Pressure and Temperature Table	9
II	Fixture Test Battery Series FTMC-1 through FTMC-7	18
IIIA	Fixture Test Battery Series, Voltage Table FTMC-8-32	19
IIIB	Fixture Test Battery Series, Pressure Temperature Table	20
IV	Magnesium Corrosion Study, Pressure and Temperature Table	23

## LIST OF ANALYSIS CHARTS

<u>Chart</u>		<u>Page</u>
I	Analysis Summary of $\frac{1}{2}(2)^5$ Factorial Designs	10
II	Actual Voltage Levels at 4 Minutes of Closed Circuit Operation	12
III	Actual and Delta Pressures at 20 Seconds of Closed-Circuit Operation	13

## 1. INTRODUCTION

This supplement is a description of the technical progress in a special power-source program. This program is directed toward the development of a (practical reserve-type primary battery based on the NOLC liquid-ammonia activated) cell. The requirements for such a power source can be characterized as follows:

- a. High energy-to-weight ratio.
- b. High energy-to-volume ratio.
- c. High current-density drain-rate capability with a voltage output regulation of  $\pm 10\%$  for 6 to 10 minutes.
- d. Rapid activation.
- e. Uniformity of electrical characteristics over the military range of operating temperature.
- f. Safety and reliability of operation
- g. Ease and economy of fabrication.

The active constituents of the cell, magnesium and meta-dinitrobenzene, have, theoretically, the capability of meeting the energy requirements. Liquid ammonia, the solvent for the system fulfills the temperature requirements. Additionally, the investigation of single-cell characteristics has demonstrated the practicability of utilizing the NOLO cell at high current-density drain rates. However, many problems are involved in the transistion from a single-cell unit to a completely self-contained multi-cell device. It is the prime object of this program to accomplish this transistion.

## 2. TECHNICAL EFFORT

### 2.1 Description of Technical Effort

During this report period emphasis was placed on the fabrication and fixture testing of 28-volt series battery packs. These units were tested in the new multi-cell test fixture described in the twelfth quarterly report. Different modes of activation and details of fabrication were evaluated in terms of voltage output, pressure rise, and temperature rise. These tests included a fractional factorial series of experiments, comprising five battery variables at two levels.

An investigation of the effect on the magnesium-stainless steel couple of ammonia solutions was also initiated during this quarter. This study was undertaken to determine the extent to which galvanic corrosion contributed to the pressure and temperature rises within the battery.

Additionally during this period a punch card code system for handling battery test data was devised. Details of the preceding as well as pertinent data are given in subsequent sections.

### 2.2 Results of Technical Effort

Battery performances are evaluated on the basis of the peak output voltage, the voltage levels at 1, 2, 3, 4, and 5 minutes, the peak internal pressure, and the peak internal temperature. These values in addition to construction and activation parameters are contained in the tabulated data.

The test units were all edge-fed laminate series battery assemblies. These units were composed of electrodes formed by spot welding 0.006-inch-thick magnesium discs to 0.0018-inch-thick stainless steel discs. A segment was cut from the discs to provide a free volume section along the



periphery of the plate stack. The electrode area was approximately 10 cm<sup>2</sup>. A nominal 0.33 grams of cathode matrix was applied to the stainless steel surface of the magnesium-stainless steel combination. The magnesium was the anode side of cell, the matrix the cathode side. All units contained 15 cells, i.e., 14 magnesium-stainless steel-matrix combinations as described above, and one each end-matrix cathode and end-magnesium anode. All electrodes within each unit were set on a center mandrel of polypropylene, within the battery case shell, and locked into position at the required depth. The mandrel served a dual purpose since it also contains a thermocouple to monitor internal cell temperatures.

#### 2.2.1 Multiple Cell Evaluation; Fixture Tested, Fractional Factorial Experiments

2.2.1.1 Experimental Conditions: Sixteen multiple-cell batteries were fabricated and activated to provide data for a fractional factorial analysis. Five factors at two levels were combined according to a planned statistical approach to study their effects and interactions upon cell voltage and pressure. These factors were the following:

- a. Activation Pressure - initial pressure over the electrolytic solution.
- b. Cell Thickness - distance of separation between the surface of the anode and the cathector.
- c. Electrolyte quantity - amount of electrolyte in the electrolyte reservoir.
- d. Toggle time - length of time the activation valve was open.

e. Anode Geometry - solid magnesium anode and perforated magnesium anode with 140 holes each 1/16 inch in diameter.

Battery pressure was not permitted to exceed 1000 psia. When pressures approached this level the system was vented back into the electrolyte reservoir. Venting altered the voltage level and, therefore, could have affected voltage analysis results.

The data used for this analysis are found in table 1A and 1B. Voltage levels of 2, 4, and 5 minutes were used for the voltage analyses. The pressure analysis was based on the change in pressure from the activation pressure after 20 seconds of closed circuit operation.

The numerical results of the experiment and the factors of significance are shown in chart I. Voltage and Pressure comparison aids are found in charts II and III.

Statistical procedures are based on fractional factorial designs contained in the Design and Analysis of Industrial Experiments, O. S. Davies.

2.2.1.2 Estimation of Error Variance: The estimate of error variance (an estimate of the random sampling fluctuations after controlling a set of factors believed to affect the parameters of the distribution) in this analysis is based on all negligible mean squares. Although this system of error estimation is biased, giving significance to factors which might not be significant, it assures the inclusion of all possible significant factors in future experiments.

Another estimation of error variance was calculated at the 4- and 5-minute voltage levels, using all mean squares except those of factors "B"

TAB  
 FRACTIONAL FACTORIAL EXPERIMENTAL PRO  
 15 CELL SERIES BATTERY. ELECTROLYTE C  
 AVERAGE CATHODE WEIGHT 0.33±0.05 GRA

RUN NO	DATE RUN	AVERAGE CELL THICKNESS INCHES	ELECTROLYTE AVAILABLE GRAM	VALVE TIME SECONDS	ACTIVATION PRESSURE PSIA	ANODE GEOMETRY	PEAK VOLTAGE VOLTS
TC-1	9 JULY	0.041	14.5	10	250	SOLID	29.5
TC-2	16 JULY	0.040	14.5	10	520	PERFORATED	29.3
TC-3	22 JULY	0.048	14.5	10	250	PERFORATED	29.1
TC-4	9 JULY	0.048	14.5	10	520	SOLID	29.2
TC-5	22 JULY	0.040	15.5	10	250	PERFORATED	29.1
TC-6	19 JULY	0.040	15.5	10	520	SOLID	28.2
TC-7	23 JULY	0.048	15.5	10	250	SOLID	29.4
TC-8	26 JULY	0.048	15.5	10	520	PERFORATED	29.3
TC-9	17 JULY	0.040	14.5	3	250	PERFORATED	28.3
TC-10	24 JULY	0.040	14.5	3	520	SOLID	23.7
TC-11	12 JULY	0.048	14.5	3	250	SOLID	29.4
TC-12	20 JULY	0.048	14.5	3	520	PERFORATED	29.0
TC-13	18 JULY	0.040	15.5	3	250	SOLID	29.3
TC-14	26 JULY	0.040	15.5	3	520	PERFORATED	28.6
TC-15	18 JULY	0.048	15.5	3	250	PERFORATED	29.6
TC-16	17 JULY	0.048	15.5	3	520	SOLID	29.6

1

TABLE 1 A

FACTORY FACTORIAL EXPERIMENTAL PROGRAM 5 VARIABLES 2 LEVELS VOLTAGE TABLE  
 5 CELL SERIES BATTERY. ELECTROLYTE CONCENTRATION 34% BY WEIGHT  $\text{NH}_4\text{SCN}$  IN  $\text{NH}_3$ .

AVERAGE CATHODE WEIGHT  $0.33 \pm 0.05$  GRAM PER PLATE. EXTERNAL LOAD 50.2 OHM

VARIATION NUMBER	ANODE GEOMETRY	PEAK VOLTAGE VOLTS	VOLTAGE 1 MINUTE	VOLTAGE 2 MINUTES	VOLTAGE 3 MINUTES	VOLTAGE 4 MINUTES	VOLTAGE 5 MINUTES	PEAK INTERNAL TEMPERATURE °C	PEAK PRESSURE PSIA	PEAK CURRENT DENSITY MA/CM <sup>2</sup>
50	SOLID	29.5	28.8	27.7	25.2	21.2	17.8	88	810	58.8
20	PERFORATED	29.3	27.6	27.7	25.2	22.0	18.1	78	VENT 1:00 990	58.3
50	PERFORATED	29.1	27.2	26.0	25.3	24.1	21.5	79	630	57.9
20	SOLID	29.2	28.4	27.3	25.5	22.8	18.5	82	VENT 1:30 820	58.3
50	PERFORATED	29.1	27.9	27.4	25.0	20.3	15.0	94	792	58.1
20	SOLID	28.2	27.6	25.3	23.1	20.3	--	85	VENT 0:38 990	58.3
50	SOLID	29.4	28.3	28.0	27.2	26.0	23.6	83	670	58.6
20	PERFORATED	29.3	27.5	27.4	26.5	25.2	23.5	82	956	58.4
50	PERFORATED	28.3	27.2	23.8	19.8	15.3	--	79	VENT 0:48 996	58.6
20	SOLID	23.7	22.6	20.2	18.0	16.0	13.5	-	VENT 0:38 952	47.3
50	SOLID	29.4	28.6	27.8	26.3	24.2	20.0	84	820	58.6
20	PERFORATED	29.0	27.0	26.4	24.8	22.6	19.4	88	972	57.8
50	SOLID	29.3	27.7	26.5	23.8	19.6	--	96	836	58.2
20	PERFORATED	28.6	27.7	27.2	25.2	19.5	15.4	94	VENT 0:36 992	56.9
50	PERFORATED	29.6	28.4	28.0	26.8	25.2	22.2	95	876	59.0
20	SOLID	29.6	27.6	27.7	26.4	23.8	17.2	88	VENT 0:50 990	59.0



TABLE I B

FRACTIONAL FACTORIAL EXPERIMENTAL PROGRAM  
ALL PRESSURES IN PSIA. ALL TEMPER

RUN NO	DATE RUN	INITIAL PRESSURE	PRESSURE 20 SECONDS	VENT PRESSURE AND TIME	PRESSURE 1 MINUTE	PRESSURE 2 MINUTES	PRESSURE 3 MINUTES	PRESSURE 4 MINUTES	PRESSURE 5 MINUTES
TC-1	9 JULY	250	336		512	704	784	806	810
TC-2	16 JULY	520	708	00:52 <sup>900</sup> 614	614	638	650	650	648
TC-3	22 JULY	250	312		396	488	548	584	608
TC-4	9 JULY	520	576	01:30 <sup>820</sup> 646 02:52 <sup>816</sup> 694	736	728	694	730	740
TC-5	22 JULY	250	436		660	760	792	784	768
TC-6	19 JULY	520	748	00:38 <sup>900</sup> 588	588	592	592	592	592
TC-7	23 JULY	250	332		442	494	602	636	658
TC-8	26 JULY	520	592		704	804	828	888	916
TC-9	17 JULY	250	676	00:48 <sup>996</sup> 452	452	468	482	486	-
TC-10	24 JULY	520	760	00:38 <sup>952</sup> 576	680	682	682	688	TH
TC-11	12 JULY	250	452		648	772	812	818	820
TC-12	20 JULY	520	648		772	868	916	960	972
TC-13	18 JULY	250	500		680	812	832	836	836
TC-14	26 JULY	520	820	00:36 <sup>992</sup> 600	692	758	748	740	728
TC-15	18 JULY	250	448		640	784	840	860	872
TC-16	17 JULY	520	772	00:50 <sup>992</sup> 626	680	768	760	754	740

1

TABLE I B

ADDITIONAL FACTORIAL EXPERIMENTAL PROGRAM PRESSURE AND TEMPERATURE TABLE  
 ALL PRESSURES IN PSIA. ALL TEMPERATURES IN ° CENTIGRADE

PRESSURE 3 MINUTES	PRESSURE 4 MINUTES	PRESSURE 5 MINUTES	INITIAL TEMPERATURE	TEMPERATURE 30 SECONDS	TEMPERATURE 1 MINUTE	TEMPERATURE 1 1/2 MINUTES	TEMPERATURE 2 MINUTES	TEMPERATURE 3 MINUTES	TEMPERATURE 4 MINUTES
784	806	810	24	33	49	64	74	84	88
650	650	648	25	40	64	73	76	78	78
548	584	608	24	36	46	54	61	72	77
604	730	740	24	32	43	52	60	75	81
792	784	768	24	46	70	85	91	94	94
592	592	592	28	62	82	84	85	85	83
602	636	658	23	41	48	59	67	78	81
828	888	918	25	37	49	60	67	75	80
482	486	-	26	72	76	75	75	77	78
682	688	THERMOCOUPLE MALFUNCTION							
812	818	820	25	41	61	73	80	84	84
916	960	972	28	40	54	66	74	83	87
832	836	836	27	45	72	87	94	96	95
748	740	728	28	56	80	90	94	94	94
840	860	872	30	45	63	77	86	92	95
760	754	740	25	41	69	81	87	88	86



CHART I  
 Analysis Summary of  $\frac{1}{2}(2)^5$  Factorial Design

Treatment Combinations	Testing Sequence	Mean Squares				Effect and Alias
		2-Min Voltage	4-Min Voltage	5-Min Voltage	A Pressure (20 sec)	
1	2	--	--	--	--	--
ae	6	2.25	0.86	0.72	42	A, -BCDE
be	13	10.24**	98.51**	114.49**	45,150**	B, -ACDE
ab	3	1.00	2.64**	12.25**	132	AB, -CDE
ce	14	6.40*	8.56**	5.52*	1,980	C, -ABDE
ac	15	0.12	0.05	0.20	5,700*	AC, -BDE
bc	11	0.07	0.11	1.44	1,122	BC, -ADE
abce	1	0.56	0.33	0.09	182	-DE, ABC
de	7	5.29*	15.41**	28.62**	67,340**	D, -ABCE
ad	16	0.64	0.08	0.30	5,112*	AD, -BCE
bd	5	8.41**	7.71**	1.44	30	BD, -ACE
abde	12	0.16	0.03	0.49	4,830*	-CE, ABD
cd	9	8.70**	4.31**	0.42	1,806	CD, -ABE
acde	4	5.52*	0.01	8.12**	11,342**	-BE, ACD
bcde	10	10.56**	9.77	17.64**	7,140*	-AE, BCD
abcd	8	0.72	0.01	0.64	1,640	-E, ABCD

(Continued on next page)

CHART I: Analysis of  $1/2(2)^5$  Factorial Designs (Cont'd.)

Estimate of Error Variance and Degrees of Freedom							
2-Min Voltage		4-Min Voltage		5-Min Voltage		$\Delta P$ (20 sec)	
Error Variance	Df	Error Variance	Df	Error Variance	Df	Error Variance	Df
0.73	8	0.46	8	0.64	9	867	8

Level of Significance							
2-Min Voltage		4-Min Voltage		5-Min Voltage		$\Delta P$ (20 sec)	
1%	5%	1%	5%	1%	5%	1%	5%
8.25	3.88	2.15	0.97	6.82	3.28	9,797	4,612

\*\* Significant at 1% level

\* Significant at 5% level



**CHART II**  
**Actual Voltage Levels at 4 Minutes of Closed Circuit Operation**  
**(Volts)**

Activation Pressure	Cell Thickness	Electrolyte Quantity (-) C 14.5 gm				Electrolyte Quantity (+) C 15.5 gm			
		Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC		Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC	
		Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated
A	(-) 0.040 <sup>m</sup>	21.2	(de)* 15.3			(ce) 20.3		(cd) 19.6	
			(bd)			(bc) 26.0		(bcde) 25.2	
	(+) 0.048 <sup>m</sup>			24.2					
			(ae) 22.0	(ad) 16.0		(ac) 20.3		(acde) 19.5	
520 psia	(-) 0.040 <sup>m</sup>								
			(ab) 22.8			(abce) 25.2		(abcd) 23.8	
	(+) 0.048 <sup>m</sup>								

\* Small Letters in Corners of value blocks are actual treatment combinations.

**CHART III**  
**Actual and Delta Pressures at 20 Seconds of Closed Circuit Operation**  
 (psia)

Activation Pressure	Cell Thickness	Electrolyte Quantity C (-)						Electrolyte Quantity C (+)					
		14.5 gm			14.5 gm			15.5 gm			15.5 gm		
		Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC	Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC	Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC	Toggle Time D (-) 10 SEC		Toggle Time D (+) 3 SEC
Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated	Anode Geom. E (-) Plain	Anode Geom. E (+) Perforated
A	(-) 0.040*	336	(de)* 676		(ce) 436				(cd) 500				
		[86]**	[426]		[186]				[250]				
	(+) 0.048 <sup>u</sup>	312	(bd) 452		(bc) 332								
		[62]	[202]		[82]								
(+) 520 psia	(-) 0.040*	708	(ad) 760		(ac) 748								
		[188]	[240]		[228]								
	(+) 0.048*		(ab) 576		(abce) 592								
			[56]		[72]								

\* Small letters in corners of value blocks are actual treatment combinations.  
 \*\* Bracketed values are the ΔP values.

and "D". (Factors "B" and "D" appeared to be significant and, therefore, should not be used in the estimation of error.) In both levels, factor "B" was significant at the 1-percent level and factor "D" significant at the 5-percent level. These two percentages are levels of significance usually associated with a significance test. These percentages imply the chance of concluding erroneously that a certain factor is significant. More data and further analysis will be necessary for precise conclusions as to what is the true estimate of error.

#### 2.2.1.3 Evaluation of Main Effects and Interaction:

##### a. Voltage Analysis:

(1) Cell Thickness (Effect "B") is the most significant factor. All batteries activated with the higher cell thickness performed superior to those activated at the lower cell thickness.

(2) Toggle Time (Effect "D") is the next most significant factor. Changing from 10 seconds of toggle time to 3 seconds seems to have a negative result. The evidence indicates that either insufficient cell flooding or a gasing condition exists at the 3-second toggle time. Battery performance is partially improved by the 10 second toggle time.

(3) The other main effect which seems to have some significance is the quantity of electrolyte (Factor "C"). The greater amount of electrolyte seems to improve battery performance.

(4) Analysis tabulation of Interaction AB at 5-minute voltage:

Pressure (A)	Cell Thickness (B)	
	(-) 0.040"	(+) 0.048"
(-) 250	14.7	21.7
(+) 520	16.1	19.7

The interaction between pressure and cell thickness does not become appreciably significant until the 4- and 5-minute voltage level as shown in chart 1. The higher cell thickness combined with the low pressure best maintains voltage output.

(5) Analysis tabulation of Interaction BD at 4-minutes voltage:

Cell Thickness (B)	Toggle Time (D)	
	(-) 10 sec	(+) 3 sec
(-) 0.040"	20.95	17.60
(+) 0.048"	24.53	23.95

Interaction BD affects voltage life during the earlier stages of battery life (2 and 4 minutes). The higher cell thickness combined with the longer toggle time gives maximum performance. The effect of toggle time is of less significance at the higher cell thickness.

(6) Analysis tabulation of Interaction CD at 4-minutes voltage:

Electrolyte Quantity (C)	Toggle Time (D)	
	(-) 10	(+) 3
(-) 14.5	22.53	19.53
(+) 15.5	23.20	22.03

Interaction CD appears to affect early voltage levels, but is non-significant at 5 minutes of voltage output. Battery performance is improved by the addition of the greater amount of electrolyte at the longer toggle time.

(7) Interactions BE and AE are believed to be confused with their aliases ACD and BCD respectively. (In a half factorial design it is necessary that a comparison which estimates a required effect also estimates one other effect. These effects are then confounded and may be said to be aliases of one another. Careful choice of experimental design may insure that important effects which are required to estimate are not confounded with one another.) Interaction BCD is probably a combination of interactions between BD and CD. Further study of these factors is necessary to determine their true significance.

b. Pressure Analysis: There are two main effects which seem to affect the battery pressure as analyzed at 20 seconds of closed circuit operation. Increased cell thickness, reduced pressure, and shortened toggle time increased battery pressure. Some of the interactions (AC, AD, ABD, ACD and AE), or their aliases, could have influenced the pressure level.

2.2.1.4 Summary: An analysis of the factorial experiment shows these results:

a. A higher cell thickness not only benefits battery output, but also reduces internal battery pressure.

b. A short toggle time seems to accelerate voltage deterioration and gives a non-beneficial pressure increase.

c. The greater amount of electrolyte gives the best results at the

two cell thickness studied.

d. A low activation pressure helps voltage performance and reduces the possibility of internal pressure venting.

e. Optimum conditions for best cell performance of the factor studied by this experiment are: high cell thickness, long toggle time, high electrolyte quantity, and low activation pressure.

#### 2.2.2 Multiple-Cell Evaluation, Fixture Testing

This series preceded in time the fractional-factorial series of experiments and provided guiding information for devising that series. The data for these batteries are given in table II, IIIA, and IIIB. The first seven batteries activated, FTMC-1 through FTMC-7, table II, were entirely exploratory in nature. These units were activated under both load and no-load condition with an excess amount of electrolyte available. These units were tested in an unlined epoxy glass fixture. Rapid pressure and temperature build-up, as well as poor performance, were evidenced in these runs. Two factors were suspect for this behavior: (1) excess electrolyte, and (2) reaction of container material with electrolyte. To remove the latter possibility, a polypropylene liner was fabricated for the epoxy glass fixture and used from run FTMC-8 and on.

Batteries FTMC-8 through FTMC-32 were activated under load with controlled amounts of electrolyte. These units were constructed with average nominal intercell spacings of 0.030 inches, 0.035 inches, or 0.040 inches. Battery performance, as shown in tables IIIA and IIIB, is dependent on average cell thickness and available electrolyte. There appears to be a limiting ratio between the amount of electrolyte available and the cell thickness. An excessive amount of electrolyte at any given

TABLE II  
 FIXTURE TEST BATTERY SERIES RUNS FTMC-1 THROUGH FTMC-7  
 15 CELL SERIES BATTERIES  
 ELECTROLYTE 34% BY WEIGHT NH<sub>4</sub>SCN IN NH<sub>3</sub>

RUN NO	AVERAGE		ELECTROLYTE AVAILABLE GRAM	VALVE TIME SECONDS	ACTIVATION PRESSURE PSI	ANODE GEOMETRY	PEAK LOAD VOLTAGE
	CELL THICKNESS INCHES	THICKNESS INCHES					
FTMC-1	0.032	0.032	16.8	10	200	PERFORATED	26.3
FTMC-2	0.036	0.036	16.6	3	220	PERFORATED	16.6
FTMC-3	-	-	-	-	-	-	-
FTMC-4	0.032	0.032	16.7	3	150	PERFORATED	23.3
FTMC-5	0.035	0.035	16.7	3	140	PERFORATED	20.1
FTMC-6	0.035	0.035	16.6	3	140	PERFORATED	18.4
FTMC-7	0.034	0.034	18.0	3	220	PERFORATED	13.5

NOTES

- FTMC-1 ACTIVATED UNDER LOAD. INTERNAL PRESSURE HELD AT 300 P.S.I. 13 VOLTS AT 3 MINUTES
- FTMC-2 NO LOAD ACTIVATION O.C.V. 25 VOLTS PEAK PRESSURE 524 P.S.I.
- FTMC-3 NEGLIGIBLE OUTPUT
- FTMC-4 ACTIVATED UNDER LOAD VENTED AT 700 P.S.I. PRESSURE PEAKED AT 10 SECONDS
- FTMC-5 NO LOAD ACTIVATION O.C.V. 31.4 VOLTS PEAK PRESSURE 520 P.S.I.
- FTMC-6 NO LOAD ACTIVATION O.C.V. 27.3 VOLTS VENTED AT 680 P.S.I. PEAK TEMPERATURE 98°C
- FTMC-7 NO LOAD ACTIVATION O.C.V. 21.8 VOLTS VENTED AT 594 P.S.I.

FIGURE 7  
 15 CELL SERIES BATTERY, 10 CM<sup>2</sup> PLATE  
 AVERAGE CATHODE WEIGHT 0.33 ± 0.05 GR

RUN NO.	AVERAGE CELL THICKNESS INCHES	WEIGHT ELECTROLYTE AVAILABLE GRAM	VALVE TIME SECONDS	ACTIVATION PRESSURE PSIA	ANODE GEOMETRY	PEAK VOLTAGE VOLTS
FTMC-8	0.035	9.0	10	180	PERFORATED	23.5
FTMC-9	0.035	11.0	10	200	PERFORATED	24.2
FTMC-10	0.035	11.0	10	EVP	PERFORATED	23.1
FTMC-11	0.031	11.0	10	180	PERFORATED	24.8
FTMC-12	0.031	13.0	10	280	PERFORATED	27.4
FTMC-13	0.035	13.0	10	240	PERFORATED	30.3
FTMC-14	0.035	11.0	10	220	PERFORATED	30.2
FTMC-15	0.035	15.0	10	-	-	EQUI
FTMC-16	0.036	15.0	10	220	PERFORATED	23.7
FTMC-17	0.035	13.0	10	220	PERFORATED	28.3
FTMC-18	0.035	14.0	10	220	PERFORATED	30.3
FTMC-19	0.035	14.0	10	220	PERFORATED	29.1
FTMC-20	0.035	14.0	10	220	PERFORATED	26.9
FTMC-21	0.035	14.0	10	220	SOLID	29.5
FTMC-22	0.035	14.0	10	220	PERFORATED	30.0
FTMC-23	0.040	14.0	10	220	SOLID	29.1
FTMC-24	0.035	15.0	10	220	SOLID	24.8
FTMC-25	0.035	14.0	10	220	SOLID	29.4
FTMC-26	0.039	15.0	10	220	SOLID	22.6
FTMC-27	0.040	14.5	10	220	SOLID	29.7
FTMC-28	0.040	14.5	10	220	SOLID	29.9
FTMC-29	0.040	14.5	10	220	PERFORATED	28.6
FTMC-30	0.035	15.0	10	220	SOLID	28.4
FTMC-31	0.040	14.5	10	220	SOLID	ERR
FTMC-32	0.042	14.5	10	220	SOLID	27.6

1



TABLE III A

## FIXTURE TEST BATTERY SERIES, VOLTAGE TABLE

16 CELL SERIES BATTERY, 10 CM<sup>2</sup> PLATE AREA. ELECTROLYTE CONCENTRATION 34% BY WEIGHT NH<sub>4</sub>SCN IN NH<sub>3</sub>  
 AVERAGE CATHODE WEIGHT 0.33 ± 0.05 GRAM PER PLATE. EXTERNAL LOAD 50.2 OHM (EXCEPTIONS WHERE NOTED)

ACTIVATION PRESSURE PSIA	ANODE GEOMETRY	PEAK VOLTAGE VOLTS	VOLTAGE 1 MINUTE	VOLTAGE 2 MINUTES	VOLTAGE 3 MINUTES	VOLTAGE 4 MINUTES	VOLTAGE 5 MINUTES	PEAK TEMPERATURE °C	PEAK PRESSURE PSIA	PEAK CURRENT DENSITY MA/CM <sup>2</sup>	NOTES
180	PERFORATED	23.5	8.3	-	-	-	-	-	364	44.8	
200	PERFORATED	24.2	21.6	12.1	5.4	-	-	74	442	50.2	
EVP	PERFORATED	23.1	15.5	10.6	8.5	-	-	74	330	46.0	
180	PERFORATED	24.8	19.7	18.6	16.9	14.3	12.6	88	442	49.3	
280	PERFORATED	27.4	21.7	20.8	20.2	19.4	18.0	95	664	54.6	
240	PERFORATED	30.3	26.6	23.3	21.7	20.2	18.3	92	650	60.3	
220	PERFORATED	30.2	23.4	20.3	20.5	19.5	18.3	96	596	60.2	
-		EQUIPMENT		MALFUNCTION							
220	PERFORATED	23.7	19.0	21.7	15.7	15.4	14.8	105	728	47.2	
220	PERFORATED	28.3	25.5	19.5	15.7	12.6		91	516	56.4	38.8% KCNS
220	PERFORATED	30.3	25.8	21.2	16.7	11.7	8.8	88	590	60.3	
220	PERFORATED	29.1	25.2	23.6	22.0	21.7	19.4	90	710	57.5	
220	PERFORATED	26.9	20.7	19.3	-	-	-	90	700	97.8	27.5 OHM LOAD
220	SOLID	29.5	27.3	24.5	22.7	21.2	19.6	78	610	58.8	
220	PERFORATED	30.0	25.8	24.7	23.0	21.8	20.8	90	748	30.0	100.1 OHM LOAD
220	SOLID	29.1	28.2	26.2	23.8	21.8	19.7	88	776	57.8	
220	SOLID	24.8	20.5	19.4	19.2	19.3	18.5	97	802	49.3	31.7% NH <sub>4</sub> SCN
220	SOLID	29.4	25.2	21.6	20.0	18.2	16.8	86	676	58.6	
220	SOLID	22.8	18.8	16.5	14.7	13.5	12.4	91	VENT 01:36 812	45.0	18 COLD VOLTS
220	SOLID	29.7	28.6	26.8	24.4	22.2	-	90	822	59.1	
220	SOLID	29.9	29.0	27.7	25.4	22.6	18.2	84	766	59.6	
220	PERFORATED	28.6	28.0	26.7	25.3	22.5	18.1	85	768	57.1	
220	SOLID	28.4	26.6	23.4	21.5	19.8	18.0	86	734	56.6	
220	SOLID	ERRATIC BEHAVIOR DUE TO CHANGE IN CATHODE PREPARATION PROCEDURE									
220	SOLID	27.6	27.4	26.3	24.3	22.5	21.0	87	810	55.1	



TA

FIXTURE TEST BATTERY SERIES,  
ALL PRESSURES PSIA. ALL TE

RUN NO.	INITIAL PRESSURE	PRESSURE 20 SECONDS	VENT PRESSURE AND TIME	PRESSURE 1 MINUTE	PRESSURE 2 MINUTES	PRESSURE 3 MINUTES	PRESSURE 4 MINUTES	PRESSURE 5 MINUTES
FTMC-8	180	180		220	-	-	-	-
FTMC-9	200	268		316	388	436	442	440
FTMC-10	EVP	140		178	208	232	252	280
FTMC-11	180	196		226	276	340	386	420
FTMC-12	200	376		520	572	628	648	660
FTMC-13	240	292		428	556	608	637	644
FTMC-14	220	250		320	394	476	522	556
FTMC-15	220					EQUIPMENT MALFUNCTION		
FTMC-16	220	352	2:15 <sup>728</sup> 592	590	726	644	592	604
FTMC-17	220	240		308	420	480	516	-
FTMC-18	220	348	1:20 <sup>590</sup> Slow Vent	544	484	412	374	344
FTMC-19	220	320		464	620	664	688	700
FTMC-20	220	320		476	636	-	-	-
FTMC-21	220	368	1:20 <sup>748</sup> 648	536	580	592	600	604
FTMC-22	220	400		700	648	642	636	626
FTMC-23	220	332		502	620	696	730	764
FTMC-24	220	352		540	712	764	788	802
FTMC-25	220	348		464	572	622	650	658
FTMC-26	220	368	1:36 <sup>812</sup> 718 2:48 <sup>788</sup> 686	656	740	688	692	690
FTMC-27	220	352		508	646	750	804	-
FTMC-28	220	324		492	634	710	744	764
FTMC-29	220	330		460	608	696	730	760
FTMC-30	220	360		544	672	724	742	752
FTMC-31	220					ERRATIC BEHAVIOR DUE TO CHANGE CATHODE PREPARAT		
FTMC-32	220	364		500	644	736	780	800

1

TABLE III B

FIXTURE TEST BATTERY SERIES, PRESSURE AND TEMPERATURE TABLE  
 ALL PRESSURES PSIA. ALL TEMPERATURES DEGREES CENTIGRADE

PRESSURE MINUTES	PRESSURE 3 MINUTES	PRESSURE 4 MINUTES	PRESSURE 5 MINUTES	INITIAL TEMPERATURE	TEMPERATURE 30 SECONDS	TEMPERATURE 1 MINUTES	TEMPERATURE 1-1/2 MINUTES	TEMPERATURE 2 MINUTES	TEMPERATURE 3 MINUTES	TEMPERATURE 4 MINUTES
-	-	-	-	-	-	-	-	-	-	-
388	436	442	440	24	42	50	57	64	73	74
308	232	252	280	23	34	39	43	47	53	57
276	340	388	420	25	38	45	50	55	67	76
572	628	648	660	24	52	77	86	92	94	95
556	608	637	644	25	46	58	73	82	90	91
394	476	522	556	28	42	50	56	65	79	87
EQUIPMENT MALFUNCTION										
726	644	592	604	27	51	76	93	101	105	100
420	480	516	-	28	38	45	56	70	83	91
484	412	374	344	25	46	70	85	88	87	87
620	664	688	700	25	40	56	70	79	86	88
636	-	-	-	25	42	56	72	82	-	-
580	592	600	604	20	42	62	72	76	77	78
648	642	636	626	25	50	73	86	90	89	88
620	696	730	764	24	36	52	63	71	80	85
712	764	788	802	25	47	68	82	89	96	97
572	622	650	658	24	43	58	68	75	82	85
740	688	692	690	20	40	65	82	89	91	91
646	750	804	-	24	42	56	66	73	83	89
634	710	744	764	24	38	51	61	68	77	82
608	696	730	760	24	38	50	60	69	79	84
672	724	742	752	28	50	65	74	80	85	86
NR DUE TO CHANGE CATHODE PREPARATION PROCEDURE										
644	736	780	800	23	32	44	55	69	77	82



thickness depresses closed circuit voltages (batteries FTMC-16 and FTMC-24). Insufficient amounts of electrolyte can also, in extreme cases, cause low closed-circuit voltages (battery FTMC-8). However, the run-out times are also brief. This condition can also derive from low activation pressures; i.e., pressures very near the equilibrium vapor pressure of the electrolytic solution. (Compare batteries FTMC-10 and 11 to FTMC-14). There is also an indication that anode geometry has small influence on battery performance. This evidence is contained in batteries FTMC-21 and FTMC-22. The anomalous behavior of battery FTMC-26 is attributed to an internal defect, possibly incompletely cured cathodes. This is suspected since the battery showed 18 volts on open-circuit voltage measurement prior to activation. The pressure levels were generally higher at higher battery performance levels. This was not true with respect to temperature.

### 2.2.3 Anode-Cathector Couple Effect

An indication of the contribution, which the magnesium-stainless steel couple makes to the pressure and temperature rises within the battery, was obtained through a series of experiments in which both magnesium and magnesium-stainless steel laminate discs were subjected to various ammonia solutions. The conclusions, although quite clear, are admittedly for a limited set of conditions and are single runs. Further work is planned to check these data.

The magnesium-stainless steel laminate discs used in this study were standard battery components. Five plates of either magnesium or magnesium-stainless steel laminate were used in each run, except run No. 10 which

a control. This run contained only standard fiberglass separators. The plates in each run were separated an average distance of 0.040 inches by standard fiber-glass separators. None of the plates contained the reactive cathode matrix. The series to and including run 10 was conducted on perforated magnesium discs. Each experiment was conducted with excess available solution. The data for this series are given in table IV.

Runs 9 and 10 were control runs. Run 9 checked magnesium alone against  $\text{NH}_3$ ; virtually no reaction was evident.

Run 10, in which the fiber-glass separators were in  $\text{NH}_4\text{SCN-NH}_3$  solution, also showed no resultant reaction. The general tentative conclusions from the remainder of this series of experiments are:

a. Reaction occurs between magnesium and both electrolyte solutions; i.e.,  $\text{KCNS-NH}_3$  and  $\text{NH}_4\text{SCN-NH}_3$ . (Note runs 2 and 4).

b. The reaction is much more severe in the  $\text{NH}_4\text{SCN-NH}_3$  solution than in the  $\text{KCNS-NH}_3$  solutions for both the magnesium and the magnesium-stainless steel laminate. (Compare runs 1 and 2 to runs 3 and 4.)

c. There exists a definite galvanic effect in the Mg-stainless couple. (Compare runs 1 and 3 to runs 2 and 4.)

d. The addition of m-DNB to  $\text{NH}_3$  or  $\text{NH}_4\text{SCN-NH}_3$  solutions alters the corrosion reaction. (Compare run 6 to 9 and runs 3 and 4 to 7 and 8.) This effect requires a more detailed study.

Runs No. 11 and No. 12 were conducted on solid (nonperforated) magnesium. In addition, the edges of the laminate in run No. 12 were sealed with plater's tape. This run may be compared with run No. 3 where the system was vented into the electrolyte reservoir when the pressure

TABLE  
MAGNESIUM CORROSION STUDY,  
5 PLATE TEST UNIT. AVG 1.15 GRAM. MAGNESIUM  
TEMPERATURE IN °C. PRESSURE IN PSIA ALL M

RUN NO.	PLATE	SOLUTION	CONCENTRATION BY % WEIGHT	INITIAL PRESSURE	PRESSURE 20 SECONDS	PRESSURE 1 MINUTE	PRESSURE 2 MINUTES	PRESSURE 3 MINUTES	PRESSURE 4 MINUTES
1	MG-SS	KCNS in NH <sub>3</sub>	38.8	280	288	316	344	362	374
2	MG	KCNS in NH <sub>3</sub>	38.8	278	280	294	308	322	336
3	MG-SS	NH <sub>4</sub> SCN in NH <sub>3</sub>	34.0	280	368	732 VENT 420	524	584	596
4	MG	NH <sub>4</sub> SCN in NH <sub>3</sub>	34.0	280	390	548	568	564	560
5	MG-SS	mDNE in NH <sub>3</sub>		284	288	296	306	308	308
6	MG	mDNE in NH <sub>3</sub>		280	278	306	336	358	372
7	MG-SS	NH <sub>4</sub> SCN+mDNE in NH <sub>3</sub>	34.0	280	290	414	468	464	450
8	MG	NH <sub>4</sub> SCN+mDNE in NH <sub>3</sub>	34.0	276	432	500	548	540	516
9	MG-SS	NH <sub>3</sub>	0	280	300	304	300	308	308
10	BLANK	NH <sub>4</sub> SCN in NH <sub>3</sub>	34.0	276	278	276	274	272	270
11	MG-SS	NH <sub>3</sub> SCN+mDNE+NH <sub>3</sub>	34.0	276	344	440	456	460	464
12	MG-SS	NH <sub>4</sub> SCN in NH <sub>3</sub>	34.0	220	372	556	612	628	636

1

TABLE IV

## MAGNESIUM CORROSION STUDY, PRESSURE AND TEMPERATURE

ATE TEST UNIT. AVG 1.15 GRAM. MAGNESIUM PER RUN. 14 GRAM SOLUTION 0.040 INCH SPACING  
 MPERATURE IN °C. PRESSURE IN PSIA ALL MAGNESIUM PERFORATED EXCEPT RUNS 11 AND 12

PRESSURE 1 MINUTE	PRESSURE 2 MINUTES	PRESSURE 3 MINUTES	PRESSURE 4 MINUTES	PRESSURE 5 MINUTES	INITIAL TEMPERATURE	TEMPERATURE 30 SECONDS	TEMPERATURE 1 MINUTE	TEMPERATURE 2 MINUTES	TEMPERATURE 3 MINUTES	TEMPERATURE 4 MINUTES	WEIGHT CHANGE
316	344	362	374	392	26	30	32	34	35	35	-0.0787
294	308	322	336	348	25	28	28	28	28	29	-0.0185
732 VENT 420	524	584	596	598	25	43	55	58	59	58	-0.2933
548	566	564	560	558	24	49	56	56	54	51	-0.1802
296	308	308	308	308	27	33	34	35	35	35	-0.0683
306	336	358	372	388	27	31	32	33	33	34	-0.0118
414	468	464	450	432	25	39	48	55	55	55	-0.3274
500	548	540	516	496	26	61	72	75	75	72	-0.2376
304	300	308	308	308	26	27	27	27	27	27	-0.0062
276	274	272	270	270	27	27	27	27	27	27	---
440	456	460	464	470	22	47	59	68	69	68	-0.3045
556	612	628	638	644	25	41	47	48	47	46	-----



reached 800 psia.

Generally speaking it may be said that a corrosion reaction is responsible for some of the pressure and temperature rise within the battery; however, the extent of the weight loss suggests that the corrosion reaction may be coupled with other reactions when the battery is under discharge to provide the entire pressure and temperature use.

Further studies on the galvanic corrosion effect are planned for the next quarter and are detailed in section 4.

#### 2.2.4 Data Handling

A system has been devised for retrieval of battery data using a punch card method. This method involves the extrapolation of necessary information from the new data, setting this information up in a predetermined coding form, and punching it on punch cards.

Other methods are also being looked at for possible added benefits over the punch card method. It may be possible to devise a method of feeding the information obtained from the data into an electronic computer. Research on this subject will be done during the next quarter. The intention is to reach agreement with the Corona Laboratory on the most desirable data storage and retrieval method.



### 3. CONCLUSIONS AND RECOMMENDATIONS

The output performance of multicell units continues to improve. Under present optimized conditions of activation, it is possible to obtain nearly 5 minutes of useful life at high current densities from a 15-cell series unit. Battery TC-7 (see figure 1), operating at an initial current density of approximately  $59 \text{ ma/cm}^2$ , attained a peak voltage of 29.4 volts, i.e., 1.96 volts/cell. At the end of 5 minutes of operation, the voltage level was 23.6 volts. Battery TC-15 (see figure 2) peaked at 29.6 volts and showed 22.2 volts after 5 minutes of operation. Both units were discharged across 50.2 ohms. These units were activated with 15.5 grams of available electrolyte. The intercell spacing for the units was 0.048 inches.

The analysis of the fractional factorial series appears to indicate a path by which battery performance can be further improved. The factors of cell thickness, electrolyte quantity, and toggle time bear on the output of the system. The preliminary indications are that an increase in the factors will extend the useful life of the units. An additional fractional factorial series will evaluate this conclusion.

A performance dependence on anode geometry apparently does not exist in the multicell units at the intercell spacings now employed. This conclusion has been established in the analysis of the fractional factorial series of experiments. In this analysis, the mean square value for anode geometry (i.e., factor E) in every case is less than the value needed to indicate significance. The use of the perforated anode therefore is being discontinued.

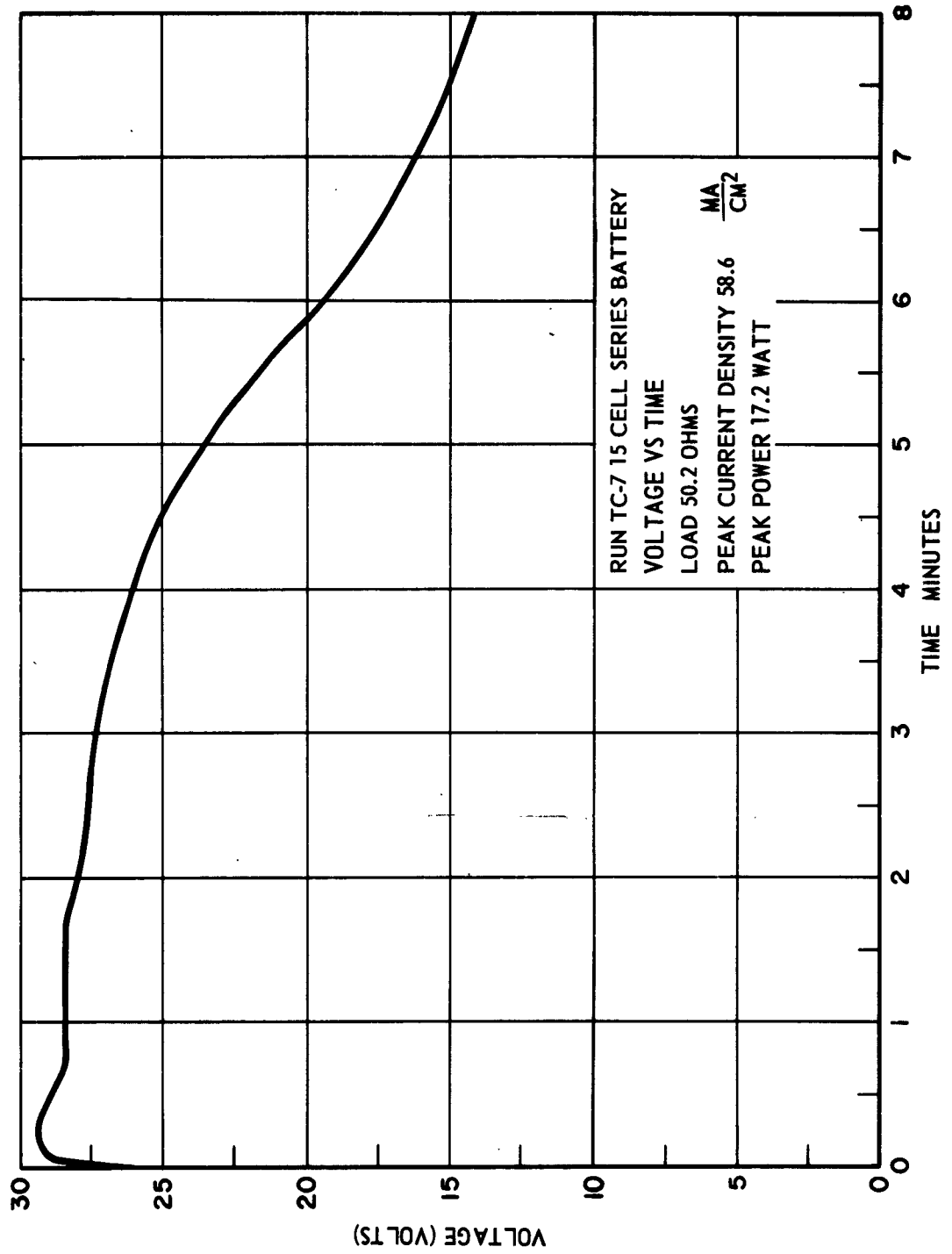


Figure 1. Voltage Curve Battery Run TC 7

R6440

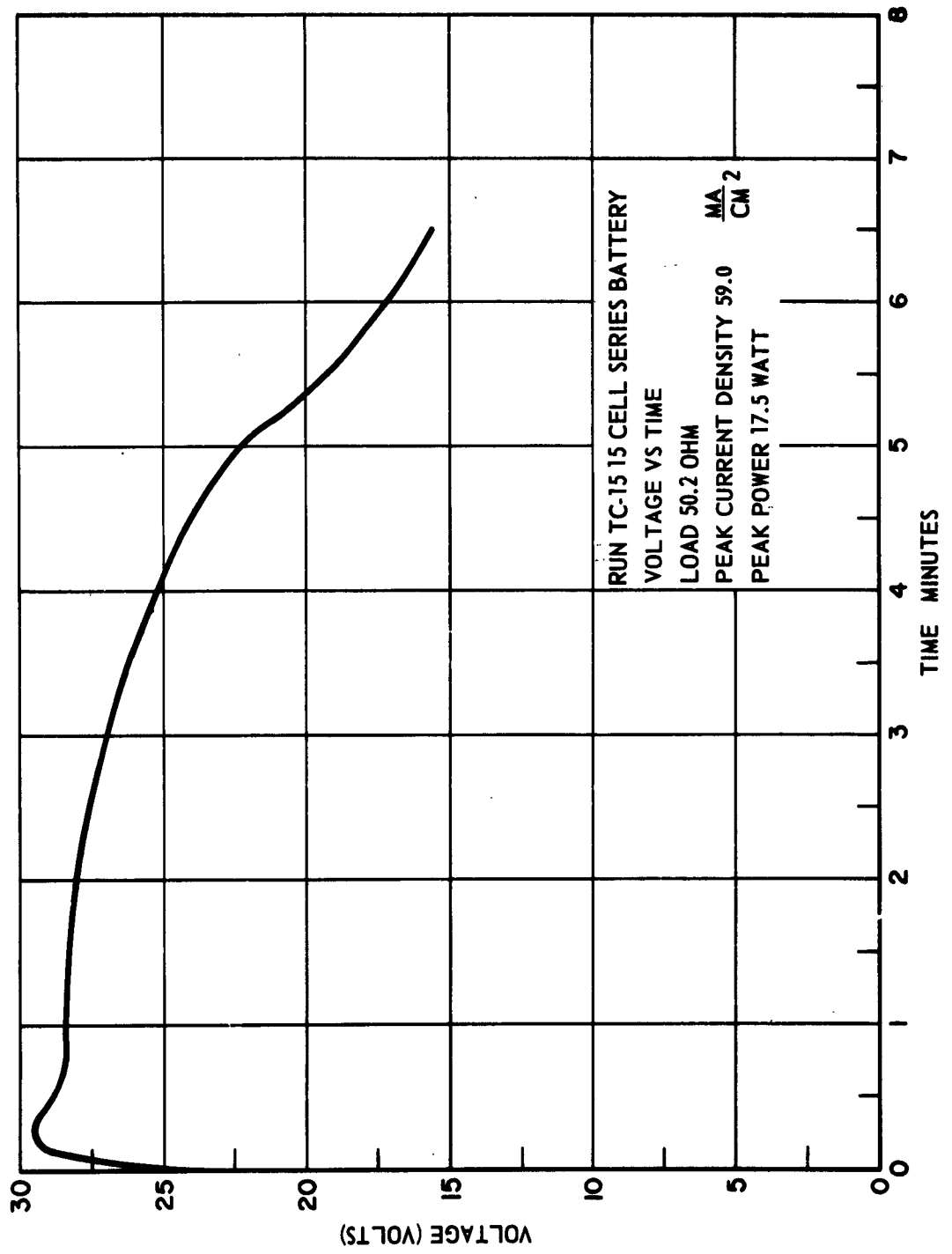




Figure 2. Voltage Curve Battery Run TC 15

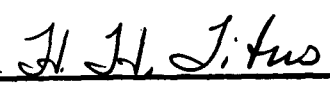
#### 4. PROGRAM FOR NEXT QUARTER


The evaluation of simulated multicell units of 28-volt rating will continue during the next quarter. Further statistical studies will be undertaken to determine the effect on the multicell units of such parameters as m-DNB particles size, cathode weight, and toggle time. These studies will be extended to include an investigation of the low-temperature characteristics of these units..


The investigation of the anode-cathector couple effect will be expanded to provide data for comparison with the pressure, temperature, and weight-loss characteristics of the multi-cell units under discharge. The investigation will be conducted on 15-plate units of both magnesium and magnesium-stainless steel laminates. The experimental conditions of spacing, solution quantity, activation pressure, etc. will match those under which the multicell units have been activated. Data thus acquired should establish conclusively the extent of the effect of the anode-cathector couple.

Prepared by:   
R. T. Foley  
Project Scientist

  
J. Smit  
Principal Investigator

  
H. H. Titus  
Chemist

Submitted by:   
C. Feldman  
Section Head  
Physical Electronics

Approved by:   
P. E. Ritt  
Vice President-Research