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RESEARCH ON AMMONIA
BATTERY SYSTEM

JULY 1, 1962 TO SEPTEMBER 30, 1962
REPORT NO. 9
FIRST QUARTERLY PROGRESS REPORT

CONTRACT NO. DA-36-039-sc-89188
(Continuation of Contract No. DA-36-039-sc-85396)

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DEPARTMENT OF THE ARMY PROJECT NUMBER 3A99-09-001

Signal Corps Technical Guidelines
for PR&C 60-ELE/R-4905
dated March 7, 1960

The object of this contract is to perform research to establish the feasibility of the liquid ammonia system in galvanic cells and develop the use of this system in batteries.

U. S. ARMY ELECTRONICS RESEARCH AND DEVELOPMENT AGENCY
FORT MONMOUTH, NEW JERSEY

THE EAGLE-PICHER COMPANY
COUPLES DEPARTMENT
P. O. BOX 290
JOPLIN, MISSOURI

PREPARED BY:

D. J. Doan, Project Supervisor
L. R. Wood, Project Engineer

REVIEWED BY:

J. F. Dittmann, Engineering Supervisor

APPROVED BY:

E. M. Morse, Engineering Manager

DATE OF REPORT: October 30, 1962

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ABSTRACT

A better understanding has been obtained of the factors contributing to the previously observed slower practical activation of the -53°C batteries as compared to single cells under ideal conditions. Tests indicate a larger percentage of the batteries activate in less than 3 seconds, with several about 1 second. A new high-rate cathode possibility (silver sulfate) has been found. Permion 300 separator has been found much better than cellophane for limiting the silver diffusion in the low-rate, long duration application.

PURPOSE

The purpose of this contract is to carry on research and development on the use of liquid ammonia as an electrolyte solvent with various cathode and anode materials, toward determining the applicability of the system generally in batteries. Specifically, the program will include the following investigations:

A. A comprehensive study of possible cathode materials for use with conventional anodes, such as: magnesium and zinc immersed in liquid ammonia electrolytic solutions. Cathodic materials of possible use in liquid ammonia systems fall into several classes which include oxides, both inorganic and organic, reducible inorganic salts and metallic nitrides, which are the "oxides" of the nitrogen system. Studies shall include measurement of E.M.F., electrode voltage as a function of discharge current density and temperature, determination of reaction products, effect of local action on electrode potential and coulometric capacity, and effect of contaminants in liquid ammonia on electrode properties. Desirable characteristics of cathodic materials include: (1) ability to maintain good cell voltage level under high and low rate discharge conditions over a wide range of temperature (-65° F to $+165^{\circ}$ F); (2) stability in electrolyte for long periods of time over a wide temperature range; (3) small temperature coefficient of voltage, and, (4) ease of fabrication into practical electrode structures.

B. Investigation of liquid ammonia electrolyte systems will be undertaken for optimum performance of new ammonia battery systems. Acid, neutral and alkaline solutions are to be investigated. Mixed salts can be used to impart desirable electrolyte properties. The physical and chemical properties of the salts and solutions will be investigated including such measurements as solubility, vapor pressure and conductivity as functions of temperature and concentration. Determination of stability and interaction with other cell components will also be made.

Studies will be made to establish cell design information best suited to achieve optimum performance throughout the temperature range of -65° F to $+160^{\circ}$ F. The investigations should provide practical design information which can be used for future battery development.

The Power Sources Division of USAELRDL has performed much work in this area and is continuing with its studies. Since this Division is monitoring the present contract, it is important to state that the contractor will maintain close liaison and will be prepared to cooperate with their suggestions through conferences, etc., toward accelerating and any altering of the plans of the contract effort. This, of course, may be brought about by the bringing to light of information by both laboratories and by changes as to emphasis on certain type batteries, etc. The contractor will obviously assume full responsibility for the quality of the contract effort.

CONFERENCES

On July 25, 1962, a conference was held at Joplin with Mr. Howard Knapp from the Signal Corps, and Mr. E. M. Morse, Mr. Lyle Wood, Mr. J. F. Dittmann and Dr. D. J. Doan from The Eagle-Picher Company. The details of the final report of the previous contract on this subject were reviewed. The program for the current effort was discussed and the following areas were agreed to be stressed:

A. Continue development of automatically activated multicell units. This will consist of fabrication of convenient size cell blocks (four to seven cells) to be filled from helical tube reservoirs, and activated with a conventional electric match gas generator. These will be tested as soon as satisfactory units are developed and with various loads at various temperatures to evaluate basic characteristics.

B. Perform single cell tests as required to assist in problem solving for the multicell unit studies under conditions similar to "A" above.

C. Continue screening tests for cathodes to develop useful material for low rate application.

D. Investigate mechanism and various means of controlling the reaction producing hydrogen from the anode.

E. Continue intensive investigation of silver chloride, especially as to effect of particle size, type formation, ammoniation and separation.

F. Continue research concerning the mechanisms and the sources of inefficiencies in the mercuric sulfate/magnesium system.

G. Continue effort toward concentrating the cell structure.

H. Attempt to improve the vapor pressure-temperature characteristics of the electrolyte through the possibility of varying the KSCN concentration, and use of MgSCN , MgClO_4 and KClO_4 .

I. INTRODUCTION

The general purpose and objectives of the present contract are fully outlined in the "Purpose" section of this report. This contract is actually a continuation of Contract No. DA-36-039-sc-85396 from the U. S. Army Electronics Research and Development Agency, Fort Monmouth, New Jersey. The detail of the work performed under this initial contract has been reported in seven quarterly reports and a final report during 1961 - 1962 and July 30, 1962, respectively. These should be used in connection with the work of this contract.

The main effort of the present work interval to be reported herein are toward the following immediate objectives:

- (1) Elucidate and improve the -50° C activation time of the six-cell battery.
- (2) Lessen the incidence of shorting found in the silver chloride cathode cells to improve capacity.
- (3) Improve anode material to decrease gas production in long-duration, low-rate units.
- (4) Continue investigation of separator materials for low-rate application.
- (5) Continue screening test in an effort to find improved high and low-rate cathode materials.

II. FACTUAL DATA

A. Automatic Activation

The principal effort during this quarter has been a continued study of low temperature activation problems. The basic premise of hardware geometry has not been changed significantly, although several variations have been made. Silver chloride cathodes have been used exclusively in multicell testing and further experimentation was done to determine the precise activation characteristics of the silver chloride cell. Specific results of multicell and automatically activated single cells are listed in Table Nos. I and II, respectively. Current effort has been concentrated on cathode performance, capacity improvement of multicell units, and activation time improvement.

1. Multicell Units

The significance of current leakage regarding cold activation has been previously established. Reduction of current leakage was based on isolation of cell entries from each other and from the steel cell container through geometry improvement in the system. Figure No. 1 shows this configuration used in Battery Nos. 100 through 104. The results shown in Table No. I were influenced by several factors and indicate the presence of additional deterrents to cold activation. The extremely slow activation of Unit No. 102 was due primarily to microporous rubber separation used. This battery was designed to study the cell concentrating effect of using microporous rubber, rather than Pellon and Vexar as separation material. From Unit Nos. 100 through 104, it was shown that capacity was seriously reduced due to the configuration. Regardless of battery orientation, gas formed during discharge is trapped in those cells with entries at or near the bottom of the system. "Post mortem" of these units revealed that certain areas of the anode were clean and bright, indicating little or no discharge. A.C. impedance measurements indicated a loss of electrolyte simultaneously with the premature failure of these units.

A.C. impedance measurements show a direct correlation to cell filling. A normal battery has been found to read approximately 0.6 ohm. Any loss of electrolyte increases this resistance accordingly. This technique has, therefore, been used to monitor cell filling (electrolyte transfer). Figure No. 2 shows the results of Battery No. 104. The quick drop in impedance indicates good electrolyte transfer and the subsequent rise at approximately six minutes predicts the premature failure of the unit. Open circuit of this battery was normal, but the system would not function even at reduced current densities, thus confirming the loss of electrolyte. Based on these data, it was assumed that the cell itself may be limiting cold activation as well as current leakage. To confirm this assumption, a single cell was assembled in the multicell hardware and activated at -54° C.

TABLE NO. I

BATTERY DATA

BAT- TERY NO.	ACT. TEMP. °C	ACT. TIME (SECONDS)			PEAK LOAD VOLTS	MAX. AMP.	MAX. A.C.Z.	% EFFICIENCY TO			TOTAL AMP. MIN.	THEOR. AMP. MIN.	END O.C.V.
		80% P.L.V.	90% P.L.V.	1.9 VOLTS				270 - MA/in.	145 2	75 -			
100	-54	2.5	3.0	3.5	12.2	2.38	00	2.68	2.68	2.68	2.4	89.8	12.8
101	-54		Abortive										
102	-54	12.0	15.0	25.0	12.0	2.35	00	50.0	50.0	50.0	41.4	82.3	6.5
103	-54	0.8	1.5	3.0	11.9	2.30	0.6	13.7	13.7	13.7	11.4	83.2	3.2
104	-54	3.0	5.0	10.0	11.8	2.10	2.4	27.4	27.4	27.4	22.0	80.5	12.5
105	-54		Abortive										
106	-54	5.0	8.0	8.5	12.6	2.44	0.82	64.4	64.4	64.4	48.3	75.1	12.5
107	-54	1.1	2.0	2.0	12.6	2.47	0.84	26.4	26.4	26.4	26.4	100.0	9.0
108	-54	0.9	2.0	3.5	12.3	2.42	0.77	24.0	24.0	24.0	24.0	100.0	8.0
109	-54	2.3	3.5	4.5	12.45	2.43	0.78	74.4	74.4	74.4	64.0	86.0	12.0

TABLE NO. II

SINGLE CELL DATA

CELL NO.	ACT. TEMP. °C	ACT. TIME (SECONDS)			PEAK LOAD VOLTS	MAX. AMP.	MAX. A.C.Z.	ACTIVATING MECHANISM	CATHODE MATERIAL
		80% P.L.V.	90% P.L.V.	TO P.L.V.					
951	-54	8.0	10.7	14.7	1.98	2.06	.054	Battery Container	AgCl
952	-54	1.0	2.0	7.0	2.00	2.00	.050	"	"
953	-54	O.C.V. 0.3	O.C.V. 0.35	P.O.C.V. 1.0	1.8	1.7	.044	Vertical Reservoir	"
954	-54	0.6	1.0	1.8	1.98	1.8	.040	"	"
943	-46	1.5	3.0	5.2	2.02	1.75	--	"Dunk" Test	"
944	-54	2.5	8.5	32.0	1.94	1.67	--	"	HgSO ₄
945	-54	1.15	2.5	4.0	2.01	1.76	--	"	AgCl
946	-52	2.0	3.25	4.3	2.02	1.76	--	"	"
947	-52	1.4	3.25	4.75	2.03	1.75	--	"	"
948	-52	O.C.V. 0.4	O.C.V. 0.55	P.O.C.V. 1.0	--	--	--	"	"
949	-54	0.35	0.85	2.24	1.98	1.70	--	Vertical Reservoir	"
950	-54	0.56	1.16	2.58	1.98	1.70	--	"	"

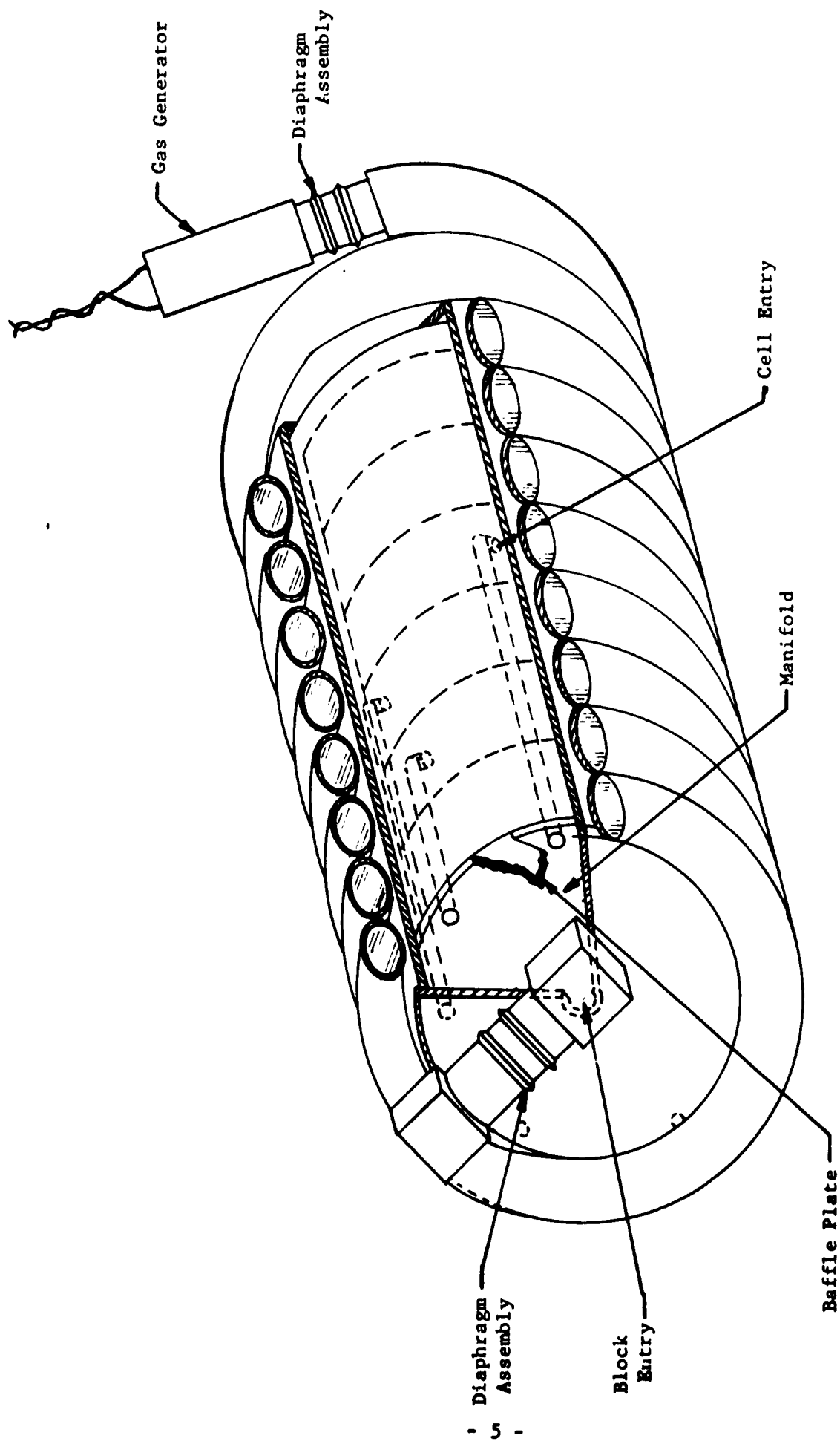


FIGURE NO. 1

CYLINDRICAL SYSTEM HARDWARE DESIGN

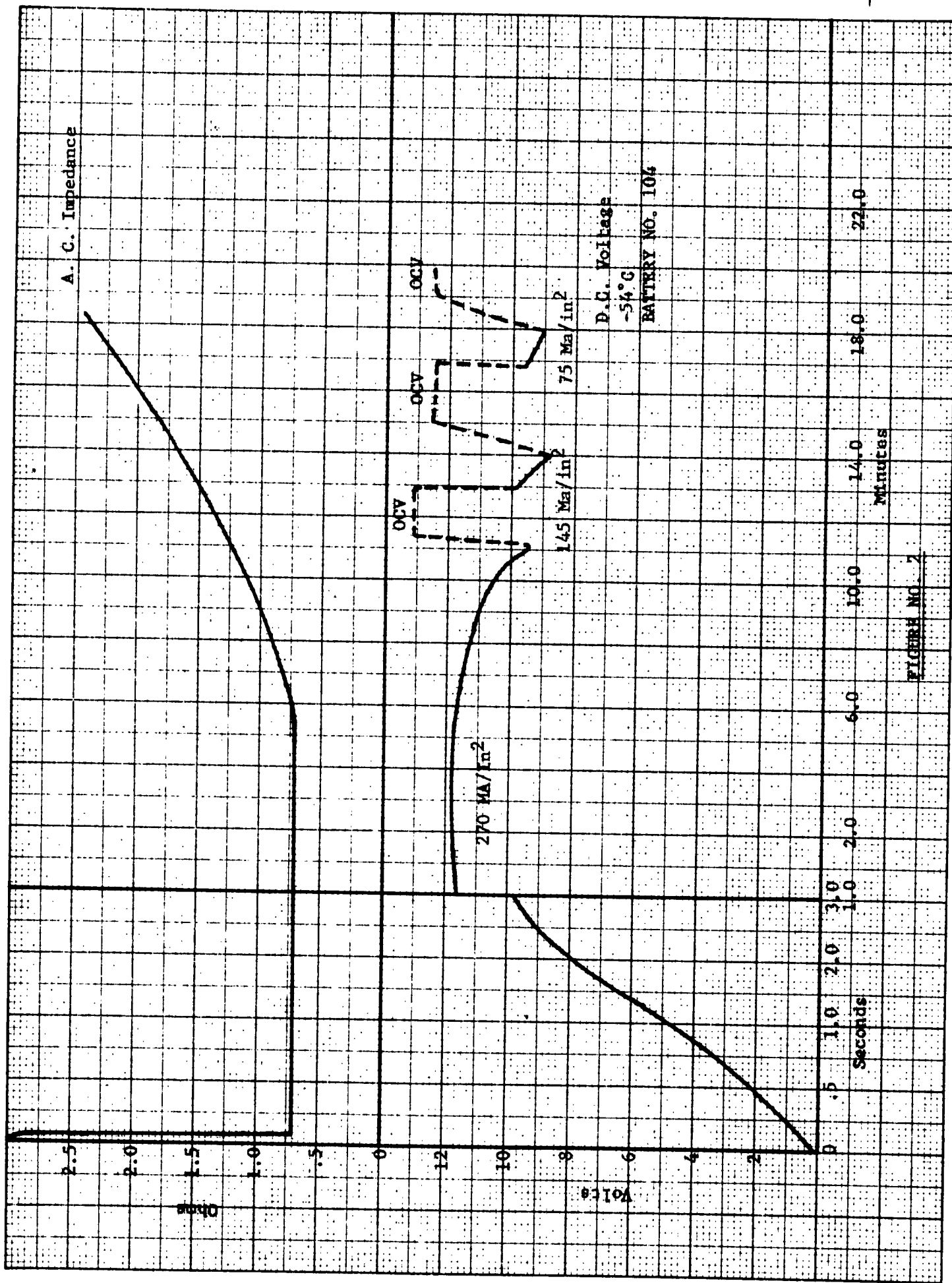


FIGURE NO. 2

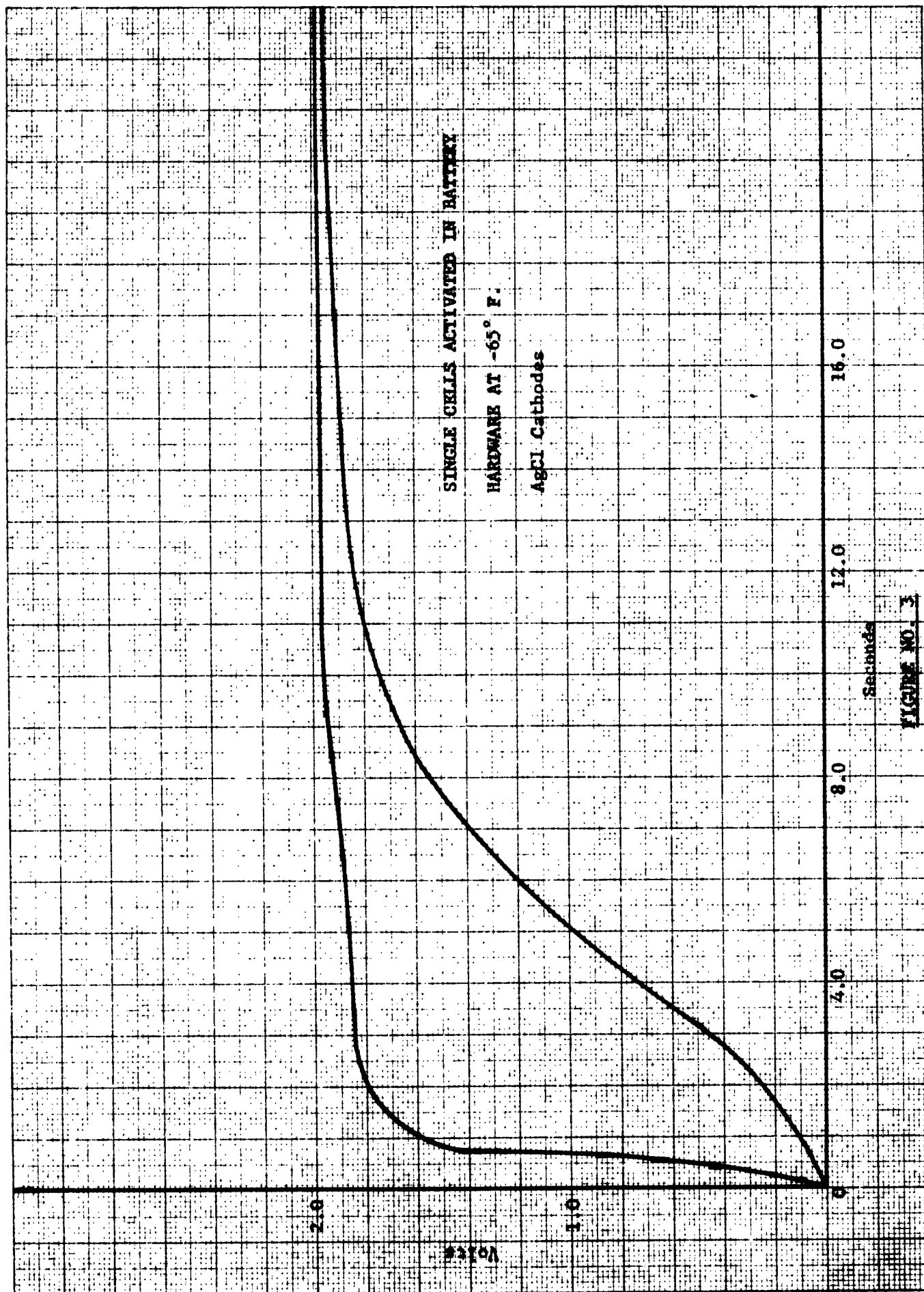
2. Automatically Activated Single Cells

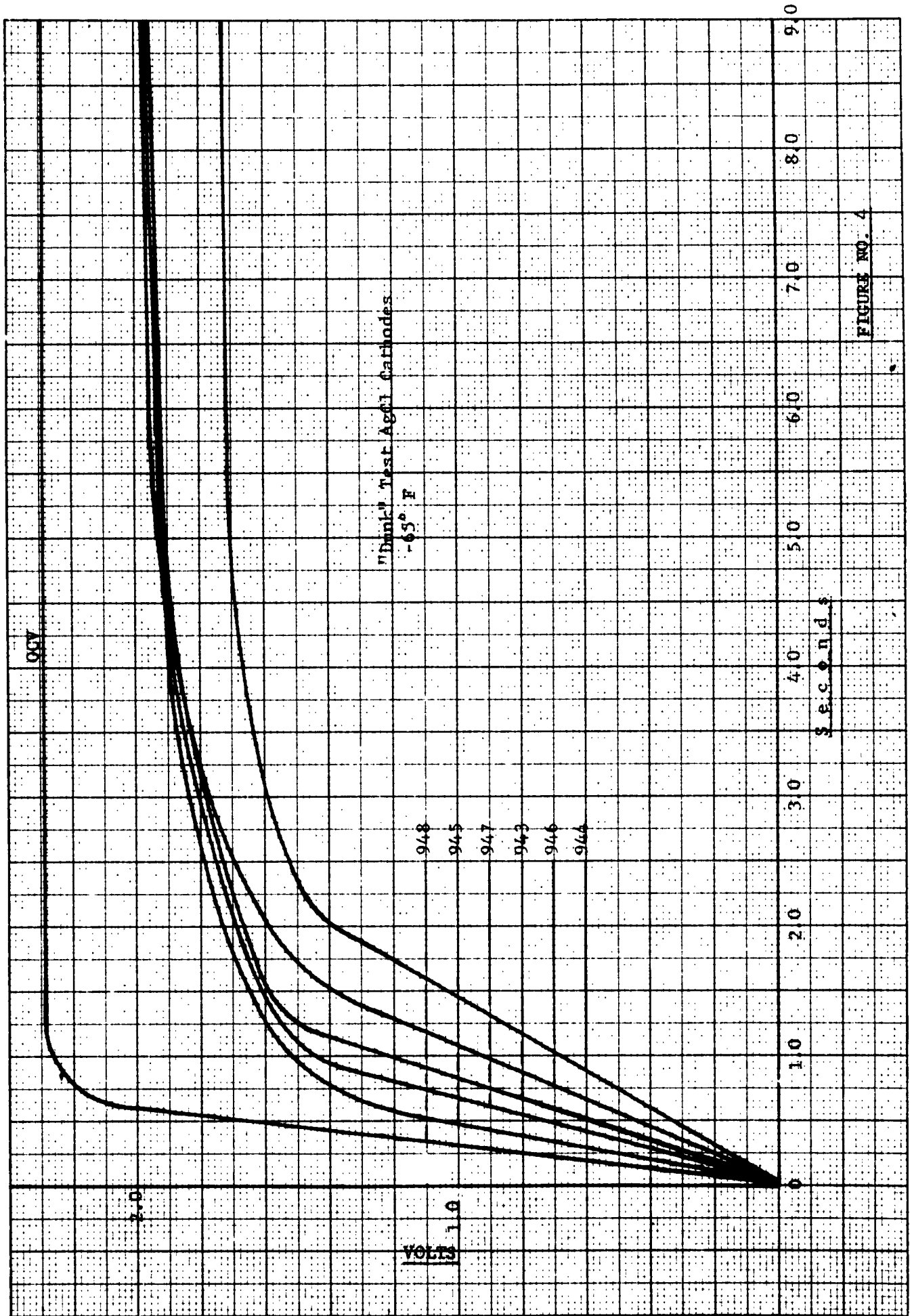
Activation of single cells in battery hardware shows an extreme variation as illustrated in Figure No. 3. This variation in voltage rise time necessitated testing a series of single cells to determine precisely the characteristics of the cell.

The first series of cells studied (Table No. II) were activated using a "dunk" test variation. Cells and electrolyte were pre-cooled to -54°C and activated under load to duplicate battery operation. A Bristol recorder was used to monitor voltage, and the cell was activated by pouring electrolyte over the plates. Figure No. 4 shows these activation curves and indicates a variation in cells plus a serious lag time from 80% peak load voltage to peak voltage.

Further testing was done using a gas generator and vertical reservoir device shown in Figure No. 5. This procedure is superior to the "dunk" test since temperature can be more accurately controlled and pouring electrolyte over the cell is eliminated. Figure No. 6 shows the results of this test. The lag time to initial voltage is due to time necessary for squib gases to reach diaphragm rupture pressure (600 psi). In this device, electrolyte transfer is instantaneous and the 0.1 second voltage rise to 80% P.L.V. shows the capability of the cell disregarding the influence of current leakage in a multicell unit. The most significant observation from these tests is the slow rise from about 80% P.L.V. showing potentially a possible limitation of the cell. In a multicell unit, current leakage depresses the activation curve even further. Peak voltage is not ordinarily obtained until approximately one minute or more in either the single cell or multicell unit.

The cathode was assumed to be the limiting plate. Cell No. 954 was assembled with a reference anode which was monitored during activation with the cell. Figure No. 7 shows steady voltage on the reference anode which confirms the assumption that the cathode is the limiting plate. Any lag from the anode would produce a voltage drop at the reference open circuit plate.





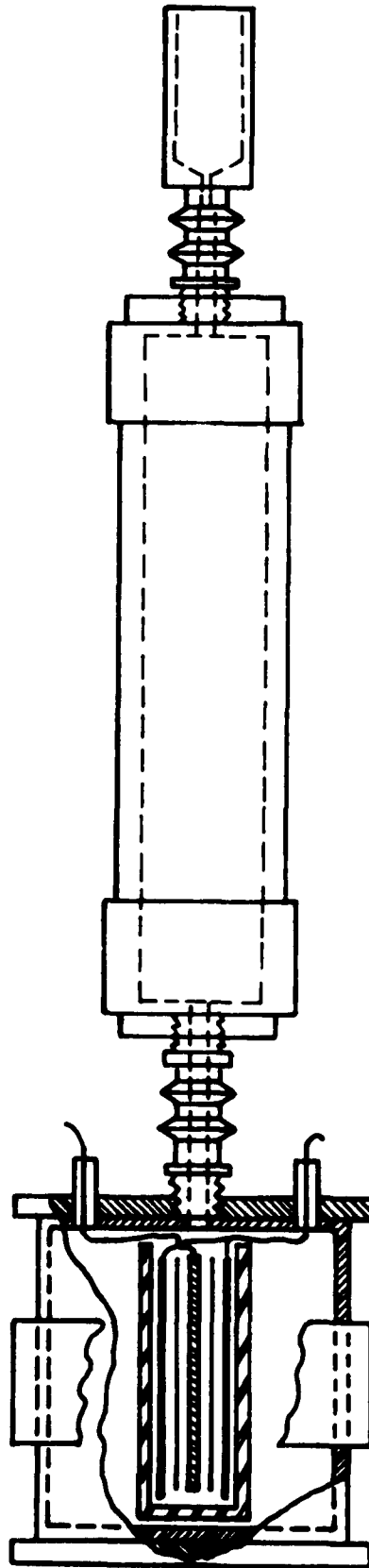
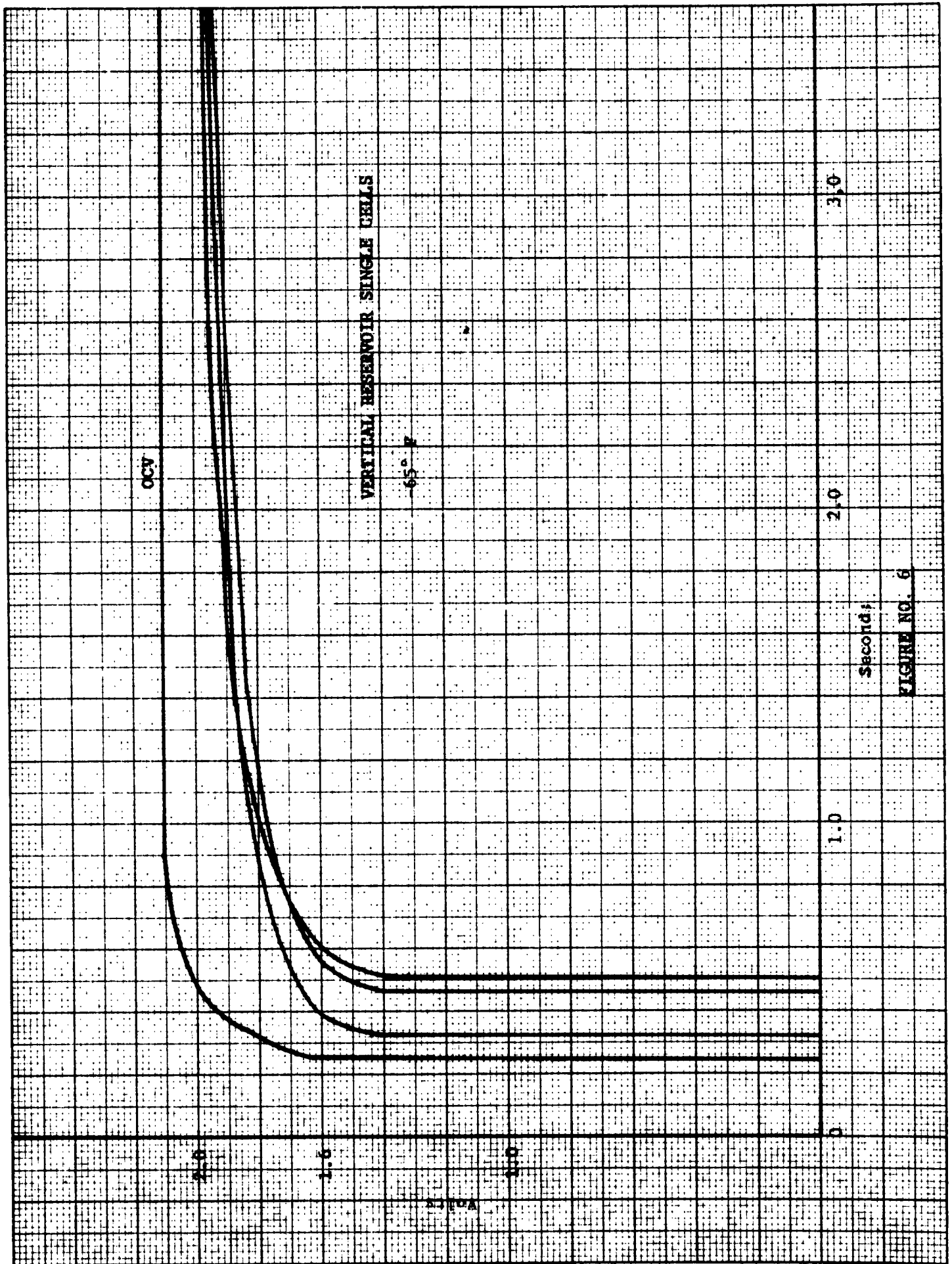
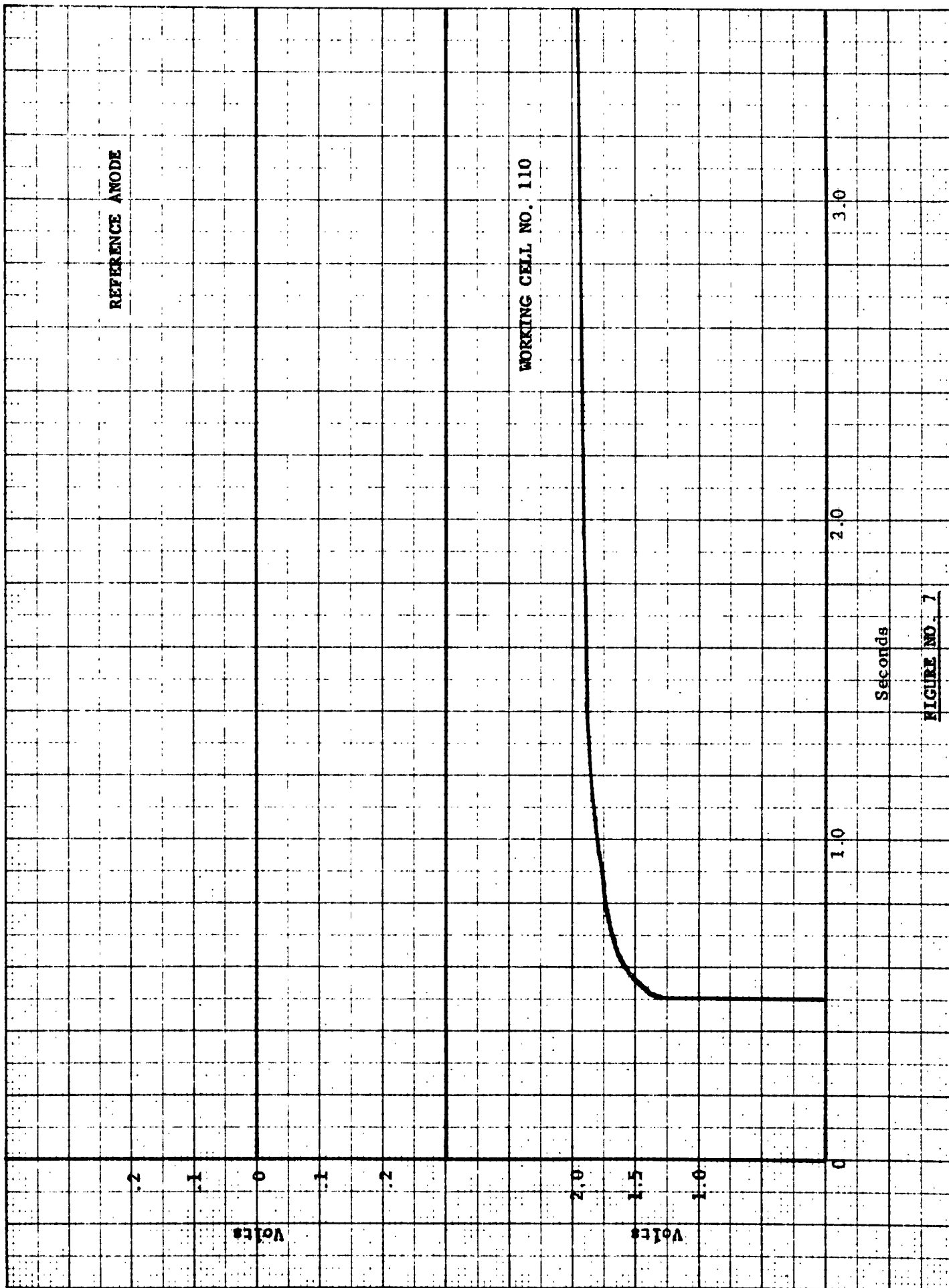


FIGURE NO. 5

VERTICAL RESERVOIR ACTIVATING DEVICE





3. Hardware Design

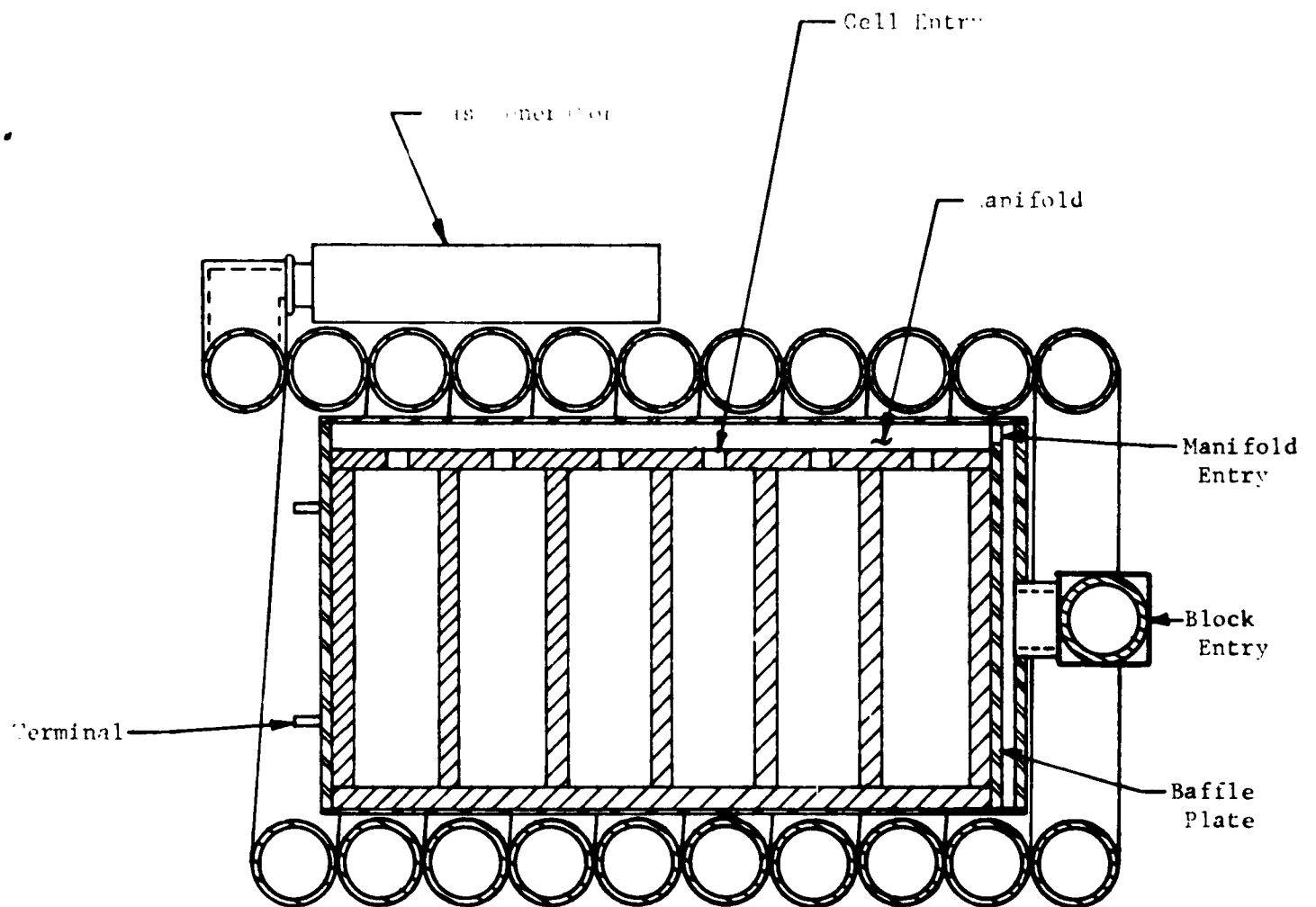
Continued multicell testing was based on the data previously discussed concerning hardware geometry and single cell activation. The use of a cylindrical cell case with cell entries located on a descending spiral as shown in Figure No. 1 was modified to prevent loss of electrolyte by gas displacement. The baffle plate was retained since it cushions activation stresses on the cell block. The cell entries are located on top of the cell block and isolated from the steel container with Teflon tape. Figure No. 6 shows the modified configuration. This design is a compromise which allows good cell filling, maximum gas evolution without electrolyte loss, reduces current leakage, and reduces excess cell block material.

The results of Battery No. 106 indicate the effectiveness of the modification in regard to capacity.

Battery Nos. 101 and 105 were abortive due to hardware ruptures during activation. In Unit No. 101, the rupture occurred in the coil at a silver solder joint which was evidently weakened by excessive heat during fabrication. This method was used to hold the coil firmly during activation and has been successful on prior units. Figure No. 9 shows a typical coil soldered into a compressed position. To prevent future failures, soldering has been eliminated. The coil is presently held firm with adjustable mechanical clamps. Battery No. 105 ruptured the squib holder seal due to air pockets formed in the adhesive. Air pockets in the adhesive can be removed by puncturing with a small wire or similar object prior to hardening or applying vacuum while liquid. The squib holder design is shown in Figure No. 10 and is considered very satisfactory.

Battery Nos. 107 and 108 were assembled using the modified configuration shown in Figure No. 8, and the cell structure was changed to two cathodes and one anode. The purpose of these units was to attempt to improve both activation and capacity by reducing current density on the cathode by discharging the porous electrode from one side and increasing the theoretical cell capacity, respectively. Activation was very good (1 second to 80% P.L.V.), but each unit only ran approximately 11 minutes. The theoretical energy per unit weight of these batteries was approximately five watt-hours per pound, but due to shorting through the separation, the units were only 25.0% efficient; thus, no overall improvement in capacity was obtained. Internal shorting has been a marginal problem with silver chloride cathodes and improvement in this area has been initiated.

The variable in Battery Nos. 106, 107 and 108 was cell structure. The data in Table No. I indicates that activation was improved, and that efficiency was reduced by the cell design. It is believed that some discharge by diffusion from the back of the cathodes actually reduces current density, thus facilitating activation. The reduced capacity efficiency is probably a result of tight cell cavities due to the additional swelling from double cathodes in the same original thickness cell cavity.



CYLINDRICAL SYSTEM HARDWARE DESIGN

FIGURE 10.8

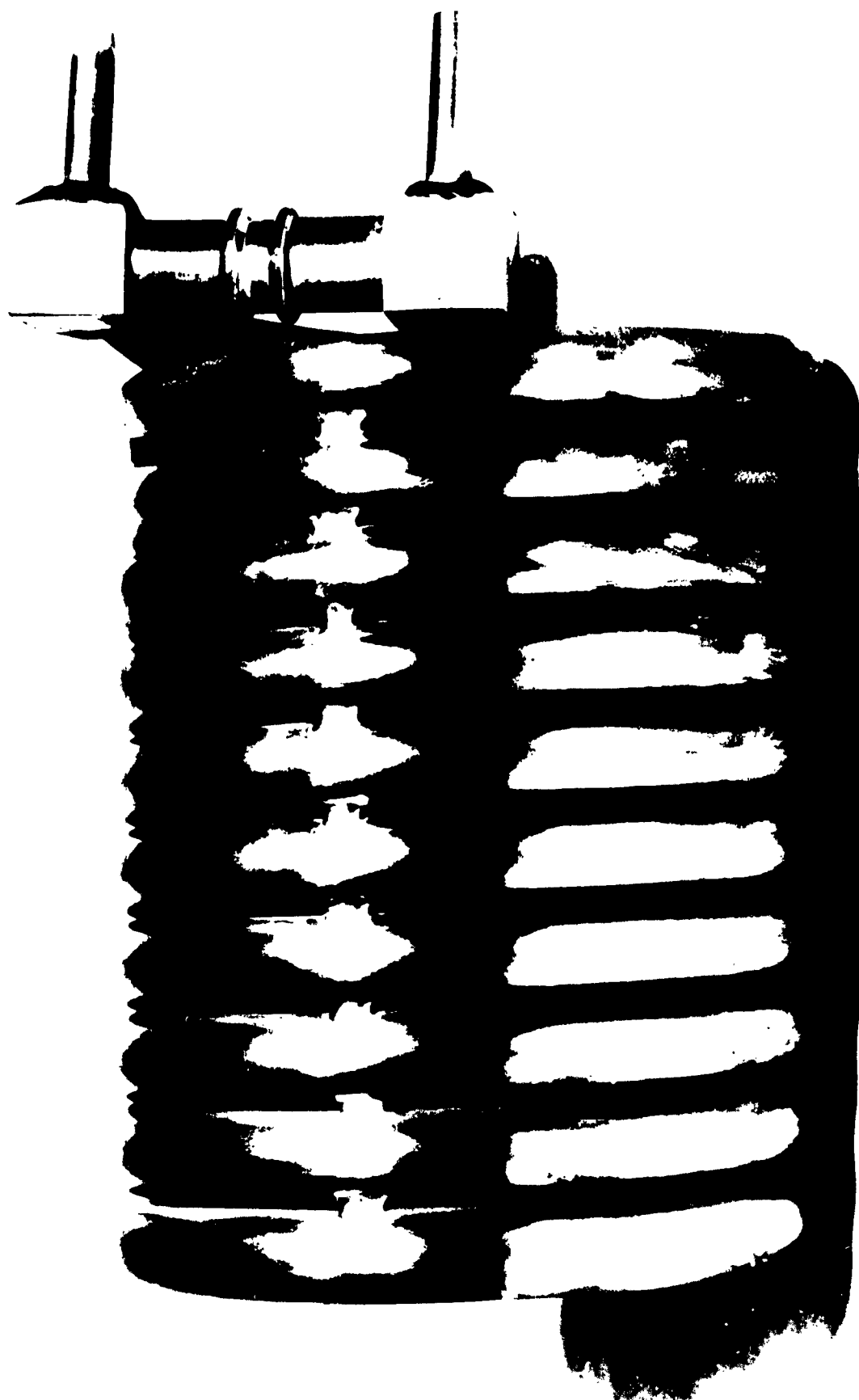
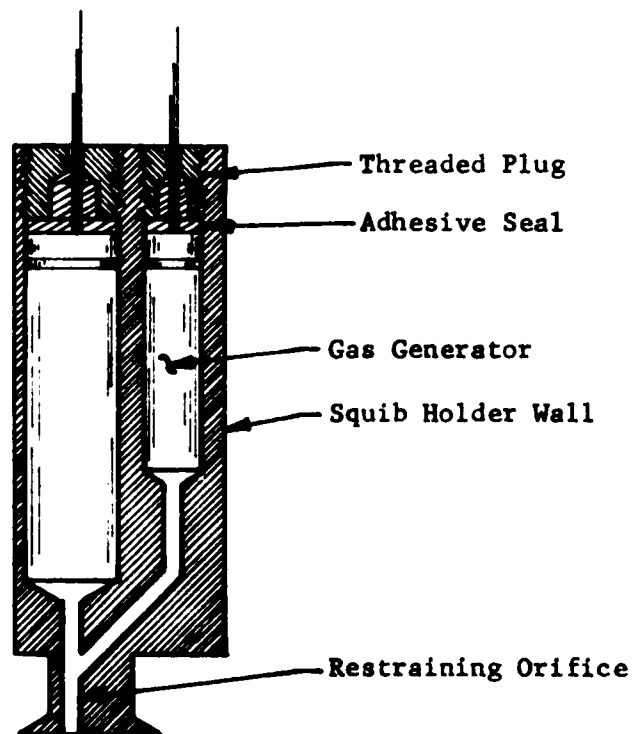


FIGURE NO. 9

COIL SILVER SOLDERED INTO COMPRESSED POSITION



SQUIB HOLDER SHOWING ADHESIVE SEAL

FIGURE NO. 10

4. Capacity Efficiency Study

Several single cell tests were made to investigate the assumptions concerning cell design. Table No. III shows the data from these cells. The attempt to concentrate cells in Battery Nos. 107 and 108 was reasoned to be of limited success because cathode swelling facilitated silver growth and shorting through the separation. Cell Nos. 955 - 958 were designed using various separator materials that would reduce cell thickness. The results indicate no overall gain and Cell Nos. 959 and 960 show the effect of 1/4 inch Vexar and Pellon separation, and the necessity of loose cell construction.

The effect of increased current density discharge is shown by Cell Nos. 961 and 962. Efficiency is not improved and activation is reduced at higher rates. Further evaluation of cell design included using Aldex with Pellon and Vexar separation to retard silver migration in the tighter cell cavities. Cell Nos. 963 and 964 were designed for this study and indicate that activation time is retarded by the Aldex, and efficiency is not significantly improved.

For comparison purposes, several HgSO_4 cells were tested. The cells were constructed using pre-ammoniated HgSO_4 and the results show inconsistent activation times and approximately 25% efficiency, which is typical of pre-ammoniated HgSO_4 cells under these conditions.

From information concerning these single cells, it is concluded that silver chloride cells with 1/4 inch Vexar and Pellon separation in cell cases that allow room for cell swelling are the most efficient structures. Cell Nos. 969 through 971 verify this conclusion, and Battery No. 109 was assembled using this cell design. Explanation of this effect may be the need for free space in which the gas formation from the anode can break the silver chains and disperse the silver powder in the electrolyte. Silver powder dispersed in the electrolyte, and not forming electronically conducting chains would, of course, not result in shorting. Formation of the silver initially is, of course, due to the solubility of silver chloride in the electrolyte, diffusion to the anode, and galvanic precipitation. The necessity of using "soluble" cathode material is discussed more completely under the "Screening Tests" section of this report.

Based on information from single cells as described and shown in Table No. III, Battery No. 109 was fabricated using additional free cell cavity volume. The added volume was accomplished by increasing the cell cavity width slightly (0.03 inch) which does not significantly affect the size or total weight of the system.

The results of Unit No. 109 implicate the necessity of added volume. The unit functioned for approximately 28 minutes at 2.3 amps, yielding 64 ampere-minutes. Activation time was 2.3 seconds to 80% P.L.V.

TABLE NO. III - SINGLE CELL DATA

CELL NO.	ACT. TEMP. °F	ACT. TIME (Seconds) 80% PLV	PEAK LOAD VOLTS	MAX. AMPS	CURRENT DENSITY MA/in. ²	THEOR. AMP. MIN.	TOTAL MIN. TO 30% PLV	% EFF. TO 80% PLV CUT-OFF	END O.C.V.	SEPARATOR MATERIALS	NO. OF CATHODES & MATERIAL	NO. OF ANODES	CELL CASE
955	-30	----	1.90	1.57	270	100.0	6.5	9.0	1.4	1/8 inch Vexar	2 AgCl	1	Loose
956	"	1.5	2.00	1.66	"	"	16.0	22.4	1.8	Micro-porous Rubber	"	"	"
957	"	1.4	2.10	1.67	"	"	8.0	12.0	2.0	Pellon	"	"	"
958	"	1.1	1.94	1.61	"	"	1.5	2.25	1.4	Micro-porous Rubber	"	"	"
959	"	0.8	2.0	1.67	"	"	37.0	57.6	2.08	Pellon	"	"	"
960	"	0.75	1.99	1.70	"	"	4.5	7.6	0.9	1/4 inch Vexar	"	"	Tight
961	"	0.75	1.88	3.4	600	"	8.0	25.0	2.05	Pellon	"	"	Loose
962	"	3.25	1.72	5.0	950	"	6.0	27.0	2.20	1/4 inch Vexar	"	"	"
963	"	1.4	1.97	1.67	270	50.0	8.0	24.8	1.70	Pellon	"	"	"
964	"	1.25	1.98	1.68	"	"	15.5	48.0	2.04	1/4 inch Vexar	1 AgCl	2	Tight
965	"	1.20	1.96	1.64	"	34.2	6.5	29.9	2.22	Pellon - Aldex	"	"	"
966	"	2.1	1.90	1.60	"	32.2	5.0	24.2	2.22	Pellon	1 HgSO ₄	"	"
967	"	1.4	1.96	1.65	"	35.1	5.25	23.2	2.22	Vexar	"	"	"
968	"	1.1	1.98	1.68	"	31.4	6.5	31.4	2.20	Pellon	"	"	"
969	"	6.7	2.05	1.74	"	50.0	25.0	80.0	2.10	Vexar	1 AgCl	"	"
970	"	0.9	2.02	1.70	"	"	24.0	76.8	2.02	Pellon	"	"	"
971	"	1.2	2.04	1.75	"	"	24.0	75.8	2.10	Vexar	"	"	"

5. Cathode Orientation Study

Following experimentally from the previous multicell and single plate information developed, it was decided to determine the effect of collector grid position within the cathode plate. It is already indicated that use of two positives and one anode yields more consistent and shorter activation time. The capacity is reduced by premature shorting when the cell cavity thickness is not sufficient to provide free space within the large pore areas of the Vexar net. Therefore, for the 0.5 inch cell thickness, the same quantity of silver chloride should be distributed over two cathodes in place of one. Following previous procedure, single cell tests (Table No. IV) were used to pilot the battery fabrication.

From these data, activation times appear appreciably shorter when the amount of silver chloride over the collector on the anode (discharge) side is appreciable (not 0%), but less than 100%. This indicates wherein the single cathode cell configuration contained a contribution to longer activation time; that is, time involved for the plate to come up to at least 80% of peak voltage due to thickness. This might involve the time required for silver chloride wetting, solution and discharge. The A.C. impedance value would probably be substantially the same whether the D.C. mechanism is operable or not, since the A.C. impedance would be low when the electrolyte reaches and wets the collector screen. This effect of the A.C. path not necessarily following the same as for the D.C. mechanism casts doubt on the interpretation of the A.C. impedance.

It is concluded that use of 25% of each plate active material toward the anode, use of the same total quantity distributed over two cathodes, use of one anode, and the cavity remaining the same would potentially improve activation time and provide additional assurance that premature shorting will not decrease capacity.

TABLE NO. IV
SINGLE CELL TESTS INVESTIGATING
ORIENTATION OF CATHODE PLATES IN CELLS

CELL NO.	ACT. TEMP. °F	ACTIVATION TIME (Sec.) TO			PLV	MAX. AMP.	MINUTES TO 80% PLV CUT-OFF	THEOR. AMP. MIN.	TOTAL AMP. MIN.	% EFF. TO 80% PLV	LOCATION OF AgCl	NO. POS.	NO. NEG.	CELL CAVITY
		80% PLV	90% PLV	1.9 V.										
973	-30	0.75	1.20	2.1	2.07	1.74	18.0	58.0	28.8	49.6	50% next to anode	2	1	0.5
974	"	0.60	1.50	3.0	2.06	1.74	14.5	58.0	23.3	40.0	0.0% next to anode	2	1	0.5
975	"	1.00	1.50	2.8	2.04	1.74	16.0	58.0	25.6	44.0	100.0% next to anode	2	1	0.5
976	"	1.20	2.00	3.0	2.06	1.75	17.0	58.0	27.4	47.3	75.0% next to anode	2	1	0.5
977	"	0.70	1.10	1.7	1.99	1.71	23.0	58.0	35.6	61.4	25.0% next to anode	2	1	0.5
979	"	0.60	-----	---	1.99	1.68	21.3	58.0	33.0	57.0	0.0% next to anode	2	1	0.5
980	"	0.90	-----	---	2.03	1.72	21.5	58.0	32.5	56.0	100.0% next to anode	2	1	0.5
981	"	1.10	1.80	3.7	2.03	1.73	16.0	58.0	24.7	42.5	75.0% next to anode	2	1	0.5
982	"	0.60	1.10	2.8	2.02	1.72	23.5	58.0	36.9	63.5	25.0% next to anode	2	1	0.5
983	"	0.70	1.10	1.6	2.08	1.78	13.5	29.0	22.7	78.5	50.0% next to anode	1	2	0.281
984	"	1.60	2.60	3.9	2.05	1.75	13.0	58.0	20.5	35.5	50.0% next to anode	2	1	0.281

B. Hardware Fabrication

Some difficulty has been experienced with heliarc welding the thin walled coil (0.020 inch). Pin hole leaks at the joint between coil and diaphragm assembly, which can be detected prior to filling, are evidently due to the nature of the configuration. The underside of this joint is difficult to reach with the welding tip and consequently, good welding technique such as maintaining a 90 degree angle between welding tip and surface material is impossible to follow. The coil and other hardware pieces were analyzed for impurities which might be contributing to welding difficulty. The analysis is shown in Table No. V and indicates no impurities that would create this trouble.

Patching these leaks by heliarc welding is difficult since the heat necessary to flow the previously welded joint tends to burn additional holes in the thin walled coil. When welding the leaks was unsuccessful, silver or soft soldering has been used. This technique of patching is very successful and has been considered for possible production fabrication purposes. Figure No. 11 shows test joints that were pressurized hydraulically to 4000 psi. The tin soldered sweat type joints held firmly, thus indicating the adaptability to the ammonia system. If heliarc welding remains a problem, the soldering technique may be used.

Figure No. 12 shows photomicrographs of two pieces of silver solder "before" and "after" exposure to the ammonia electrolyte for 30 days at 73° C. The exposed piece was soldered to a section of mild steel to produce galvanic corrosion if any might exist. No apparent corrosion was seen.

TABLE NO. V

CHEMICAL ANALYSIS OF STEELS

<u>HARDWARE</u>	<u>% Carbon</u>	<u>% Sulfur</u>	<u>% Phosphorous</u>	<u>% Manganese</u>
Coil	.08-.18	.055	.05	0.30-0.60
Diaphragm Adapters, etc.	.15-.20	.050	.04	0.60-0.90

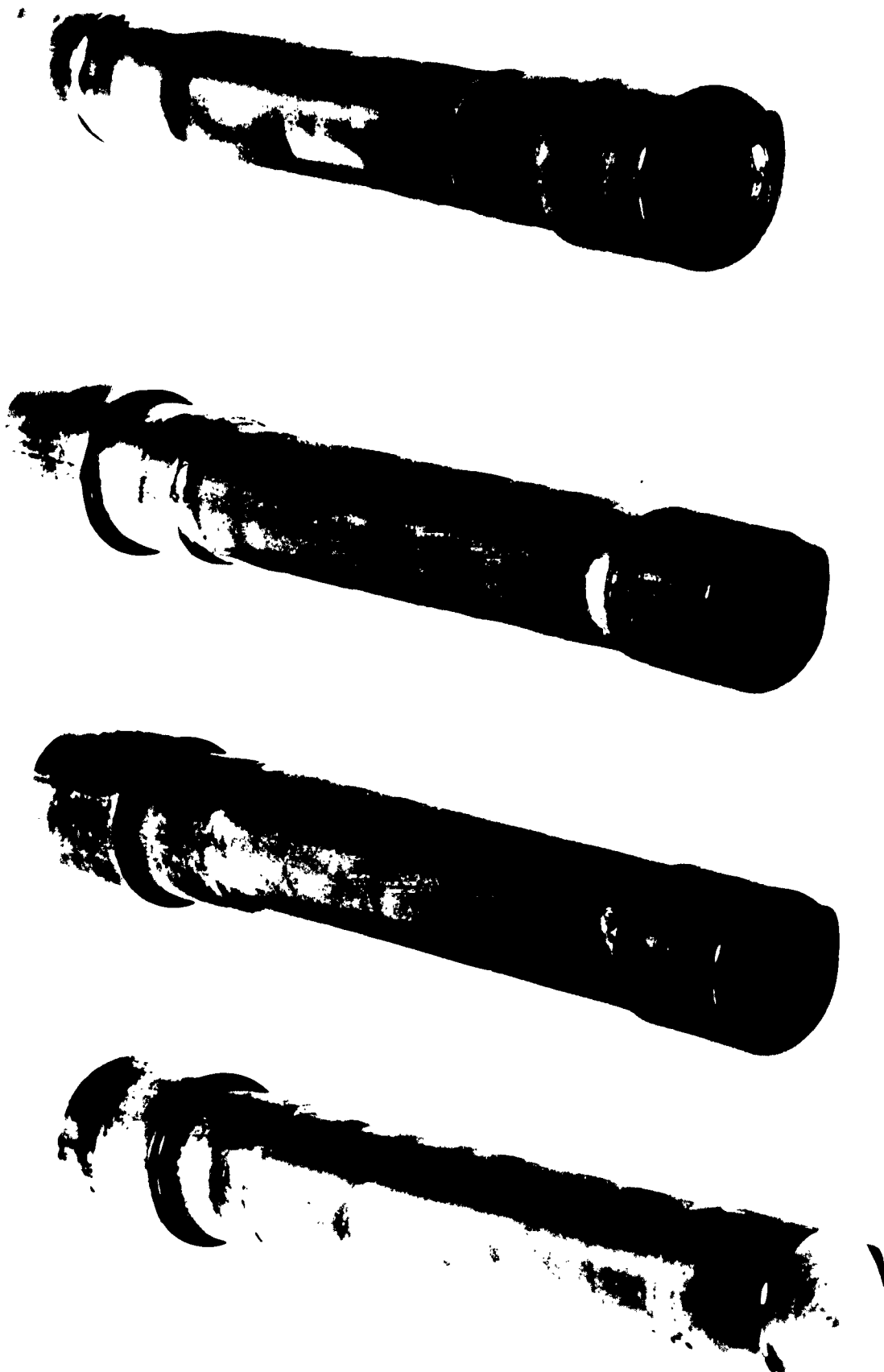


FIGURE NO. 11
SOFT SOLDERED SWEAT TYPE TEST JOINTS



FIGURE NO. 12

SILVER SOLDER EXPOSED TO CORROSION
EFFECTS OF ELECTROLYTE

C. Screening Tests

Screening tests were carried out on a limited scale for those materials shown in Table No. VI. Comparing the results of the two silver salts (along with those known for silver chloride), point up the importance of solubility of cathode as a requisite for appreciable cathode reduction during discharge. The amount of "sludge" found with the silver carbonate was essentially nil as compared with that found in the two "soluble" salts that yield practical capacities at high rates. It, therefore, follows to decrease the solution and diffusion of the soluble cathode active materials, a restraining separator material conductive for ions, but which would restrict fluid flow, is necessary. The long duration discharges reported in the next section of this report are explained on this basis.

Reasoning by analogy, and the screen test confirms, the silver sulfate appears to be a potential new high rate cathode active material.

D. Ammoniation of Silver Chloride

Since silver chloride offers so much possibility, it is pertinent to obtain additional comparable data to that for mercury sulfate. Previous data on ammoniation of HgSO_4 were obtained by first pressurizing (110 to 120 psig) at room temperature for approximately 24 hours, releasing the pressure, weighing immediately, and again after equilibrium has been established at atmospheric pressure (NH_3). According to Bjerrum (Chem. Rev. 46, 381 - 401, 1950), the free energy for ammoniation of the silver ion indicates a lower state (higher vapor pressure) than the mercury ion. Two tests were carried out in which the first appeared to contain liquid, and may be of higher weight due to eutectic formation rather than gas reaction to form the ammoniate. The first test showed compound formula $\text{AgCl} \cdot 1.39 \text{ NH}_3$, and the second, $\text{AgCl} \cdot 1.02 \text{ NH}_3$. Either are markedly less than the corresponding mercury sulfate ammoniate previously reported. Thus, the heat of ammoniation is probably much smaller and overheating problems of boiling electrolyte would not be so serious.

TABLE NO. VI

SCREENING TESTS

CELL NO.	CATHODE 2.25 g	OCV	C.C.V.		AMP.	MIN.	C.C.V.		AMP.	MIN.	O.C.V.		ACZ, Ohm	
			Start	Finish			Start	Finish			Start	Finish	Start	Finish
986	Ag ₂ SO ₄	2.29	2.07	1.90	1.7	(1) 10.3	1.90	1.65	1.7	(1) 1.5	---	---	0.12	0.097
987	Ag ₂ CO ₃	2.21	1.94	1.90	1.7	.08	1.90	1.00	.20	2.0	---	---	0.080	0.080
988	PbO ₂	2.65	2.02	1.90	0.10	1.0	1.90	1.06	1.7	1.5	---	---	0.20	0.20
Control	AgCl	2.26	2.04	1.90	1.7	(3) 13.0	1.90	1.63	1.7	(3) 9.0	---	---		

(1) Coulometric efficiency total: 76% at 1.90 volts and 86% at 1.65 volts (80% cutoff).

(2) Did not recover in two minutes.

(3) Coulometric efficiency total: 40% at 1.90 volts and 67% at 1.63 volts (80% cutoff).

E. Low-Rate Long-Duration

Two phases of this problem were investigated during the interval. Previous tests have established the stability of cellophane in liquid ammonia.

A comparison with Permion 300 is pertinent due to the appreciable voltage loss from high internal resistance and the relative low cost of the cellophane. Table No. VII shows an additional test with Permion and two tests with cellophane. On Cell No. 973, the automatic recorder failed to print, so the capacity could only be shown as a maximum possible. Cell No. 985 definitely showed the poor performance of the cellophane. "Post mortem" examination showed copious silver present on both sides of the separator and on the magnesium. Cells are shown in Figure Nos. 13 and 14.

Tests of gassing of magnesium on +73° C stand in electrolyte has been made by weighing the test pieces after the exposure. Table No. VIII shows the analyses for the various magnesium samples used. Table No. IX shows the loss in weight for the various tests and test pieces. These data show the purer the magnesium, the less the loss. Control testing of corrosion plus electrochemical anodic action will be carried on to correlate these data.

TABLE NO. VII

LOW-RATE SINGLE CELL

CELL NO.	SEPARATION	TEMP. °C	START O.C.V.	APPR. ACT. TIME -Min.-	START VOLT.	END VOLT.	CURRENT -MA-	TIME -hrs.-	START PRESS.	END PRESS.	% THEOR.
949	Permion 300	-30	2.20	5 *	1.87	1.40	10	85	0	55	83.6
973	One Layer Cellophane PUDO 300	-30	2.30	5 *	2.28	0.34	10	15 *	0	60	14.7*
985	3 Layers Cellophane PUDO 600	-30	2.14	5 *	2.09	1.66	10	3	0	60	2.95

* Less than

NOTES: Rate - 1.7 MA/sq.in. = 10 MA.

All cells fused AgCl sandwiched between 410 x 0.0005" thick expanded pure silver pressed.
Theoretical - 11.1 AM/g.



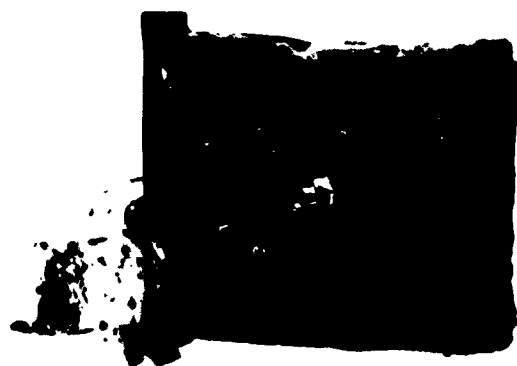
CELL NO. 249

FIGURE NO. 13

PERMION SEPARATOR AND CATHODE (APART)
AFTER DISCHARGE



CELL NO. 985



CELL NO. 935



FIGURE NO. 14
CELLOPHANE SEPARATOR CELL
SHOWING SILVER DEPOSITED

TABLE NO. VIII

ANALYSES OF VARIOUS MAGNESIUM ANODES STUDIED

DESCRIPTION	% Si	% Mn	% Fe	% Al	% Cu	% Ca	% Pb	% Ag	% Zn
Mg Strips, pure Mg sample	.0005	.01	.002	.001	.0001	.005	.000	.000	.0
Mg Strips, pure Mg sample prod.	.001	.001	.002	.004	.0002	.002	.000	.000	.0
Mg Strips, J-1	.04	.004	.001	.1	.0005	.002	.002	.0004	.5
Mg Strips, AZ 31B sample	.02	.001	.001	.04	.0005	.002	.001	.0002	1.0
Mg Strips, AZ 31B prod.	.02	.002	.001	.06	.0005	.00005	.001	.0002	1.0
Mg Strips, AZ 61B prod.	.02	.004	.001	.2	.0005	.001	.002	.0002	1.0
Sublimed Magnesium	.01	.00005	.0005	.001	.0001	.002	.000	.0001	.005

TABLE NO. IX

WEIGHT LOSS ON EXPOSURE
TO ELECTROLYTE
 (approximately 24 hours and 73° C)

MATERIAL	WT. LOSS - G/CM ² x 10 ⁻³				
	Test 1	Test 2	Test 3 *	Test 4	Test 5 *
Prod. Pure Mg	2.52	1.03	4.17	5.85	6.00
Prod. Pure Mg	2.52	1.50			
J-1 Alloy	6.80				
J-1 Alloy	7.11				
AZ-61A	6.41		5.45		
AZ-61A	6.62				
AZ-31B (Prod.)		1.69	5.77		
AZ-31B (Prod.)		2.43			
94197 (3.5% Hg)		2.20			
94197 (3.5% Hg)		2.74			
AZ-31B (new ship.)		5.15			
AZ-31B (new ship.)		3.18			
Pure Mg (new ship.)		4.94			
Pure Mg (new ship.)		0.84			
Dist. Mg				3.38	4.09

* Free circulation; all other tests held in D4/4 Vexar.

III. CONCLUSIONS

1. Gas formation in multicell units limits the system in regard to orientation and hardware design.
2. Silver chloride cathodes are capable of 0.1 second activation to 80% P.L.V., but vary considerably in reaching 1.9 volts.
3. The cathode is the limiting plate regarding activation time.
4. Double cathode and single anode cell structure improves cold activation.
5. Efficiency of multicell units is affected significantly by free volume of cell cavities.
6. Discharge at higher current densities (over 600 MA/in.²) increases activation time and does not improve efficiency.
7. As a substitute for heliarc welding, silver and pure tin soft soldering has possible application.
8. Silver sulfate has good possibility as a high rate active material.
9. Silver carbonate and lead peroxide were found to be poor high rate cathode active materials. Solubility was very low, although lead peroxide had the highest open circuit voltage observed thus far (2.65).
10. Moles of ammoniation for silver chloride were shown to be much less than mercury sulfate under the same test conditions.
11. Although cellophane structurally was stable, three layers of PUDO-600 permitted silver diffusion, precipitation and shorting in three hours, as compared to one thickness of Permion 300 for which shorting was not observed in about 100 hours.

IV. IDENTIFICATION OF KEY PERSONNEL

Following is a list of key personnel who worked on this project during the first quarter, their job titles, and the number of hours contributed:

<u>NAME</u>	<u>TITLE</u>	<u>MAN-HOURS</u>
Doan, D. J.	Project Supervisor	142
Wood, L. R.	Project Engineer	464
Dittmann, J. F.	Engineering Supervisor	34
Witherspoon, S.	Senior Engineer	139
Sieglinger, G. F.	Senior Engineer	20
Costley, W. R.	Technician	58
Graham, J. L.	Technician	42
Kernohan, T.	Technician	<u>12</u>
TOTAL		911

APPENDIX

AD _____ Accession No. _____ The Eagle-Picher Co., Joplin, Mo. RESEARCH ON AMMONIA BATTERY SYSTEM. D. Doan - L. Wood Report No. 9, First Quarterly Progress Report, Contract No. DA-36-039-sc-89188 (Continuation of Contract No. DA-36-039-sc-85396), DA Proj. No. 3A99-09-001, July 1, 1962 to September 30, 1962, Date of Report October 30, 1962, 34 pp, illustrations, tables. A better understanding has been obtained of the factors contributing to the previously observed slower practical activation of the -53°C batteries as compared to single cells under ideal conditions. Tests indicate a larger percentage of the batteries activate in less than 3 seconds, with several about 1 second. A new high-rate cathode possibility (silver sulfate) has been found. Permion 300 separator has been found much better than cellophane for limiting the silver diffusion in the low-rate, long-duration application.	Unclassified 1. Activation time 2. High-rate cathode 3. Permion 300 separator
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