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RESEARCH INVESTIGATIONS LEADING TO THE DEVELOPMENT AND EVALUATION OF A CADMIUM - SILVER OXIDE BATTERY HAVING A HERMETICALLY SEALED CONSTRUCTION

OCTOBER 1, 1962 THROUGH DECEMBER 31, 1962

REPORT NO. 10

SECOND QUARTERLY PROGRESS REPORT

CONTRACT NO. DA-36-039-sc-89065
(Continuation of Contract No. DA-36-039-sc-85370)

THE EAGLE-PICHER COMPANY
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CONTRACT NO. DA-36-039-sc-89065
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Signal Corps Technical Requirements SCL-7537
dated December 11, 1959

DEPARTMENT OF THE ARMY TASK NUMBER 3A99-09-002

SECOND QUARTERLY PROGRESS REPORT
OCTOBER 1, 1962 THROUGH DECEMBER 31, 1962

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

THE EAGLE-PICHER COMPANY
COUPLES DEPARTMENT
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DATE OF REPORT: January 31, 1963

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TABLES

I. PURPOSE

The purpose of this contract is to develop and evaluate a hermetically sealed, readily reproduced, rechargeable cadmium - silver oxide battery having a high level of reliability and performance.

II. ABSTRACT

Development work is being directed toward the attainment of a reliable sealed cadmium - silver oxide battery. Effort is directed toward the development of both cadmium - divalent silver oxide and cadmium - monovalent silver oxide cells, and studying the performance characteristics of each system.

Of the major problems encountered, probably the most important is the relative inconsistency of available separator materials, including both areal resistance and cycle life measurements. Continuous overcharge at rates up to C/10 have been attained, but only at a loss in capacity efficiency of approximately 35%. Problems of this nature must be surmounted before reliable cadmium - silver oxide cells can be produced.

III. REFERENCES AND CONFERENCES

A. References

1. Final Report, "Research Investigations Leading to the Development and Evaluation of a Cadmium - Silver Oxide Battery Having a Hermetically Sealed Construction", (1962) The Eagle-Picher Company, Contract No. DA-36-039-sc-85370

2. Final Report, "Effects of Ambient Temperature on Performance Characteristics of Vented Nickel - Cadmium Battery, BB-422/U", (1962) The Eagle-Picher Company, Contract No. DA-36-039-sc-89122

B. Conferences

Date	Location	USAELRDL Representatives Present	Eagle-Picher Representatives Present
10-11-1962	Ft. Monmouth, New Jersey	H. Mandel P. Rappaport A. Frink	E. M. Morse J. K. Wilson
12-5-1962	Joplin, Missouri	A. Frink	J. F. Dittmann J. K. Wilson

The purpose of the conference of October 11, 1962 was to discuss the progress made during the first quarter and the plans for work to be done during the second quarter.

The purpose of the conference of December 5, 1962 was to discuss the First Quarterly Progress Report, the work done during the second quarter, and the plans for work to be done during the third quarter.

IV. FACTUAL DATA AND DISCUSSION

A. Improvements in Type Insulation

Because of the great importance of the film type separation in the cadmium - silver oxide system, a large portion of the work devoted to this project will be conducted in the area of the procurement and evaluation of membranes. The work during this quarter was divided into two areas:

(1) Electrical resistance of membranes

(2) Accelerated membrane evaluation.

1. Electrical Resistance of Membranes

During the first quarter, electrical resistance of membranes was measured at both 0° F and +80° F. This task involved measurements of various Permion membranes and cellophane. During this quarter, these measurements were extended to +120° F. Table No. IV contains data reported in the First Quarterly Progress Report extended to include +120° F data. It is indicated in the table that resistance figures on Permion 305 and Cellophane 300 could not be calculated because of the inaccuracy of the equipment at extremely low resistance values. No resistance increase was noted between the blank and the actual membrane. The ohm-square inch per degree, or the resistance change with temperature, has been recalculated to include the temperature range of 0° F to +120° F (where possible), and is shown in the table. Cellophane 600 is again apparently the best membrane concerning resistance change over a wide temperature range, and again Permion 302 is the membrane whose resistance is most adversely affected by changing temperature.

TABLE NO. IV

ELECTRICAL RESISTANCE OF

MEMBRANES, ONE LAYER (8.98 SQ.IN.)

TYPE MEMBRANE	POLYETHYLENE LOW DENSITY WEAK ACID P-300			POLYETHYLENE HIGH DENSITY WEAK ACID P-302			POLYETHYLENE LOW DENSITY STRONG ACID P-305			POLYETHYLENE HIGH DENSITY STRONG ACID P-307			REGENERATED CELLULOSE C-300			REGENERATED CELLULOSE C-600		
Wet Thickness, in.	.0025	.0025	.0025	.0015	.0015	.0015	.0025	.0025	.0025	.0015	.0015	.0015	.003	.003	.003	.0045	.0045	.0045
Electrical Resistance, ohm.-in.^{2}	80°F	0°F	120°F	80°F	0°F	120°F	80°F	0°F	120°F	80°F	0°F	120°F	80°F	0°F	120°F	80°F	0°F	120°F
1 hr. Soak	.0171	.081	.0036	.0171	.206	.00104	.0116	.036	**	*	.081	.0072	.0054	.027	**	.0116	.027	.0072
24 hr. Soak	.0116	.081	.0036	.0171	.206	.00104	.0116	.036	**	*	.081	.0072	.0054	.027	**	.0116	.027	.0072
Ohm.-in.^{2} per degree	6.45×10^{-4}			1.71×10^{-3}			3.05×10^{-4}			6.07×10^{-4}			2.70×10^{-4}			1.65×10^{-4}		

* This membrane was not available at time of test.

** This resistance was so small, it could not be determined with our equipment.

2. Accelerated Membrane Evaluation

Evaluation of multiple layers of membranes with and without nylon by cycling continued during this quarter. Table No. V, (Table No. II of the First Quarterly Progress Report extended) discloses the completion of the cycling test of two layers of membrane and two layers of membrane with nylon. Noteworthy inconsistencies appear in this table, the most obvious of which are found in the case of Cellophane 600 and Permion 305. These two membranes yielded no capacity increase with the addition of nylon, and a fewer number of cycles was actually observed in the case of Cellophane 600. These discrepancies or inconsistencies will be discussed in more detail later in this section.

Again, Cellophane 300 with nylon has yielded the greatest cycle life of the membranes tested. It was followed closely by Permion 1010-X with nylon, which yielded 686 cycles, a total of 94.6% of the cycles yielded by Cellophane 300.

In conclusion, it is emphasized that while this test is in no way a quantitative analysis of the cycle life of the membranes, it does yield indications of relative cycle life, and if enough tests of this nature are performed, an overall average should indicate the most suitable membrane available for a long cycle life cadmium - silver oxide battery. This test, coupled with electrical resistance tests, should also point out flaws in the physical nature of membranes which would be an aid in the development of future membranes.

TABLE NO. V

(Table No. II of First
Quarterly Progress Report extended)

ACCELERATED SEPARATION CYCLING TEST SUMMARY
(TWO LAYERS)

	WITHOUT NYLON	WITH NYLON
Permion 300	Failed between Cycles 43 - 63	Failed between Cycles 321 - 344
Permion 302	Failed between Cycles 116 - 133	Failed between Cycles 383 - 408
Permion 305	Failed after 520	Failed after 520
Cello. 300	Failed between Cycles 116 - 133	Failed after 715 - 725
Cello. 600	Failed between Cycles 383 - 408	Failed between Cycles 116 - 133 *
P-1010-X **	Was not run without Nylon	Failed after 686

* This poor showing is thought to be due to a pin hole or tear in the membrane. Judging from the improvement in other separators with nylon, this should still be running.

** Permion 1010-X is an experimental teflon film produced by R.A.I.

As indicated in the Schedule of Work section of the First Quarterly Progress Report, a series of test cells was next evaluated containing three layers of membranes. These cells were cycled automatically at a frequency of 70 minutes, a 5-ampere discharge for 8.5 minutes and a charge of 0.762 ampere for 61.5 minutes. The cycling schedule again represents a discharge depth of 25% with an overcharge of 10%.

The membranes tested were Permion 305, Permion 306, Permion 307, Cellophane 300, and Cellophane 600. Three layers of all membranes were used except Cellophane 600, where two layers were used. Two layers of Cellophane 600 exhibit approximately the same wet thickness as three layers of Cellophane 300. Table No. VI contains a summary of this test.

TABLE NO. VI

ACCELERATED SEPARATION CYCLING TEST SUMMARY
(THREE LAYERS WITHOUT NYLON)

TYPE MEMBRANE	CYCLES TO FAILURE
Permion 305	99
Permion 306	143
Permion 307	145
Cellophane 300	484 *
Cellophane 600 **	484 *

* Cellophane 300 and Cellophane 600 are still cycling; 484 cycles have been obtained to date.

** Cellophane 600 was tested using only two layers.

Table No. VI reveals the relative cycle life obtained from three layers of the more acceptable Permion membranes and Cellophane 300, and two layers of Cellophane 600. The table discloses the noteworthy superiority of the cellophanes for room temperature cycling, as they have completed 484 cycles, compared to 145 for the best of the Permion membranes tested. Perhaps the most important observation from Table No. II of the First Quarterly Progress Report and Table No. V of this report, is the inconsistency of the expected cycle life of the Permion membranes. For example, two layers of Permion 305, without nylon, yielded 520 cycles, whereas three layers of Permion 305, again without nylon, yielded only 99 cycles. Differences of this magnitude supposedly far exceed the range of experimental error, and although some of the Permion membranes appear very promising on some tests, their cycle life is far exceeded by an equivalent thickness or resistance of cellophane in an identical test. Differences are also observed in the case of the cellophanes although not of this magnitude. These differences are a possible explanation of the wide difference in actual cycle life of control cells cycling under practically identical circumstances.

Suggestions will be made to Radiation Applications, Inc. in an effort to help them produce more consistent products as well as more superior products.

B. Capacity and Weight Improvements

1. Number of Plates per Cell

A series of eight cells was fabricated and tested in order to determine the capacity performance characteristics as affected by the number of plates per cell. The plates were varied in the C-6 container (4.5" x 0.75" x 1.5") from three to 15 to establish the maximum watt-hours per pound utilization at low rates of discharge as well as the ultimate in a high rate rechargeable cell of this size. Two cells of each type were tested. Construction details of these cells are shown in Table No. VII. Following activation, the cells were charged individually at a constant potential of 1.55 volts per cell for 48 hours. Initial charge current was limited to 0.5 ampere. The cells were discharged at a current density of 0.15 ampere per square inch to an end voltage of 0.60 volt per cell. Discharge capacities as well as watt-hour per pound yields are also revealed in Table No. VII.

The two cells containing two positive and three negative plates yielded the greatest ampere-hour capacity as well as the highest watt-hour per pound outputs. A possible explanation of their performance, which was greater than the cells containing only one positive and two negative plates, is the relative inefficiency of charge and discharge of extremely thick plates even though very low discharge rates are used. It is also suspected that the plates of Cell Type Nos. A-1 and A-2 were never completely charged, thus explaining the low discharge efficiencies of both types of cells.

A-4 cells will be fabricated during the next quarter, as well as obtaining various temperature charge and discharge data for this group of cells.

TABLE NO. VII

CONSTRUCTION DETAILS
NUMBER OF PLATES PER CELL STUDIES

CELL NO.	A-1	A-2	A-3	A-4
Number of Positive Plates	1	2	4	7
Positive Weight, grams	89	86	55	Under Construction
Number of Negative Plates	2	3	5	8
Negative Weight, grams	88	83	56	Under Construction
Capacity, Amp.-hrs. 80° F	14.1	14.6	10.7	--
Watt-Hours per Pound	23.8	25.7	21.3	--
% Theoretical Capacity of Positive Plates	66.0	70.9	81.2	--

NOTE: All cells were separated identically as follows:
Positive Plates: One wrap of #9625 Nylon
Six wraps of #300 Cellophane

Negative Plates: One wrap of R-35-D Viskon

2. Studies of Positive-to-Negative Active Material Ratio at Various Temperatures

A series of cells was fabricated in an effort to establish an optimum cell for a cycling operation at -20° F and +120° F. Past studies have revealed the optimum positive-to-negative ratio by efficiency tests to be .95:1 for -20° F, 1.6:1 for +80° F, and 1.45:1 for +120° F operation. These calculations were made using Figure Nos. 79 and 80 of the Final Report of Contract No. DA-36-039-sc-85370, and assuming the monovalent oxide to be 50% as efficient at each temperature as the divalent silver oxide. These active material ratios were calculated for cells operating at the cadmium - monovalent silver oxide capacity level. A total of eight cells was fabricated. Four of the cells were designed to operate at -20° F and four at +120° F. All of the C-6 size cells contained nine plates and were separated in an identical manner. The separation consisted of one wrap of nylon, six wraps of 300 Cellophane, and one wrap of R-35-D Viskon.

Initial room temperature discharges revealed that the cells designed for +120° F operation outperformed the -20° F cells on a capacity basis. The +120° F cells yielded an average capacity of 9.2 ampere-hours, and the -20° F cells yielded 6.25 ampere-hours. These results are as expected because of the positive limitation of the -20° F cells for +80° F operation. However, when recharged at room temperature and discharged at -20° F, the cells designed for +120° F again outperformed the cells designed for -20° F operation. The -20° F designed cells yielded 3.06 ampere-hours, and the +120° F cells yielded 4.25 ampere-hours. This fact can possibly be explained by the efficiency-of-charge of the two different cells.

Because of the greater amount of positive material in the +120° F cells, both the positive and negative groups were able to attain a greater overall charge than the -20° F cells. As a further explanation, even though the -20° F cells had theoretically enough negative material to sustain a discharge exceeding the negative material of the +120° F design, it was impossible to charge this material while the cells were sealed, and although the balances as used were probably optimum for a vented cadmium - silver oxide cell (which could be overcharged), they lacked optimum qualities for a sealed cell.

Following their discharge at -20° F, it was planned to recharge the cells at room temperature and obtain a +120° F discharge on the cells following a room temperature charge. The +120° F discharge was not attained because the cells were inadvertently ruined during their room temperature charge. The cells were ruined by overcharging at a constant potential which was mistakenly set at 16.0 volts instead of the 12.8 volts. The overcharge resulted in very bad bulging of the covers and unsupported sides of the cells, leaving the cells unsafe for further testing.

C. Improvements in Recombination

During this quarter, efforts were directed toward establishing the proper quantity of electrolyte for both high recombination rate and capacity for various combinations of separator materials. Several types of separator materials were used, including some of the more promising Permion membranes which have been proved to be superior to cellophane for high temperature applications.

1. Construction

The cells tested were fabricated in six groups with a total of four cells in each group. Two cells from each group were constructed with pressure gauges, and all cells were sealed with the nylon-teflon pressure type seals. A typical high rate construction of seven positive plates and eight negative plates was designed in order to obtain a large surface area of active cadmium for recombination to occur. In addition, three 4/0, .005 inch expanded nickel grids (the same size as the plates) were dispersed throughout the negative group and in contact with the cadmium plates. The grids, which have been evaluated in earlier work, apparently increase the availability of cadmium and thereby increase recombination. The cells were fabricated from unformed silver plates weighing 0.77 gram per square inch, giving a theoretical capacity of 8.5 ampere-hours with respect to divalent silver oxide capacity. The unformed negative plates, cadmium oxide, weighed 1.7 grams per square inch, giving a theoretical capacity of 18.8 ampere-hours, and a practical capacity (if fully charged) of approximately 9.4 ampere-hours. Detailed construction variables of each cell are shown in Table No. VIII.

The only variable within a particular group is the amount of electrolyte in each cell. The cells were activated in the following manner: Cell No. 1 from each group was flooded with 1.300 sp gr KOH under a vacuum of 26 inches. After a soak period of one hour, the cell was drained under a vacuum of 26 inches so that the only electrolyte remaining was that which was retained in the separation and the pores of the plates. The remaining three cells from each group were then activated with 1 cc each less electrolyte than the preceding cell number. For example, Cell No. 1 of Group No. H was activated with 23 cc, Cell No. 2 was activated with 22 cc, Cell No. 3 with 21 cc, and Cell No. 2 with 20 cc.

TABLE NO. VIII

CONSTRUCTION VARIABLES AND OVERCHARGE TEST SUMMARY

GROUP NO.	H				I				F					
CELL NO.	1	2	3	4	1	2	3	4	1	2	3	4	1	
SEPARATION	9526 Nylon 307 Permion-40/30 (six wraps) & R-35-D Viskon				9526 Nylon 307 Permion-40/20 (six wraps) & R-35-D Viskon				9526 Nylon 307 Permion-40/45 (six wraps) & R-35-D Viskon				9526 Nylon 40/45 (thre Cello. (thr R-35-D Visk	
ELECTROLYTE VOLUME, cc	23	22	21	20	24	23	22	21	23	22	21	20	23	2
CAPACITY, A.H. FIRST DISCHARGE	5.6	5.4	5.2	2.6	5.6	4.6	5.3	2.3	5.5	4.9	5.5	1.6	4.1	2
MAX.VOLT.-PRESSURE @ FOLLOWING CHARGE RATES: (Volts-PSIG)														
0.10 ampere	1.78- 8	1.56	1.68-0	1.52	1.73- 2	1.46	1.71-1	1.52	1.53-54.5	1.52	1.54-17	1.52	1.82-33.5	1
0.20 ampere	1.96- 27	1.56	1.97-0	1.56	1.93-100	1.53	2.02-1.5	1.99	1.58-100	1.54	1.57-42.5	1.53	1.76-67.5	1
0.25 ampere	1.82-100+	1.59	1.99-0	1.59	*	1.56	1.98-2	2.15	*	1.55	1.58-58.5	1.54	1.84-67.5	1
0.30 ampere	*	1.60	2.01-0	1.65	-	1.57	2.18-1.5	2.38	-	1.57	1.60-89.5	1.55	1.81-68	1
0.35 ampere	-	1.58	2.01-0	1.64	-	1.56	2.14-1.5	2.42	-	1.58	1.65-93	1.56	1.85-68	1
0.40 ampere	-	1.67	1.99-0	1.78	-	1.68	7.60-0**	3.09	-	1.59	1.68-105	1.58	1.96-68	1
0.45 ampere	-	1.70	*	1.92	-	1.64	-	*	-	1.60	*	1.60	2.06-67.5	1
0.50 ampere	-	1.75	-	2.12	-	1.59	-	-	-	1.60	-	1.60	2.02-67.5	1
0.55 ampere	-	1.82	-	2.48	-	1.60	-	-	-	1.63	-	1.66	1.98-67.5	1

NOTE: Cell Nos. 1 and 3 of each group contain pressure gauges.

* Removed from circuit on preceding overcharge.

** This cell was removed from circuit. Apparently, it developed a leak.

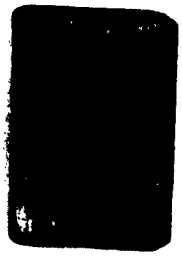

TABLE NO. VIII

ICTION VARIABLES AND OVERCHARGE
TEST SUMMARY

F			G				J				K			
2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
ı ɔn-40/45 (six ɜkon			9526 Nylon 307 Permion-40/45 (three wraps) 300 Cello. (three wraps) & R-35-D Viskon				307 Permion-40/30 (seven wraps) & R-35-D Viskon				9526 Nylon 300 Cello. (six wraps)& R-35-D Viskon			
22	21	20	23	22	21	20	23	22	21	20	24	23	22	21
4.9	5.5	1.6	4.1	2.1	2.3	0.3	5.26	2.46	4.06	2.46	5.06	4.6	2.2	0
1.52	1.54-17	1.52	1.82-33.5	1.52	1.79-4	1.52	1.89- 1	1.52	1.86-1	1.54	1.53-39	1.53	1.53-31.5	1.55
1.54	1.57-42.5	1.53	1.76-67.5	1.54	1.92-14	1.55	1.98- 2	1.55	1.97-3	1.62	1.54-98	1.54	1.54-78	1.75
1.55	1.58-58.5	1.54	1.84-67.5	1.56	1.94-20	1.56	1.98- 2.5	1.58	1.98-4.5	1.85	*	1.55	1.55-69	2.86
1.57	1.60-89.5	1.55	1.81-68	1.56	1.97-27.4	1.57	1.98- 4	1.66	2.04-5	2.09	-	1.55	1.54-86	2.93
1.58	1.65-93	1.56	1.85-68	1.57	1.99-41	1.59	2.04- 4.5	1.68	2.08-6	2.0	-	1.56	1.55-93.5	**
1.59	1.68-105	1.58	1.96-68	1.57	2.01-32	1.61	2.46-14.5	1.69	2.15-4.5	1.94	-	1.57	*	-
1.60	*	1.60	2.06-67.5	1.57	1.98-14	1.63	2.40- 7.5	1.80	2.14-2	2.12	-	1.61	-	-
1.60	-	1.60	2.02-67.5	1.57	1.90- 7	1.64	2.41- 6.5	1.78	2.16-2	2.02	-	1.62	-	-
1.63	-	1.66	1.98-67.5	1.61	1.98-10	1.73	2.63- 4	1.85	2.27-2	2.16	-	1.68	-	-

essure gauges.

ge.

ently, it developed a leak.

2. Testing

Following activation and sealing, all cells were charged at a constant current of 0.5 ampere to an on-charge voltage of 1.65 volts per cell. They were then given a discharge so that their initial capacities could be determined. These capacities are presented in Table No. VIII. It can be observed that in general, decreasing electrolyte volume below that amount held by the pores of the plates and separation decreases cell capacity. This is a reasonable result since theoretically, one mole of water is consumed during the discharge of one mole of cadmium. It is believed that this consumption of water hastens polarization due to the loss of electrolyte at the interface of the negative plates. This consumption is equivalent to 0.33 gram of water per ampere-hour of cadmium discharged. It can be observed in the table that in every case, capacity is not decreased with decrease in electrolyte volume being perhaps associated with other inherent variables. The overall drop of 3 cc in each group yields an average loss of 1.2 ampere-hours per cubic centimeter of 1.300 sp. gr. KOH removed. Therefore, the overall percentage loss in capacity is 70% due to the loss of 3 cc's of electrolyte. (These data were calculated from maximum and minimum capacity data in Table No. VIII.)

After initial capacities were determined, it was decided to determine the maximum overcharge rate of each cell so that this information could be correlated with capacity, and an optimum set of design information attained.

Overcharge testing was initiated at a rate of which it was assumed that all of the cells would accept. The initial rate of overcharge was established at 0.1 ampere, or the 60-hour charge rate of the higher

capacity cells. The cells were then charged at this rate for 60 hours, and all cells accepted this rate of overcharge without exceeding 3.0 volts per cell, or 100 psig pressure. The charge current was then increased to 0.2 ampere. The cells were charged at this rate for 24 hours. The charge rate was then raised in increments of 0.05 ampere on acceptable cells. Each current was applied to the fully charged cells for 24 hours. Table No. VIII gives a summary of the maximum voltages and pressures attained by the cells at each charge rate.

It can be observed that only two of six of the cells with the maximum amount of electrolyte would overcharge at 0.55 ampere, the maximum rate tested. All six cells of the type activated with one cc less electrolyte than would be held by the pores of the plate overcharged satisfactorily up to the 0.55 ampere rate. Two of the six cells containing two cc's less than that held by the pores of plates and separators accepted a continuous overcharge at 0.55 ampere, and four of the six cells containing three cc's less than that held by the pores of the plates and separation passed the test.

The cells containing the greatest quantity of electrolyte yield greatest capacity and energy per unit weight, and from past experience, the cells with a small volume of electrolyte yield the best continuous overcharge characteristics. However, if the test were terminated at this point, it would be obvious that one cc less than that held by the core would be the optimum amount of electrolyte both for best capacity and maximum continuous overcharge.

The tests will be continued until more extensive conclusions can be made as to maximum attainable overcharge rate, as well as the maximum overcharge rate for the best cells of each group.

D. Environmental Testing

A large portion of the work performed on this contract will be an attempt to define the operating characteristics of sealed cadmium - silver oxide cells developed under Contract No. DA-36-039-sc-85370, and refined under this contract. A discussion of these tests and data obtained from them follows.

1. Cycle Life Performance of Control Cells

It was decided to cycle test control cells at various constant temperatures and different discharge depths. The first cycling test is being performed at a constant temperature of +80° F. The cycling frequency consists of four hours charge and two hours discharge. The discharge depth of three, ten-volt batteries has been set at six, four, and two ampere-hours, respectively. These depths of discharge are equivalent to 60%, 40%, and 20% of the actual capacity of the batteries. The test temperature of the batteries is maintained at +80° F ± 2° F by means of cooling coils and heaters as was discussed in the previous report. Other details of the test, equipment, and construction details of the batteries are discussed in the First Quarterly Progress Report of this contract.

At the writing of this report, the batteries cycling at 20% and 40% of their actual capacity have successfully completed 273 cycles. Failures due to cell reversals of the battery cycled at 60% discharge depth occurred as revealed in Table No. IX.

TABLE NO. IX

CELL FAILURES OF BATTERY NO. III
(60% Discharge Depth)

CELL NUMBER	CYCLES TO FAILURE BY REVERSAL
83	75
89	76
81	84
90	84
82	95
86	111
84	127
87	132
85	222
88	222

All failures observed were apparently due to capacity loss as none of the cells contained shorts of noticeable magnitude. After each failure, the failed cell was recharged and allowed to remain on open circuit. No loss in open circuit voltage has appeared in any of the failed cells.

It is possible that the constant potential of 15.5 volts was not sufficient to maintain complete charging of Battery No. III when operating at the 60% depth of discharge. Perhaps the constant potential could be increased slightly and compensate for the inefficiency of charge. If this is the cause of failure, the cells could still be made to deliver useful capacity. It is planned to remove the cells from the oil bath, give them a conditioning charge and discharge, and return them to the cycling apparatus at a constant potential of 16.5 volts.

The remaining two batteries are being monitored carefully, with the end of discharge voltage being recorded once each day. As of the writing of this report, none of the cells are apparently near failure.

Tests similar to those discussed above, carried out under N.A.S.A. Contract No. NAS 5-1318, were continued during this quarter. The batteries were cycled at a 100-minute frequency, 60 minutes charge and 40 minutes discharge. The depth of discharge was 10% of the actual battery capacity. The batteries operating at +80° F and +20° F continued to cycle. Cells from the battery operating at +80° F have completed 4300 cycles. One cell was removed after 4061 cycles because of reversal during discharge. The reason for failure is not exactly known as the cell has not been removed from the package. Extremely low charge and discharge voltages observed immediately before failure indicate the possibility of shorting. The remaining five cells have completed 4300 cycles to date.

The ten-cell battery cycling at +20° F has completed 5017 cycles. Low charge and discharge voltages for two of the cells indicate the approach of failure. Cycling of this battery will continue, and will be discussed during the following quarterly report.

A discussion of the effect of cycling temperature on the life of a cadmium - silver oxide battery was presented in the First Quarterly Progress Report on this contract. Data obtained during this quarter indicate that the effect was minimized in the first discussion. More realistic conclusions will be reported when the test has been completed.

2. Self-Discharge Characteristics

Data on single cell tests reported under Contract No. DA-36-039-sc-85370 have indicated excellent retention-of-charge characteristics for sealed cadmium - monovalent silver oxide cells. Because of these indications, an extensive test program has been initiated in order to fully define the self-discharge characteristics of sealed cells operating at the cadmium - monovalent silver oxide capacity level.

Data was presented in the First Quarterly Progress Report revealing capacity retention as affected by storage time up to three weeks at temperatures of +165° F, +120° F, +80° F, 0° F and -65° F. Table No. X reveals capacity retention for a storage period of up to three months. As revealed in the table, the cells would not deliver useful capacity following a storage period of six weeks at +165° F. Only one of the three cells stored at +120° F for three months delivered a capacity. The cell that delivered a capacity at +120° F delivered only 12.5% of its initial capacity. The one cell tested after a storage period of six weeks delivered only 17.3% of its initial capacity. "Post mortem" inspection of

TABLE NO. X

SELF-DISCHARGE CHARACTERISTICS

CELL NUMBER	STAND TEMP. -°F-	CAPACITY AFTER 1 WEEK -A.H.-	CAPACITY AFTER 3 WEEKS -A.H.-
1	+165	7.25 - 68% *	---
2	+165	---	5.25 - 49.3%
49	+120	7.16 - 67.3%	---
50	+120	---	7.66 - 72%
37	+80	10.25 - 96.3%	---
38	+80	---	8.35 - 78.2%
13	0	10.66 - 99.9%	---
14	0	---	10.58 - 99.3%
26	-65	10.6 - 99.5%	---
27	-65	---	10.75 - 101%

		CAPACITY AFTER 6 WEEKS -A.H.-	CAPACITY AFTER 3 MONTHS -A.H.-
5	+165	0	---
6	+165	0	---
7	+165	0	---
71	+120	1.85 - 17.3%	---
72	+120	---	1.33 - 12.5%
73	+120	---	0
74	+120	---	0
39	+80	9.94 - 93.2%	---
40	+80	---	9.75 - 91.5%
41	+80	---	10.50 - 98.7%
42	+80	---	8.90 - 83.5%
15	0	10.85 - 101.9%	---
16	0	---	10.1 - 94.9%
17	0	---	9.51 - 89.5%
18	0	---	9.40 - 88.3%
28	-65	---	9.1 - 85.5%
29	-65	---	9.6 - 90.2%
30	-65	---	10.6 - 99.5%

* The second figure shown in the capacity column represents the percentage of full capacity yielded. Full capacity used in these calculations was 10.67 ampere-hours.

the cells, after it was found that they would not take a charge, revealed a complete deterioration of the cellophane separator material, resulting in electronic shorting of the cells. In the future, cells fabricated for operation or storage at +120 to +165° F for three weeks or longer should be separated with multiple layers of one of the Permion membranes. These membranes have exhibited high temperature stability superior to that of cellophane.

Of the cells stored at +80° F, after six weeks storage, Cell No. 39 retained 93.2% of its initial capacity. After a storage period of three months, the three cells tested delivered 91.5, 98.7, and 83.5% of their initial capacity. This is an average of 91.2%, or excellent charge retention.

As was expected, the cells stored at 0° F and -65° F again exhibited excellent charge retention characteristics. The cells stored at 0° F retained an average of 90.9% of their initial capacity, and those stored at -65° F retained 91.2%.

Since no appreciable capacity losses have been observed due to storage at 0° F, it is presumed that similar or even smaller losses will occur at -65° F. Therefore, during the next quarter, the remaining cells stored at -65° F will be placed in +165° F and +120° F storage in order to more fully define capacity retention between three weeks and six weeks storage at these temperatures.

In general, the tests are revealing excellent storage properties of the cadmium - monovalent silver oxide system at temperatures of +80° F or lower.

The storage characteristics at greater than room temperatures are apparently superior to zinc - silver oxide cells as well as nickel - cadmium cells. Storage tests will continue such that sufficient data will be obtained to fix charge retention versus storage time characteristics at the various temperatures.

E. Hermetic Seal

As was discussed in the First Quarterly Progress Report, samples of ceramic-to-metal hermetic seals have been obtained from the Carborundum Company, Latrobe, Pennsylvania, and the Physical Sciences Corporation, Pasadena, California. During this quarter, samples have been obtained from Coors Porcelain Company, Golden, Colorado.

Samples from all three companies have been evaluated by covering the ceramic material with 1.300 sp. gr. KOH, 60 psig oxygen, in an empty cell container, while the container and contents are maintained at +165° F. The seals manufactured by the Carborundum Company and Coors were apparently unaffected by a 60-day storage period under the above conditions. The seals obtained from Physical Sciences Corporation failed after an average test duration of one week. In a conference with P.S.C. personnel, the possibility was introduced that the rapid failure might be due to cracking the insulator material during welding due to an inadequate heat sink. This theory was not tested, however, as another group of P.S.C. seals failed after submersion in 1.300 sp. gr. KOH for a period of three weeks. These failures could not have been due to cracking the insulating material, as the glass was entirely etched away in some cases. P.S.C. has been notified of the failures and are in the process of trying to duplicate their original insulating material which was reported to be unaffected by a storage period of five weeks in 50% KOH at a temperature of +165° F.

Fifty production covers with two ceramic-to-metal seals in each cover have been received from Coors, and have been incorporated into the C-6 cell design and are presently being evaluated in cell usage. The results obtained to date have been completely satisfactory. The results will be evaluated and reported in future reports.

Samples have also been obtained from Gladding-McBean Ceramics Company, Los Angeles, California. These seals are of the high alumina type which are apparently satisfactory. These seals will be evaluated during the third quarter.

V. CONCLUSIONS

1. Cellophane 600 exhibits the most stable resistance of the membranes tested over the temperature range of 0° F to +120° F.

2. Two layers of Cellophane 300 yielded more cycles than two layers of any other membrane and nylon tested. (Two layers of Cellophane 600 presumably should yield more, but it did not in this particular test.)

3. Gross inconsistencies far exceeding experimental error have been observed in cycling membranes.

4. Room temperature discharges of "C-6" cells indicate that the five-plate cells have the highest energy output per unit weight of the cells tested.

5. Sealed cell studies of positive-to-negative active material ratio reveal that the same ratio would not be used for sealed cells as would be used for vented cells.

6. Continuous overcharge up to the C/10 rate has been achieved at the expense of a 35% capacity loss due to limiting of the electrolyte volume.

7. Cycle life of "C-6" cells at a 60% actual discharge depth, six-hour cycling frequency, apparently varies from 75 to 222 cycles.

8. Standard "C-6" cells will not yield a useful capacity after a storage period of six weeks at +165° F, or three months at +120° F.

9. Ceramic-to-metal hermetic seals obtained from Coors Porcelain and The Carborundum Company have passed accelerated evaluation tests.

VI. PROGRAM FOR NEXT INTERVAL

The program planned for the next quarter will proceed as it has been scheduled. The following is an outline of this work:

A. Improvements in Type Insulation

The relative cycle life studies of various films will continue. Cycling of three layers of membranes will be completed during this quarter. Relative resistances of multiple layers of membranes at +80° F will be determined.

If time permits, an evaluation of combinations of membranes in sealed cell assemblies will be initiated.

B. Capacity and Weight Improvements

Studies of capacity performance of "C-6" cells in which the number of plates per cell were varied from three to 15 will continue. The capacity performance studies will be extended over the temperature range of -60° F to +120° F.

C. Improvements in Recombination

Overcharge studies of cells discussed in this report will be completed. An evaluation of the following methods of improving recombination will also be made:

1. Silver plate inside of can.
2. Use nickel grid insert in can.
3. Add silver to the cadmium plate.
4. Paste CdO on only one side of the grid, exposing the other side to grid metal.
5. Use thin Pellon in place of R-35-D Viskon.
6. The use of various mechanical approaches.

D. Environmental Testing

Cycle life performance testing of "C-6" cells operating at the cadmium - monovalent silver oxide capacity level at a constant temperature of +80° F will continue. An effort will be made to initiate cycle testing of similar batteries at 0° F and +120° F.

Retention-of-charge tests will continue. Cells on stand at -60° F will be placed on stand at +120° F and +165° F in order to more fully evaluate self-discharge characteristics over time periods up to six weeks.

E. Hermetic Seals

Rapid evaluation testing of ceramic-to-metal seals obtained from Gladding-McBean will be initiated during the third quarter. Evaluation of Coors' seals in sealed cell assemblies will also continue.

VII. PERSONNEL

Listed below are the personnel who worked on the contract during the second quarter, their job titles, and the number of hours contributed:

NAME	TITLE	MAN-HOURS
Wilson, J.	Project Engineer	420
Dittmann, F.	Engineering Supervisor	90
Morse, E.	Engineering Manager	40
Swanson, K.	Assistant Engineer	264
Pope, L.	Assistant Engineer	61
Kernohan, T.	Technician	531-1/2
	TOTAL	1,406-1/2

APPENDIX

Sealed Ag-Cd Development - Work Schedule - FA212

	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	April	May	June
PHASE I - Improvements in Type Insulation												
A. Accelerated method of evaluation												
1. Relative cycle life of various films	———	———	———	———	———	———						
a. One layer	———											
b. One layer and nylon	———											
c. Two layers		———										
d. Two layers and nylon			———									
e. Three layers				———								
f. Three layers and nylon					———	———						
2. Electrical Resistance of Materials	———	———	———									
a. Electrical resistance at 80° F	—											
b. Electrical resistance at 0° F	———											
c. Electrical resistance at 120° F		———										
d. Relative resistance of multiple layers 80° F			—									
B. Evaluation of various materials in actual cell assemblies												
1. Series No. 1 performance of various types				———								
2. Series No. 2 effects of multiple layers					———							
3. Performance of combinations of materials						———						
a. Permion-Nylon-R-35-D Viskon						———						
b. Cellophane-Nylon-R-35-D Viskon						———						
c. Permion-Cellophane-Nylon-R35D Viskon						———						
d. Permion-Pellon						———						
e. Cellophane-Pellon						———						
f. Permion-Cellophane-Pellon						———						

Sealed Ag-Cd Development - Work Schedule - FA212

July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	April	May	June

PHASE II - Capacity and Weight Improvements

Objective: To establish the number of plates per cell, active material density, and ratio of positive to negative to give optimum performance of Ag_2O/Cd and Ag_2O_2/Cd systems.

A. Number of plates per cell.
   1. Vary plates per cell from 3 to maximum and determine capacity performance at -60°, -20°, 0°, 80°, and 120° F.
   2. Determine relative cycle life (50% depth)

B. Determine ratio of positive and negative active material by capacity tests at -20° F, 80° F and 120° F as well as by cycle life performance (Series should establish whether excess Cd is required in Ag_2O/Cd system.)

C. Relative performance of Ag_2O/Cd and Ag_2O_2/Cd systems. (Construct cells of optimum design using both systems and establish capacity and life characteristics, and rates of self-discharge.)

Sealed Ag-Cd Development - Work Schedule - FA212

July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	April	May	June

PHASE III - Improvements in Recombination

Objective: To establish a means of improving recombination rate such that the Ag_2O_2/Cd system can be utilized.

A. Establish electrolyte volume such that sufficient recombination will occur to allow cells to work at the Ag_2O_2 level and determine their performance characteristics. ———————

B. Evaluate the following methods for improving recombination.
   1. Silver plate inside of can. ———
   2. Use of nickel grid insert in can. ———
   3. Add silver to cadmium plate. ———
   4. Paste CdO on only one side of grid exposing the other side to grid metal. ———
   5. Use pellon in place of R-35-D. ———
   6. Use mechanical approach. ———

Sealed Ag-Cd Development - Work Schedule - FA212

July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	April	May	June

PHASE IV - Environmental Testing

Objective: To define the performance characteristics of a line of cells constructed with both the Ag_2O/Cd and Ag_2O_2/Cd systems.

A. Capacity performance at -60° to 120° F at various rates.

B. Cycle life performance at 0°, 80°, and 120° F at various discharge depths.

C. Retention-of-charge characteristics at -65°, 0°, 80°, 120° F, 165° F

Note: These tests should be constructed for at least three different size cells.

PHASE V - Hermetic Seal

Objective: To develop an improved hermetic seal and establish its reliability.

A. Improvements in existing seals.

B. Search and evaluation of ceramic seals.

AD__________ Accession No. __________

The Eagle-Picher Co., Joplin, Mo.

SEALED, RECHARGEABLE CADMIUM - SILVER OXIDE CELLS - J. Wilson

Report No. 10, Second Quarterly Progress Report, Contract No. DA-36-039-sc-89065 (Continuation of Contract No. DA-36-039-sc-85370), DA Proj. No. 3A99-09-002, October 1, 1962 to December 31, 1962, Date of Report January 31, 1963, 30 pp, tables.

Development work is being directed toward the attainment of a reliable sealed cadmium - silver oxide battery. Effort is directed toward the development of both cadmium - divalent silver oxide and cadmium - monovalent silver oxide cells, and studying the performance characteristics of each system.

Of the major problems encountered, probably the most important is the relative inconsistency of available separator materials, including both areal resistance and cycle life measurements. Continuous overcharge at rates up to C/10 have been attained, but only at a loss in capacity efficiency of approximately 35%. Problems of this nature must be surmounted before reliable cadmium - silver oxide cells can be produced.

Unclassified

1. Contract No. DA-36-039-sc 89065
2. Sealed AgO-Cd Battery
3. Separator Materials
4. Continuous Overcharge

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