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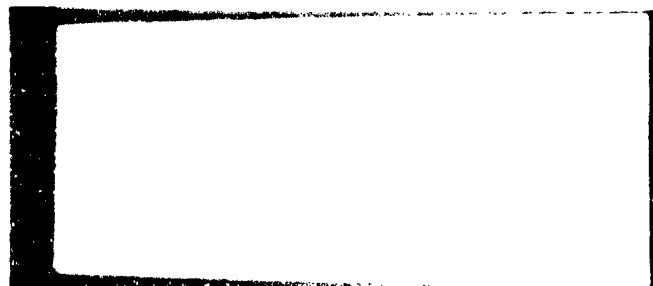
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Alkaline Battery Division

GULTON INDUSTRIES, INC.

Metuchen, N. J.

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**INVESTIGATIONS
LEADING TO THE DEVELOPMENT OF IMPROVED
SEALED NICKEL-CADMIUM BATTERIES
REPORT NO. 6**

**Signal Corps Contract No. DA 36-039 SC-85390
Department of the Army Project No. 3A9909002**

**Sixth Quarterly Progress Report
1 October 1961 to 31 December 1961**

**U.S. Army Signal Research and Development Laboratory
Fort Monmouth, New Jersey**

**Gulton Industries, Inc.
Alkaline Battery Division
Metuchen, New Jersey**

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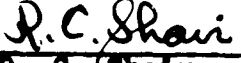
Sixth Quarterly Progress Report
1 October 1961 to 31 December 1961

The Testing of Hermetic Seals and Separators, Positive
Plate Studies, and Investigations of the Uniformity of
Sealed Nickel-Cadmium Cells

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I. PURPOSE

The purpose of this work is to conduct investigations which will lead to improved hermetically sealed nickel-cadmium batteries having a high level of reliability and performance. Efforts will be directed toward: (1) development of a ceramic-to-metal seal for 5 ampere-hour cells; (2) development of a good separator; (3) development of a greater uniformity between cells, and test methods for rapidly determining uniformity; (4) a fundamental study of the positive electrode directed at increasing its stability and reducing self-discharge; and (5) fabrication of improved 5 ampere-hour hermetically sealed cells.

II. ABSTRACT

Screening tests were started on three new separator materials; (1) a modified polypropylene from RAI, (2) a nonwoven nylon paper from Eaton Dikeman, and (3) a modified nonwoven polyamide from Pellon. The Pellon material is the only one of these three which appears satisfactory for continued testing.

Laboratory cells of the VO-6 type were prepared with two kinds of polypropylene separator and with a nonwoven polyamide material. The pressures during a C/10 overcharge of the cells with polypropylene materials reached over 150 PSIG. They were vented to the atmosphere and the gas was found to contain hydrogen as well as oxygen. The voltages during this overcharge averaged about 1.56 volts. The cells with the polyamide separator averaged 45 PSIG and 1.43 volts.

Hermetically sealed cells, VO-6HS, were charged for 8 hours at 1.0 amperes. The capacity obtained appears to indicate insufficient charge. An overcharge versus discharge capacity curve at room temperature indicates that about a 20% overcharge is required.

A formation study was instituted. The results indicate that capacity, voltage and pressures subsequent to formation are not significantly dependent upon the mode of formation charge (fast or slow), the mode of formation discharge (fast or slow), nor does it depend upon whether the formation discharge is carried to the exhaustion of the negatives.

The program for the study of the slow self-discharge process is underway. Oxygen analysis were run by the spectro-

chemical procedure. The electrochemical procedure requires a dilution of the electrolyte to obtain reproducible results, but this procedure consequently reduced the sensitivity of the analysis.

III. REFERENCES AND CONFERENCES

A. REFERENCES

1. Gulton Industries, First Quarterly Report, S. C. Contract DA 36-039 SC-85390, 30 September, 1960.
2. Gulton Industries, Second Quarterly Report, S. C. Contract DA 36-039 SC-85390, 31 December, 1960.
3. Gulton Industries, Fifth Quarterly Report, S. C. Contract DA 36-039 SC-85390, 30 September, 1960.
4. Gulton Industries, 11th Monthly Progress Report, Contract No. AF 33(600)-41670, October, 1961.
5. Gulton Industries, Fourth Quarterly Report, S. C. Contract DA 36-039 SC-85390, 30 June, 1961 .
6. E. Baars, Proc. 12th Ann. Power Sources Conf. Asbury Park, 1958.

B. CONFERENCES

Two conferences were held at USASRDL at Fort Monmouth, New Jersey on November 3rd and November 28th, 1961. Messrs. A. Frink, P. Rappaport, H. J. Mandel, Doctors E. Baars, and A. Fischbach represented USASRDL. Dr. R. C. Shair and Messrs. H. N. Seiger and W. Lutz represented Gulton Industries. Progress on all phases of the work was discussed. After reviewing the draft of the Fifth Quarterly Report the necessary corrections were made. Plans for the work for the next quarter were discussed.

IV. SEPARATORS

Three separator samples were received during the past quarter. Eaton Dikeman submitted a nonwoven paper fabricated from a polyamide. Radiation Application Incorporated submitted a treated polypropylene material, and the Pellon Corporation submitted a modified polyamide. The separator screening tests were started on these materials, and the results thus far obtained are reported.

Cells containing test separators were prepared. These were laboratory type VO-6 cells equipped with pressure gauges. Three separator materials were tested in these cells, two of which were polypropylene and one a modified nonwoven polyamide.

A. Screening Tests

The separator samples tested were a paper fabricated from nylon, a nonwoven polyamide and a modified polypropylene. The results obtained using the methods described in earlier reports ^{1,2} are given in Tables 1 and 2.

The polyamide (X624) absorbs six times its own weight of electrolyte while the other samples are considerably lower. The Eaton Dikeman material retained only 73% of the absorbed electrolyte during acceleration.

The modified polypropylene (X622) was not uniform with regard to thickness and with regard to resistance. The nylon paper had a region in one of the samples which could not be wetted.

B. Laboratory Cell Data

Laboratory cells of the VO-6 type were prepared using several different separators. The separators were Pellon 960, American Felters X661 and X662 (both nonwoven polypropylenes). The cells were filled with 20 ml of 34% KOH. They were evacuated and sealed before charging. The charge was carried out at 0.6A with the cells in an oil bath at 77°F. The pressures and voltages at 24.6 AH input are given in Table 3. When the cells reached 150 PSIG, the cells were vented to the atmosphere. The gases in those cells which required venting contained hydrogen as well as oxygen. The capacities obtained from a 3A discharge are given in Table 4.

C. Discussion

The screening test results on resistance and electrolyte absorbed are poor for the Eaton Dikeman material and the RAI material. No further screening tests will be run on these materials. The Pellon material X624 has been able to absorb six times its own weight of electrolyte, and retains essentially all of it in the acceleration test. This material is 0.007 inches thick and has a low resistance after 24 hours in electrolyte. Further tests will be run on this particular material.

The cells constructed with the polypropylene materials yield essentially the same capacity as the group of cells that has the polyamide, Pellon 960. However, the pressures built up during charge were excessively high due to the evolution

of hydrogen. The evolution of hydrogen was suspected because the voltage at the end of the C/10 overcharge (0.6A) averaged about 1.57 volts. The comparable cells with the polyamide separator averaged 1.44 volts. This difference is very significant. Because of these observations, these particular separators, X661 and X662, are not recommended for use in hermetically sealed cells.

V. UNIFORMITY

A. General

Ten hermetically sealed VO-6 HS cells have been placed on test. These tests include capacity with no stand after charge, and also with an eight-hour stand after charge.

The cells of the HS series were charged at 200, 400, 750 and 1000 ma until the input was 12AH. The cells were discharged then at 3A to a 1.0 volt endpoint to determine the effect of charge rate on discharge capacity.

In an attempt to understand the lack of uniformity observed and reported in the previous Quarterly Reports, an investigation of electrode formation was instituted. Several combinations of formation charge and discharge rates were used. Some formation discharges were carried to the exhaustion of the negative electrodes. Cells were made from several of the formation groups, and the results of tests are reported.

B. Hermetically Sealed Cells

Ten VO-6 HS cells were made. These cells contain one ceramic-to metal seal at the positive terminal. The negative terminal was connected to the can. The data taken in the pilot plant on these cells are given in Table 5.

Subsequent to the above work, the cells were placed on a 1 ampere charge for 8 hours, allowed to stand on open circuit for 8 hours and then discharged at 1 ampere to 1.0 volt.

These cells were then recharged at 1 ampere for 8 hours followed by a discharge at 1 ampere to 1.0 volt. There was no open circuit stand in this case. The capacities obtained are

given in Table 6. The discharges of a typical cell (359) under the two conditions are shown in Figure 1. The average capacity for the 10 cells after the 8 hours stand is 6.3AH. The 8 AH input is insufficient, however, since this represents only a 5% overcharge for several of the cells. This possibly explains why there is less capacity on the discharge without the stand than on the discharge that had an 8 hour stand. The discharge capacity of VO-6 laboratory cells as a function of the amount of overcharge based on 6.8 AH, the average of the former history of the cells, is shown in Figure 2. These data indicate that cells should have a minimum of 20% overcharge.

It is planned to increase the charge to 10 AH input at the 1 ampere rate. This will correspond to a 35% overcharge for those cells with the greater capacity.

C. LABORATORY CELLS, HS SERIES

The laboratory cells of the HS series which are equipped with pressure gauges were charged at various rates. These were 200, 400, 750 and 1000 ma. The results in the previous report³ for capacity after various charge rates were for cells that had been on overcharge for different lengths of time. The criteria had been merely that $dP/dt \sim 0$. However, the pressure change is not great when the cells have a total input of about 12AH. Therefore, this experiment is being repeated with the condition that the total input is 12 AH.

After charge, at 77°C in an oil bath, the cells are discharged at 3A to a 1.0 volt endpoint. The discharge capacity as a function of the rate of charge to a constant input of 12AH is shown in Figure 3. The results are similar to that shown in the previous report. The charge efficiency at the lower charge rates

is less than that at the higher rates. It is of interest to determine whether the capacity after charge at lower rates can be increased by a greater input. To this end it is planned to use the 200 ma charge rate and determine capacity after various inputs ranging from 8AH to 120AH.

D. Formation Studies

Electrodes for the nickel-cadmium batteries are usually charged and discharged several times in order to have the materials in an electrochemically usable form. This process is termed formation of electrodes. Since uniformity was not attained previously, the formation process became suspect.

In order to investigate the effects of formation the following extremes of variables were taken:

- (a) Fast charge, 2.5A X 6 hours = 15AH
 Slow charge, 0.6A X 25 hours = 15AH
- (b) Fast discharge, 3A
 Slow discharge, 0.5A
- (c) Reversal, discharge carried to exhaustion of negatives.
 Discharge to 1.0 volt.

Eight groups of plates were made corresponding to a factorial experimental design based on the three combinations of variables. A ninth group was also made for cell testing from plates not formed.

The eight groups of plates were put through 3 formation cycles. On the last formation, the capacity of the negative electrodes were determined by a forced discharge to the exhaustion of the negative electrodes.

After formation the plates were washed and dried. Three groups of cells made from the following plates: slow charge, slow discharge, 1.0 volt; fast charge, fast discharge, 1.0 volt; fast charge, fast discharge, reversal. The fourth test group was cells made from unformed plates.

The four groups were placed on overcharge and discharge tests.

1. Experimental Data

a. Formation Cells

The experimental design and identification of the cell groups are shown in Figure 4. The discharge was monitored on a Non-Linear Systems 100 channel digital voltmeter. The capacities were determined at the 1.0 volt endpoint for the positive electrodes and the inflection point which occurs at about -0.6 volt was taken as the endpoint for the negative electrodes. Since the readings were taken with a DVM the capacities are accurate to within 1 minute, since they were scanned one each minute near the endpoints. This corresponds to an error of $\pm 0.05\text{AH}$ at the 3A discharge rate and $\pm 0.01\text{ AH}$ at the 0.5A discharge rate. The data for the formation discharges are given in Tables 7, 8 and 9.

There is no formation procedure that is really better than the others, nor is there one that is particularly poor.

b. Laboratory Cells

Sufficient cans were not available to make cells of all the plates so that only 3 groups were used. One of the groups chosen was F because it had the greatest positive capacity. Group E was also chosen because the charge and discharge rates were the

same as F, but F was discharged into reversal. Groups E and F correspond to the fast charge and fast discharge, and the last group selected was C which corresponds to the slow charge and slow discharge. A fourth group of 3 laboratory cells was made from unformed plates.

The first charge was at 0.6 A and a total of 42AH was put into the cells. The cells were in an oil bath maintained at 77⁰ F. They were then discharged at the 3A rate to the 1.0 volt endpoint. The voltage and pressure data near the end of overcharge, and the capacity from the discharge are given in Table 10. The voltages of the four groups (U is used for the cells made from unformed electrodes) are essentially the same. The pressures in the U series averaged lower than the plates which had been previously formed. It will be of interest to observe these pressures after the cells have been aged by additional charge-discharge cycles.

The average capacities are approximately the same. Apparently formation does not affect the discharge capacity.

A second discharge was obtained after a 56AH input at 0.6A. These data are given in Table 11. The average voltages are slightly higher, and so are the pressures. The cells of the U group (unformed plates) still average lower values than the cells from formed electrodes. The capacities generally are slightly lower (E, F and U groups) than on the first discharge.

It was suggested that upon an extended overcharge at the C/10 rate the pressures within a cell decreases. These cells were placed on such a charge, 32AH were put in at the C/20

rate and then the rate increased to C/10. The pressure data up to 223AH input are given in Table 12. Also in Table 12 there are five new cells, U4 through U8, inclusive. U4 has 16 ml KOH, U5 has 18 ml, U6 has 19 ml, U7 has 20 ml and U8 has 22ml of 34% KOH.

The grand averages for all the cells are in the last line of Table 12. There is a randomness in the averages with a very slight trend toward a decrease of pressure on continued overcharge. The average value of $-dP/dt$ is 0.014 Psi/Hour or 2.6×10^{-7} atmospheres per second.

2. Discussion

The type of formation does not appear to affect the overcharge characteristics. The cells with unformed plates average lower pressures than the cells from formed plates, but the unformed plates have not been aged to the same extent.

These formation experiments have not yielded more uniform cells. The capacities during the formation cycling were rather uniform within each experimental group, but when these plates were subsequently fabricated into cells the capacities were no longer uniform. The pressures built up during overcharge also indicate a lack of uniformity.

It is planned to measure the pressure decay on these 23 cells when charge is interrupted and compare these values with the calculated recombination rates during overcharge. After that the cells will be placed on an automatic cycle to accelerate aging, and the overcharge experiments repeated.

VI. STABILIZATION OF POSITIVE ELECTRODES

During the past quarter emphasis was placed on determining the rate of oxygen evolution caused by the slow self-discharge mechanism of the nickel oxide electrodes. It had been shown that the slow process accounts for the self-discharge of 90% of the capacity of a nickel oxide electrode. As for the more rapid oxygen evolution process, the effects of Li, Ba and Co are included in the investigation of the slow self-discharge process.

Impregnation has been completed. The positive electrodes were formed and storage began at various temperatures. Oxygen analyses were started. While oxygen analysis by the electrochemical method appeared feasible, it could not be used without dilution since an oxygen wave does not appear in the same manner as it does in 1M potassium hydroxide. The spectrochemical analysis using pyrogallol was used.

A. Plate Preparation

The plates were impregnated to contain approximately 1.6 grams $\text{Ni}(\text{OH})_2$. One hundred and twenty plates were prepared without foreign ions. One half of these will be used with lithiated electrolyte. The remaining 60 plates will remain without foreign ions. Sixty plates were impregnated with a solution of nickel nitrate that contained 5 atom % of Co, and another group of sixty plates were impregnated with a solution of nickel nitrate that contained 5 atom % of Ba.

The weight gains, theoretical capacity based on weight gain as Ni(OH)_2 and experimental capacity are given in Table 13. The capacities of about one quarter of the plates have been determined by subjecting the plates to charge-discharge cycles against several negative electrodes. For the undoped, Ba doped and Co doped electrodes the electrolyte was 34% KOH; for the series to be lithiated the solution contained 34% KOH and 1.3% LiOH.

B. Oxygen Evolution Data

In order to store the nickel oxide electrodes at the various temperatures a variety of constant temperature baths were used. For 10°C a refrigerator was used. Two air baths were set at 50°C and 75°C , respectively. Oil baths were set up and thermostatted for 30° , 40° , and 60°C .

It is desirable to store the electrodes in a minimum of electrolyte in order to minimize experimental errors. After impregnation, the nickel oxide electrodes were coiled into a cylinder about a 0.5 inch diameter mandril. The negative electrodes were coiled into a cylinder of slightly larger diameter. The separator for the formation was dynel-viscon. The plates were formed as follows:

Charge	-	16 hours, 75ma
Discharge	-	3 hours, 225 ma
Charge	-	3 hours, 300 ma
Discharge	-	3 hours, 225 ma
Charge	-	3 hours, 300 ma
Discharge	-	225 ma to 0.0 volts.

The plates were then charged overnight at 300 ma with a subsequent discharge at 225 ma to 1.0 volt to determine capacity.

These capacities are given in Table 13.

The plates were recharged overnight at 300 ma. The positive electrodes were carefully removed, drained, and then placed in oxygen free electrolyte for storage.

At 40°C the rate of oxygen was estimated for these plates to be of the order of 1 milliamperes hour per day. Theoretically, to get an absorbance of 0.4 a three day stand would be required for oxygen accumulation. From the initial results it was found that the oxygen evolution rate was approximately one-half the estimated rate, and that a 7 day stand was a more satisfactory interval.

1. Experimental Procedures

a. Sampling Technique

The solution containing the plates was mixed, and an aliquot removed for analysis. In brief the analytical procedure is as follows:

(1) Place 0.050 g pyrogallol in bottom of a 20mm pyrex test tube. This same tube is used later for absorbance measurements.

(2) Flush out the oxygen from the test tube with nitrogen. Raise the nitrogen tube to 1 inch from the top of the test tube.

(3) Mix the solution to be analyzed by inverting several times.

(4) Draw 25.0 ml of solution from the bottom. Add this solution to the oxygen free test tube. Withdraw the nitrogen tube and stopper immediately.

(5) Allow to stand with occasional inversion of test tube for 48 hours, then read absorbance.

b. Replacement of Electrolyte

After withdrawing the aliquot for analysis, it is necessary to replace the remaining electrolyte with oxygen-free potassium hydroxide solution. The solution is deoxygenated by sweeping out with nitrogen through a sintered glass tube. The electrolyte to be replaced is removed from the test tube. The oxygen is flushed out with nitrogen. After flushing the nitrogen tube is raised to the top of the container. The correct deoxygenated electrolyte is then placed in the container (34% KOH for series A, C, E, and lithiated electrolyte for series B) by filling to the brim. The tube is stoppered immediately forcing out the top layers of solution. The tubes are then placed in the appropriate thermostatted bath.

1. Data and Discussion

a. Polarographic Analysis of Oxygen

Aqueous solutions of KOH were prepared in 8M, 2M, 0.5M and 0.125M concentrations. These solutions were saturated with air. The air used was filtered and then passed through a KOH solution in order to prevent carbonate contamination and also to prevent concentration changes of the test solutions by evaporation.

A rotating (600 RPM) gold electrode and a Hg/HgO, KOH electrode was used to obtain polarograms. The potential change rate of the polarograph was 0.2 volt per minute. Current was recorded on a Bristol Single Pen Recorder as the drop across a 50 ohm series resistor. The recorder chart speed was 1 inch per minute. The range was 0 to 0.01 volt full scale which corresponds to 2uA per division (0.1 inch) or 200 uA full scale.

Examples of the polarograms obtained are shown in Figures 5 through 8. The polarograms for the most dilute solution, $N/8$, are normal insofar as they are reproducible and yield a well defined oxygen wave. For the more concentrated electrolyte, $N/2$ KOH, as shown in Figure 6, the limiting current region is not horizontal, and the reproducibility suffers somewhat. With the next higher electrolyte concentration level, $2N$ KOH, as shown in Figure 7 the oxygen wave is ill defined and not sufficiently reproducible to use as an analytical method.

The concentration of KOH used in hermetically sealed batteries is about $8N$. The polarogram for oxygen in an electrolyte of this concentration, Figure 8, is much different from those obtained for the more dilute electrolyte. There is a peak obtained near -0.2 volts (to the Hg/HgO electrode) that was obtained on all three polarograms. The height of this wave varied between 14 and 32 μA . The current returns to zero near -0.5 volts and in two cases out of three another small peak at -0.8 volts was obtained. At potentials more cathodic than about -0.85 volts the polarogram appears to be similar to that one would expect for hydrogen evolution. Gaseous hydrogen could not be observed, however, because the turbulence set up by the rotating electrode prevented distinguishing hydrogen bubbles.

It appears that the rotating gold electrode is appropriate for analysis of oxygen in the more dilute KOH solutions, but inadequate for determining the current-voltage relationship in $8N$ KOH. It is interesting that a peak occurred in 2 of 3 polarograms at about -0.8 volts. The potential of the Cd

electrode with respect to the Hg/HgO, KOH electrode is in the order of -0.9 volts.

2. Spectrophotometric Analysis

The data gathered so far are given in Table 14. The oxygen evolved is shown for various temperatures by each plate against the time taken for this oxygen to be evolved.

The average oxygen evolution rates are shown in Table 15 as milliampere hours per day, taken for the mean time over the interval during which oxygen was evolved.

Since the amount of data does not yet cover a long period of time, nor involve many time periods, interpretation of the data has not yet been started. After another quarter has elapsed, sufficient data should be on hand for an evaluation of the self-discharge rates.

VII. CONCLUSIONS

1. Separators

RAI material X622 and Eaton Dikeman Nylon Paper X623 are high resistance materials and will not be tested further.

The two American Felters polypropylenes gave high voltages during overcharge and high pressures, part of which is due to hydrogen. Cells with separator 960, a non-woven polyamide, gave nominal pressures (45 PSIG) and voltage (1.435V) during C/10 overcharge.

2. Uniformity

Hermetically sealed VO-6 HS cells after a 1A charge with an 8 AH input and followed by an 8 hour stand, gave 6.3 AH. At room ambient temperatures cells require a 20% overcharge to yield optimum capacity. Cells charged to the same input (12AH) yield less capacity at low charge rates.

Variations in formation such as charge rates, discharge rates, and extent of discharge, do not affect overcharge nor discharge performance of cells.

VIII. PROGRAM FOR THE NEXT INTERVAL

1. Separators

Continue separator screening tests, and testing of separators in laboratory prototype cells.

2. Uniformity

Increase input on hermetically sealed cells and measure performance characteristics.

Continue capacity determinations after charging VO-6 laboratory cells to 12AH input at various charge rates from 100 ma to 1000 ma. Also, at low rates measure capacity as a function of overcharge.

Continue ageing of formation cells, observing overcharge and discharge characteristics.

3. Stabilization of Positive Electrodes

The program will be continued as planned. In addition some of the results, especially at 75°C will be rechecked.

IX. PERSONNEL

<u>Name</u>	<u>Title</u>	<u>Hours</u>
R. C. Shair	Director of Research	32
H. N. Seiger	Head, Physical Research Section,	242
	<u>Project Engineer</u>	
H. Cohen	Research Chemist	8
R. Waldorf	Jr. Electronic Engineer	452
L. Andrews	Junior Designer	59
C. Henningsen	Jr. Engineer	208
P. B. Pinches	Research Chemist	444
		1445

TABLE I.

ELECTROLYTE ABSORPTION AND RETENTION AFTER ACCELERATION
(25G-2 min.)

<u>SEPARATOR</u>	<u>MANUFACTURER</u>	<u>THICKNESS</u> <u>mils</u>	<u>ELECTROLYTE ABSORBED</u> <u>WT. 34% KOH/WT. SEP.</u>	<u>% ELECTROLYTE</u> <u>RETAINED</u> <u>AFTER ACCELERATION</u>
X622	R. A. I.	5.0*	0.9	97
X623	EATON DIKEMAN	4.5	0.2	73
X624	PELLON	7.2	6	99

*Thickness varied between 4.0 and 7.2 mils.

TABLE II.

SEPARATOR RESISTANCE IN 34% KOH. SAMPLES SOAKED
24 HOURS AT ROOM TEMPERATURE

SEPARATOR	RESISTANCE, MILLIOHMS DUE TO 1 in. ² OF SAMPLES.
X622	167
X623	60*
X624	7

*One sample was not completely wetted; the above value is an
average of the remaining two pieces that did wet completely.

TABLE III.

VOLTAGES AND PRESSURES OF LABORATORY SEPARATOR CELLS
DURING 0.6A CHARGE AT 77°F (24.6 AH INPUT)

CELL NO.	END OF CHARGE VOLTAGE	END OF CHARGE PRESSURE (PSIG)
960-1	1.44	48
960-2	1.44	40
960-3	1.43	57
960-4	1.43	33
Average	1.435	45
661-5	1.57	Vented*
661-6	1.58	Vented
661-8	1.56	Vented
661-9	1.55	Vented
Average	1.565	Vented
662-10	1.58	Vented
662-11	1.57	Vented
662-12	1.58	50
662-13	1.56	Vented
662-14	1.58	Vented
Average	1.576	Vented

*Cells vented to atmosphere when pressures exceeded 150 PSIG

CAPACITIES OF LABORATORY SEPARATOR CELLS AFTER 0.6A CHARGE AT 77°F
TO A 24.6AH INPUT. DISCHARGED AT 3A to 1.0 VOLT

CELL NO.	CAPACITY, AH.
960-1	7.25
960-2	6.85
960-3	6.65
960-4	5.65
Average	6.60
661-5	6.10
661-6	6.75
661-8	6.45
661-9	6.10
Average	6.35
662-10	6.70
662-11	6.55
662-12	6.15
662-13	6.45
662-14	5.50
Average	6.27

TABLE V.

VO-6 HS CELLS, PILOT PLANT DATA

CELL NO.	1st. 0.5A CHARGE 24 HRS.		2nd. 0.5A CHARGE 24 HRS.		CAPACITY, AH 3A to 1.0 V SECOND CYCLE
	V	P(PSIG)	V	P(PSIG)	
354	1.40	44	1.50	40	> 6
355	1.45	18	1.42	15	6.99
356	1.50	0	1.42	5	6.00
358	1.50	3	1.42	3	7.23
359	1.40	78	1.50	19	> 6
360	1.50	25	1.42	20	7.50
362	1.50	18	1.42	13	7.50
364	1.40	32	1.50	60	> 6
369	1.40	94	1.45	40	> 6
371	1.40	49	1.45	32	> 6

TABLE VI.

VO-6HS Cells, Capacity after a One (1) ampere -
8 hour charge. Discharge rate 1 ampere with
1.0 volt end-point. Conditions are room temper-
ature with an 8 hour stand in one instance, and no
open circuit stand in the second instance.

<u>Cell No.</u>	<u>A.H. Capacity 8 Hr. Stand</u>	<u>A.H. Capacity No Stand</u>
354	6.00	5.20
355	7.52	5.45
356	4.67	4.51
358	7.63	6.45
359	6.32	5.20
360	7.65	6.63
362	7.70	6.48
~~364~~	5.27	4.58
369	4.72	4.21
371	5.60	5.08
Average	6.31	5.38

TABLE VII.

First Discharge Capacity During Formation
Groups A, B, E, F discharged at 3A, and
Groups C, D, G, H discharged at 0.5A

GROUP	A	B	C	D	E	F	G	H
POSITIVES								
	7.05	7.55	7.08	7.00	7.60	6.95	6.58	6.90
	7.05	7.20	7.33	6.83	7.80	7.40	6.88	6.90
	7.25	7.05	7.25	6.75	6.90	7.45	6.72	6.80
	7.05	7.05	7.31	7.00	7.40	7.10	6.66	6.80
	7.20	7.80	7.37	7.00	7.40	6.95	6.50	6.80
					7.40	6.95	6.42	6.58
AVERAGE	7.12	7.33	7.27	6.92	7.42	7.13	6.63	6.80
NEGATIVES								*
	10.8	10.8	10.00	10.00	8.95			
	10.8	10.8	10.19	10.19	9.50			
	10.8	10.8	10.00	10.00	9.55			
	10.8	10.8	10.42	10.42	9.55			
	10.8	10.8	10.50	10.50	9.20			
					9.25			
AVERAGE	10.8			10.22		9.33		
RATIO, N/P	1.47			1.47		1.31		

* DVM failure prevented determination

TABLE VIII.

Second Discharge Capacity During Formation
Groups A, B, E, F discharged at 3A
Groups C, D, G, H discharged at 0.5A

GROUP	A	B	C	D	E	F	G	H
POSITIVES	<u>5.20</u>	8.00	7.92	8.08	8.15	8.20	7.37	7.60
	7.90	7.65	7.98	8.07	7.95	8.25	7.48	7.55
	7.90	7.95	8.01	8.01	7.80	8.20	7.40	7.48
	7.85	8.05	8.01		7.85	8.15	7.28	7.45
	8.20	7.15	8.18	8.17	8.00	8.10	7.36	7.39
					8.05	8.25	7.27	7.38
AVERAGE	7.96	7.76	8.02	8.08	7.97	8.19	7.36	7.48
NEGATIVES								
		10.2		9.37		10.30		9.63
		10.2		9.37		10.30		9.88
		10.2		9.37		10.55		9.46
		10.2				10.60		9.79
		10.2		9.37		10.30		9.79
						10.30		9.71
AVERAGE		10.2		9.37		10.39		9.71
RATIO N/P		1.31		1.16		1.29		1.30

Underlined values omitted from average

TABLE IX.

Third Discharge Capacity During Formation
Groups A, B, E, F discharged at 3A
Groups C, D, G, H discharged at 0.5A

GROUP	A	B	C	D	E	F	G	H
POSITIVES	8.40	7.95	7.83	8.46	9.05	<u>5.85</u>	7.38	7.54
	7.90	8.00	8.13	8.46	8.00	9.00	7.23	7.38
	7.85	8.15	8.18	8.46	8.05	8.30	7.36	7.45
	7.85	8.15	8.13		8.20	9.05	7.23	7.34
	8.10	8.30	8.20	8.57	8.10	9.00	7.33	7.28
AVERAGE	8.02	8.11	8.09	8.49	8.28	8.88	7.31	7.38
NEGATIVES	11.50	9.85	<u>7.83</u>	11.33	12.00	11.65	11.18	10.37
	11.50	9.75	12.85	11.25	11.55	11.95	10.90	10.53
	11.75	10.20	12.81	11.25	11.40	11.75	11.03	10.09
	11.60	10.15	12.68		11.75	12.25	10.90	10.34
	12.00	10.20	12.67	11.38	11.65	12.05	11.05	10.42
AVERAGE	11.67	10.03	12.75	11.30	11.67	11.93	10.97	10.34
RATIO N/P	1.45	1.24	1.58	1.33	1.41	1.34	1.50	1.40

Underlined values omitted from average

TABLE X.

FIRST - OVERCHARGE CHARACTERISTICS AT 0.6A
(42AH INPUT) AND DISCHARGE CAPACITY AT 3A, T = 77°F

GROUP	C	E	F	U
VOLTAGE	1.405	1.390	1.410	1.412
	1.420	<u>1.53</u>	1.405	1.415
	1.422	1.420		1.425
	1.424	1.402	1.399	
	1.411	1.408	1.398	
			1.405	
AVERAGE	1.416	1.405	1.401	1.417
PRESSURE PSIG	17	70	101	35
	69	46	61	12
	70	102	51	64
	88	45		
	53	55	48	
			63	
AVERAGE	59	64	65	37
CAPACITY	6.40	6.25	6.40	6.95
	6.25	7.95	7.05	7.10
	6.35	6.25		6.25
	6.95	6.75	7.20	
	7.55	6.75	7.20	
			6.60	
AVERAGE	6.70	6.79	6.89	6.77

TABLE XI.

SECOND - OVERCHARGE CHARACTERISTICS AT 0.6A (56AH INPUT)
AND DISCHARGE CAPACITY AT 3A, T = 77°F

GROUP	C	E	F	U
VOLTAGE	1.42	1.39	<u>1.50</u>	1.42
	1.43	<u>1.52</u>	1.41	1.42
	1.43	1.49		1.43
	1.43	1.41	1.43	
	1.42	1.41	1.41	
			1.46	
AVERAGE	1.426	1.425	1.427	1.423
PRESSURE PSIG	25	96	109	43
	106	58	97	26
	94	115		65
	119	103	90	
	75	108	109	
			76	
AVERAGE	84	98	96	45
CAPACITY AH	5.60	5.30	7.45	6.35
	7.15	7.50	6.75	5.75
	7.20	7.35		6.50
	7.10	5.95	5.90	
	6.95	5.70	5.80	
			7.25	
AVERAGE	6.80	6.36	6.63	6.20

Table XII.

Pressure Variations During 0.6A Overcharge at 77°F
 32AH Input at 0.3A, 191AH Input at 0.6A

Cells	Pressure PSIG at XAH input								
	50	64	78	121	135	150	164	180	223
C1	23	22	22	22	21	20	22	21	20
C2	93	91	96	95	93	90	94	93	89
C3	85	83	82	85	83	80	83	81	79
C4	111	110	107	101	97	93	94	92	86
C5	86	87	86	83	82	80	83	82	79
E1	84	84	83	81	80	78	81	79	76
E2	42	46	45	48	47	44	51	48	49
E3	100	99	96	96	94	94	96	92	96
E4	89	92	91	92	89	82	91	89	87
E5	124	122	122	124	122	118	122	121	117
F1	60	59	58	57	56	54	62	56	55
F2	80	82	81	81	79	77	80	78	76
F4	87	85	83	79	76	74	76	74	72
F5	100	101	100	99	97	94	97	95	93
F6	73	76	76	74	72	69	72	71	68
U1	45	43	43	41	39	37	38	32	36
U2	31	32	31	32	30	29	32	30	29
U3	59	58	57	58	57	54	62	57	55
U4	5	9	4	4	4	3	3	7	7
U5	36	36	37	39	33	36	38	37	37
U6	34	38	34	35	34	33	34	33	33
U7	11	17	18	19	18	17	17	17	17
U8	47	47	47	49	49	46	49	48	48
Ave.	65	66	65	65	63	61	64	62	61

TABLE XIII.
Continued

PLATE	WEIGHT GAIN	THEORETICAL CAPACITY (AH)	DISCHARGE CAPACITY 0.225A (AH)
B 1	1.4630	.424	
B 4	1.8515	.536	.75
B 9	1.7552	.508	.68
B 10	1.7097	.491	.71
B 14	1.6004	.464	.74
B 17	1.6877	.489	.63
B 20	1.6877	.489	.67
B 23	1.6663	.482	.68
B 27	1.6532	.478	.63
B 30	1.7821	.517	.65
B 33	1.6010	.463	
B 37	1.7341	.501	
B 40	1.6623	.481	.63
B 41	1.8073	.522	
B 42	1.7572	.509	.72
B 45	1.6175	.468	.63
B 49	1.5957	.462	
B 50	1.6292	.472	.56
B 56	1.4665	.425	
B 60	1.6202	.468	.62

TABLE XIII.

WEIGHT GAINS As Ni (OH)₂ AND DISCHARGE
CAPACITY OF SINTERED NICKEL PLATES FOR
STABILIZATION STUDIES

PLATE	WEIGHT GAIN	THEORETICAL CAPACITY (AH)	DISCHARGE CAPACITY 0.225A (AH)
A 3	1.3706	.396	
A 5	1.3691	.396	.52
A 10	1.3728	.396	
A 13	1.6786	.486	.76
A 15	1.8032	.522	
A 16	1.7108	.495	.71
A 17	1.7181	.496	.67
A 20	1.6687	.483	.63
A 22	1.6547	.478	.67
A 23	1.8604	.538	
A 24	1.7607	.510	.67
A 25	1.7304	.500	.67
A 26	1.8778	.544	.67
A 27	1.6714	.484	
A 30	1.5862	.458	
A 37	1.5552	.450	
A 40	1.5836	.451	
A 49	1.7440	.502	.77
A 50	1.7399	.502	.67
A 52	1.5350	.444	
A 54	1.6227	.472	.56
A 59	1.7795	.515	.78
A 60	1.7316	.501	.71

TABLE XIII.
Continued

PLATE	WEIGHT GAIN	THEORETICAL CAPACITY (AH)	DISCHARGE CAPACITY 0.225A (AH)
C 1	1.6477	.477	.63
C 4	1.6518	.478	.67
C 8	1.6471	.477	.71
C 10	1.5544	.450	
C 11	1.5702	.455	
C 15	1.6207	.468	.67
C 20	1.7012	.482	.77
C 22	1.7510	.508	
C 24	1.7053	.484	.74
C 30	1.6520	.478	.67
C 31	1.7611	.510	
C 35	1.7753	.512	.67
C 36	1.7347	.500	.78
C 40	1.6470	.476	.67
C 45	1.6381	.474	.56
C 46	1.5686	.454	
C 47	1.7814	.516	.77
C 48	1.8558	.538	
C 50	1.6598	.481	.71
C 55	1.6805	.486	.77
C 60	1.5512	.448	

TABLE XIII.
Continued

PLATE	WEIGHT GAIN	THEORETICAL CAPACITY (AH)	DISCHARGE CAPACITY 0.225A (AH)
E 1	1.8221	.528	.79
E 4	1.6839	.488	
E 7	1.7738	.512	.56
E 10	1.7669	.512	.45
E 13	1.9159	.559	.80
E 15	1.6899	.489	.50
E 20	1.6094	.466	.50
E 27	1.6819	.487	.50
E 30	1.7112	.495	
E 36	1.6566	.478	.45
E 41	1.6468	.476	
E 42	1.7366	.501	
E 43	1.6397	.474	
E 44	1.6025	.468	.41
E 46	1.7262	.500	
E 50	1.6100	.466	.41
E 53	1.8031	.522	.79
E 55	1.6729	.484	.41
E 59	1.6201	.469	.73

TABLE XIV

OXYGEN ANALYSIS

MICROMOLES OXYGEN (μM) AFTER Δt DAYS ACCUMULATION

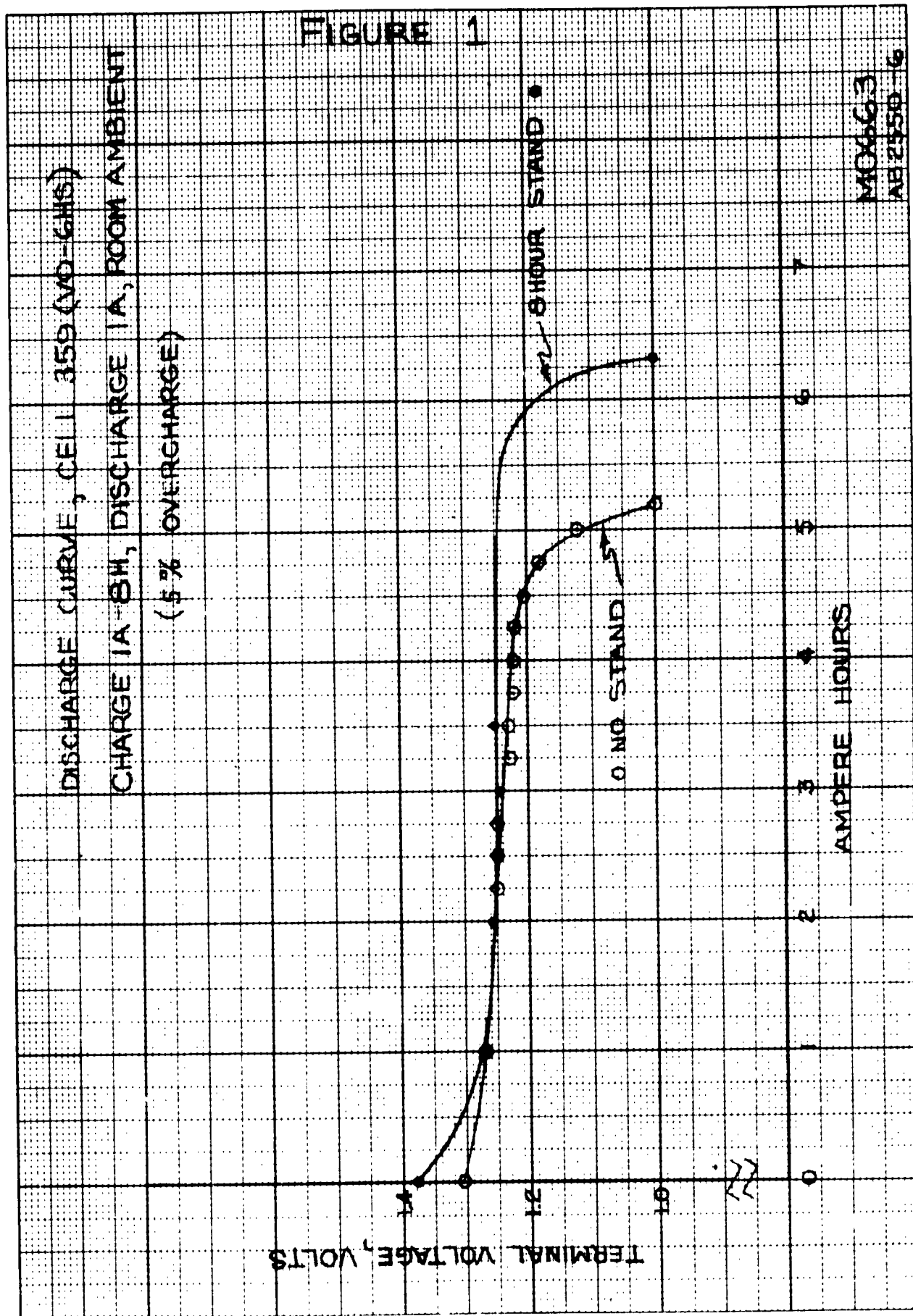
Temp. C	Plate	Δt	O_2 , μM	Δt	O_2 , μM	Δt	O_2 , μM	Δt	O_2 , μM
10	A 16	10	21.0	6	9.3				
10	B 60	10	20.2	6	10.4				
10	C 15	10	22.6	6					
10	E 7	10	22.6	6	8.7				
30	A 20	8	22.6	7		14	7.0	7	9.8
30	B 17	8	26.6	7	13.3	14	7.0	7	9.8
30	C 1	8	22.0	7	15.8	14	2.3	7	12.8
30	E 15	8	22.0	7	15.1	14	5.8	7	9.8
40	A 25	8	26.6	8	11.0	13	6.4	7	8.2
40	B 45	8	26.6	8	5.2	13	3.5	7	12.1
40	C 24	8	26.0	8	9.3	13	6.4	7	8.7
40	E 20	8	26.0	8		13	8.7	7	9.3
50	A 24	10	55.8	6	13.3	13	5.2	7	10.4
50	B 10	10	67.2	6	13.3	13	11.0	7	10.4
50	C 30	10	55.8	6	29.0	13	9.3	7	11.6
50	E 27	10	78.8	6	7.6	13	10.4	7	5.2
60	A 17	8		8	27.2	13	1.7	7	7.6
60	B 14	8		8	20.9	13	4.7	7	5.8
60	C 8	8	78.8	8	20.2	13	1.2	7	10.4
60	E 36	8	84.5	8	19.7	13		7	10.4
75	A 22	10	27.8			13	7.6	7	5.2
75	B 50	10	41.7			13		7	7.6
75	C 4	10	29.5			13	5.2	7	
75	E 10	10	31.7			13	8.7	7	9.8

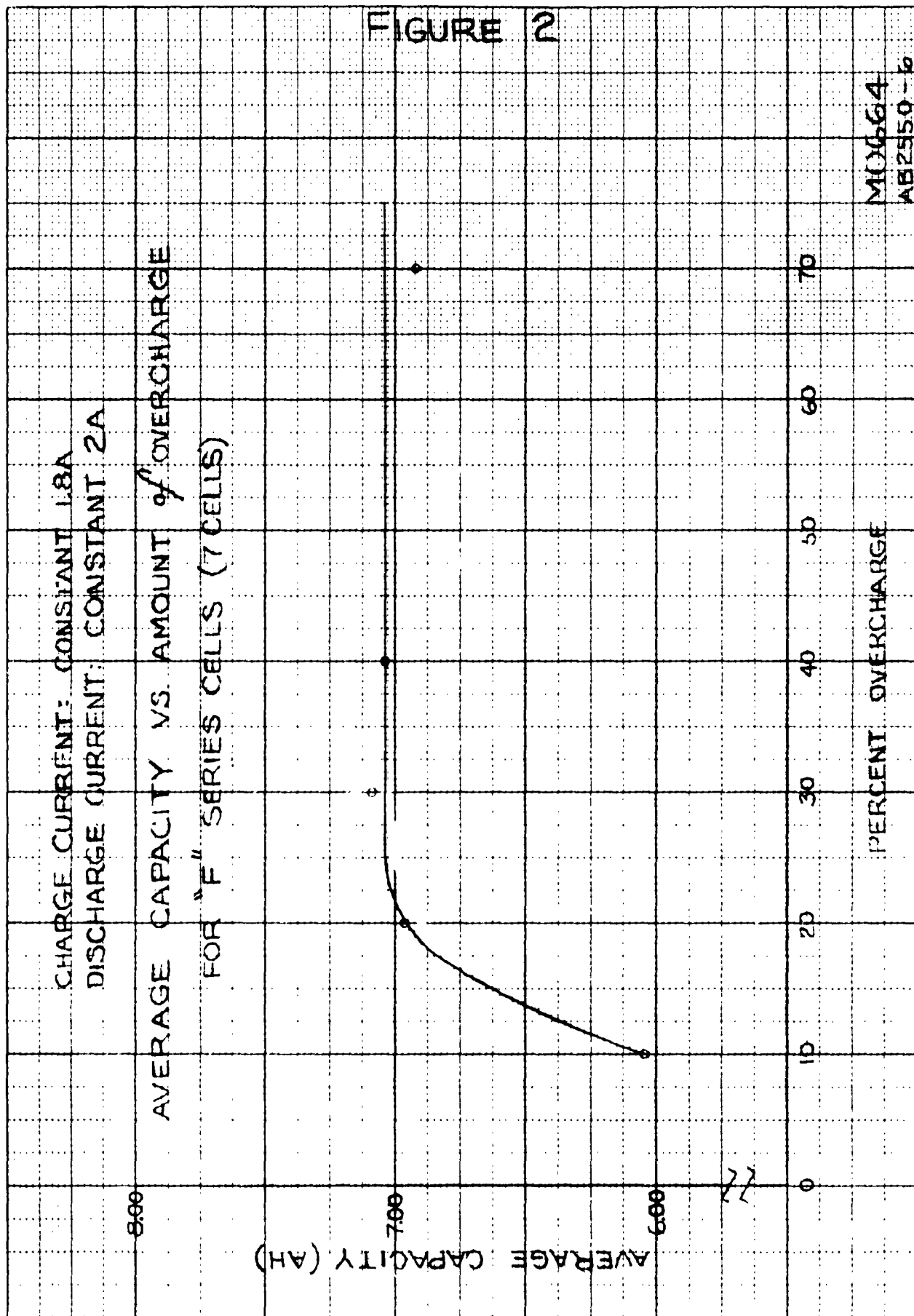
TABLE XV.

OXYGEN EVOLUTION RATES, R, mah per day.

 $\bar{t}_c$ = MEAN TIME OF ACCUMULATION

Temp. C	Plate	$\bar{t}_c$	R	$\bar{t}_c$	R	$\bar{t}_c$	R	$\bar{t}_c$	R
10	A 16	5	0.23	13	0.17				
10	B 60	5	0.21	13	0.19				
10	C 15	5	0.24	13					
10	E 7	5	0.24	13	0.15				
30	A 20	4	0.30	12.5		22	0.05	32.5	0.14
30	B 17	4	0.35	12.5	0.20	22	0.06	32.5	0.14
30	C 1	4	0.29	12.5	0.23	22	0.03	32.5	0.19
30	E 15	4	0.29	12.5	0.23	22	0.04	32.5	0.14
40	A 25	4	0.35	12.0	0.15	22.5	0.05	32.5	0.13
40	B 45	4	0.35	12.0	0.06	22.5	0.04	32.5	0.19
40	C 24	4	0.34	12.0	0.12	22.5	0.10	32.5	0.13
40	E 20	4	0.34	12.0		22.5	0.07	32.5	0.13
50	A 24	5	0.58	13.0	0.23	22.5	0.04	32.5	0.16
50	B 10	5	0.71	13	0.23	22.5	0.08	32.5	0.16
50	C 30	5	0.58	13	0.50	22.5	0.09	32.5	0.17
50	E 27	5	0.84	13	0.13	22.5	0.08	32.5	0.07
60	A 17	4		12	0.36	22.5	0.02	32.5	0.11
60	B 14	4		12	0.28	22.5	0.04	32.5	0.09
60	C 8	4	1.05	12	0.26	22.5	0.02	32.5	0.16
60	E 36	4	1.12	12	0.26	22.5	0.25	32.5	0.16
75	A 22	5	0.29			22.5	0.06	32.5	0.07
75	B 50	5	0.44			22.5	0.02	32.5	0.09
75	C 4	5	0.31			22.5	0.04	32.5	
75	E 10	5	0.33			22.5	0.07	32.5	0.14



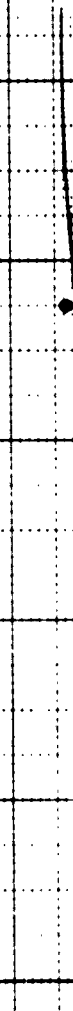


CAPACITY OF LABORATORY CELLS (HS SERIES) AFTER CHARGE AT VARIOUS RATES

CHARGE INPUT WAS 12 AH AT 77°F
DISCHARGE AT 3A TO 1.0 VOLT

FIGURE 3

DISCHARGE CAPACITY, A.H.



CHARGE RATE, MA
CHARGE RATE, C/H

1000 C/H
800 C/H
600 C/H
400 C/H
200 C/H

1000 C/H
800 C/H
600 C/H
400 C/H
200 C/H

MOG 65
AB2550-6

FIGURE 4

EXPERIMENTAL DESIGN OF FORMATION EXPERIMENT

FORMATION - DISCHARGE TO 1.0 VOLTS

		DISCHARGE	
		FAST	SLOW
CHARGE	SLOW	A	C
	FAST	E	G

FORMATION - REVERSAL ON DISCHARGE

		DISCHARGE	
		FAST	SLOW
CHARGE	SLOW	B	D
	FAST	F	H

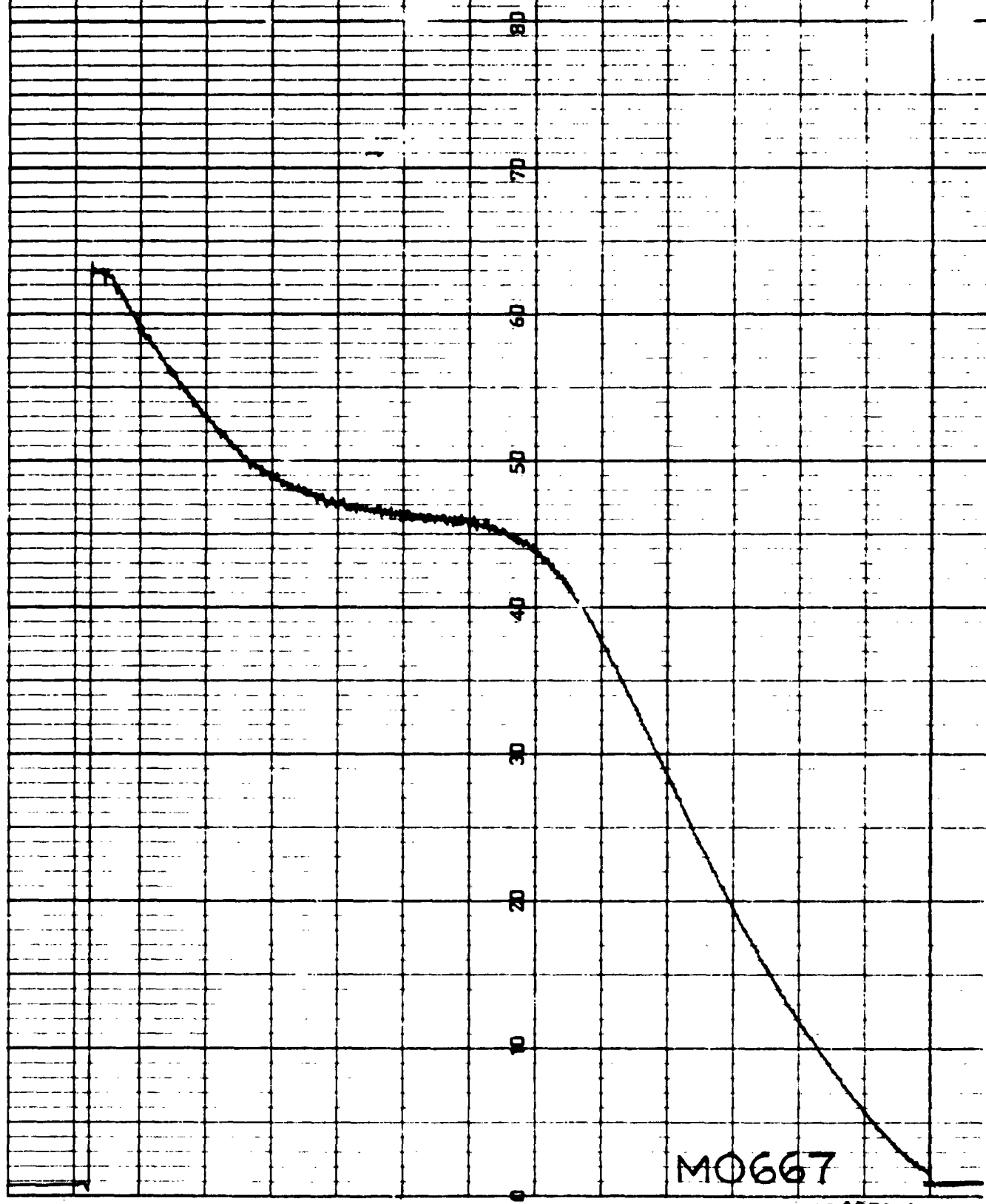
FAST CHARGE = 2.5A - 6H = 15AH

FAST DISCHARGE = 3A

SLOW CHARGE = 0.6A - 25H = 15AH

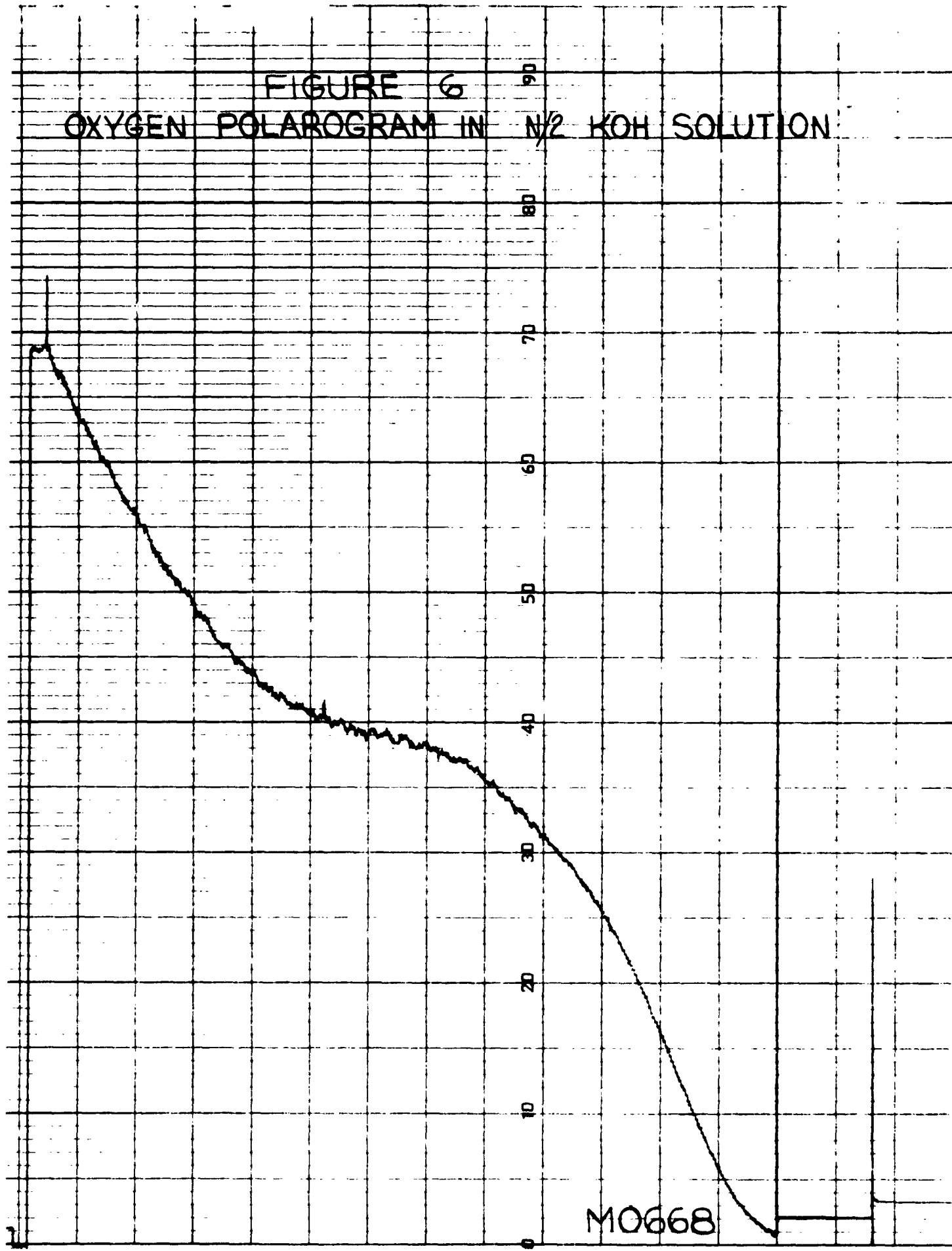
SLOW DISCHARGE = 0.5A

FIGURE 5 OXYGEN POLAROGRAM IN $N/8$ KOH SOLUTION



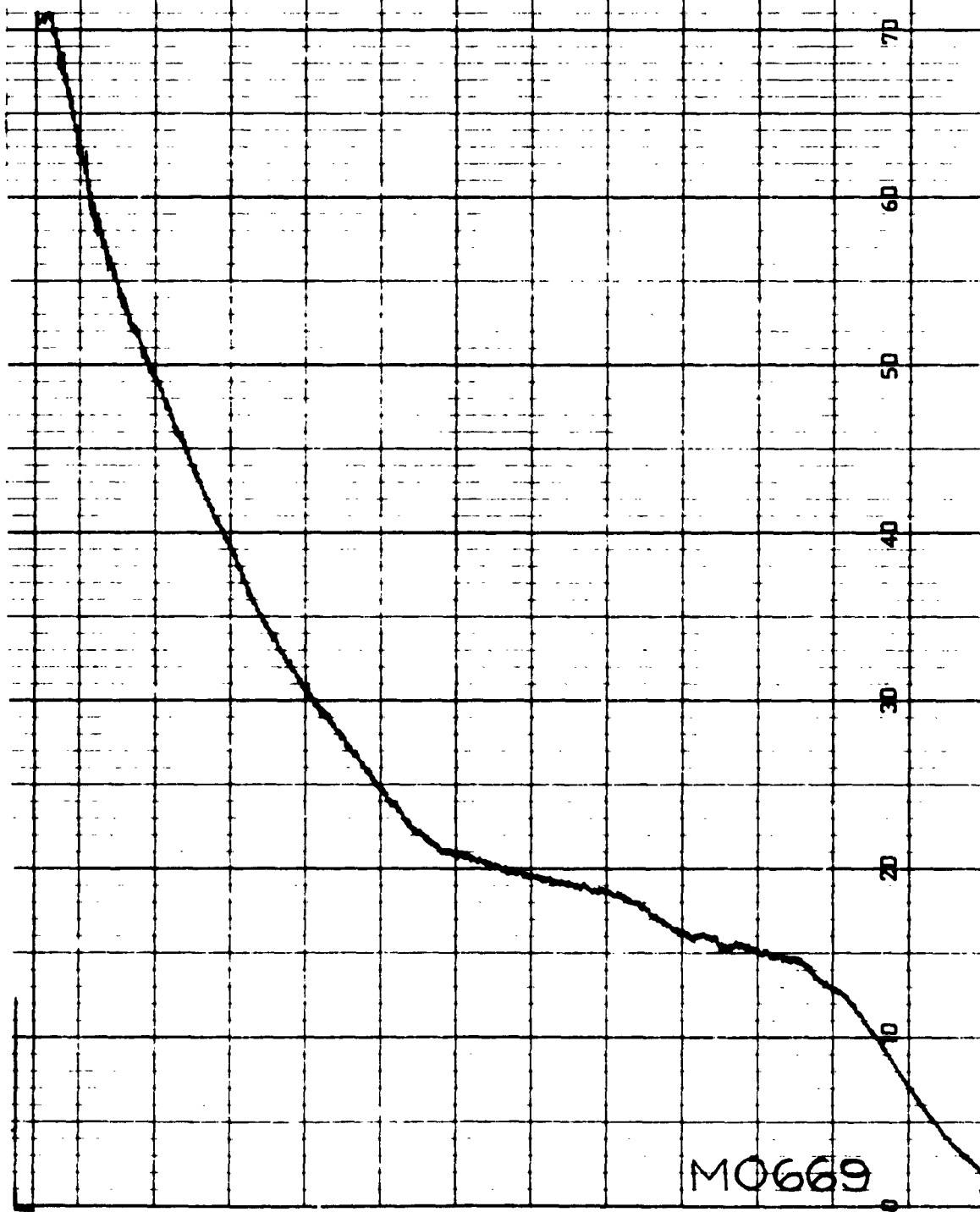
MO667

FIGURE 6
OXYGEN POLAROGRAM IN $N/2$ KOH SOLUTION



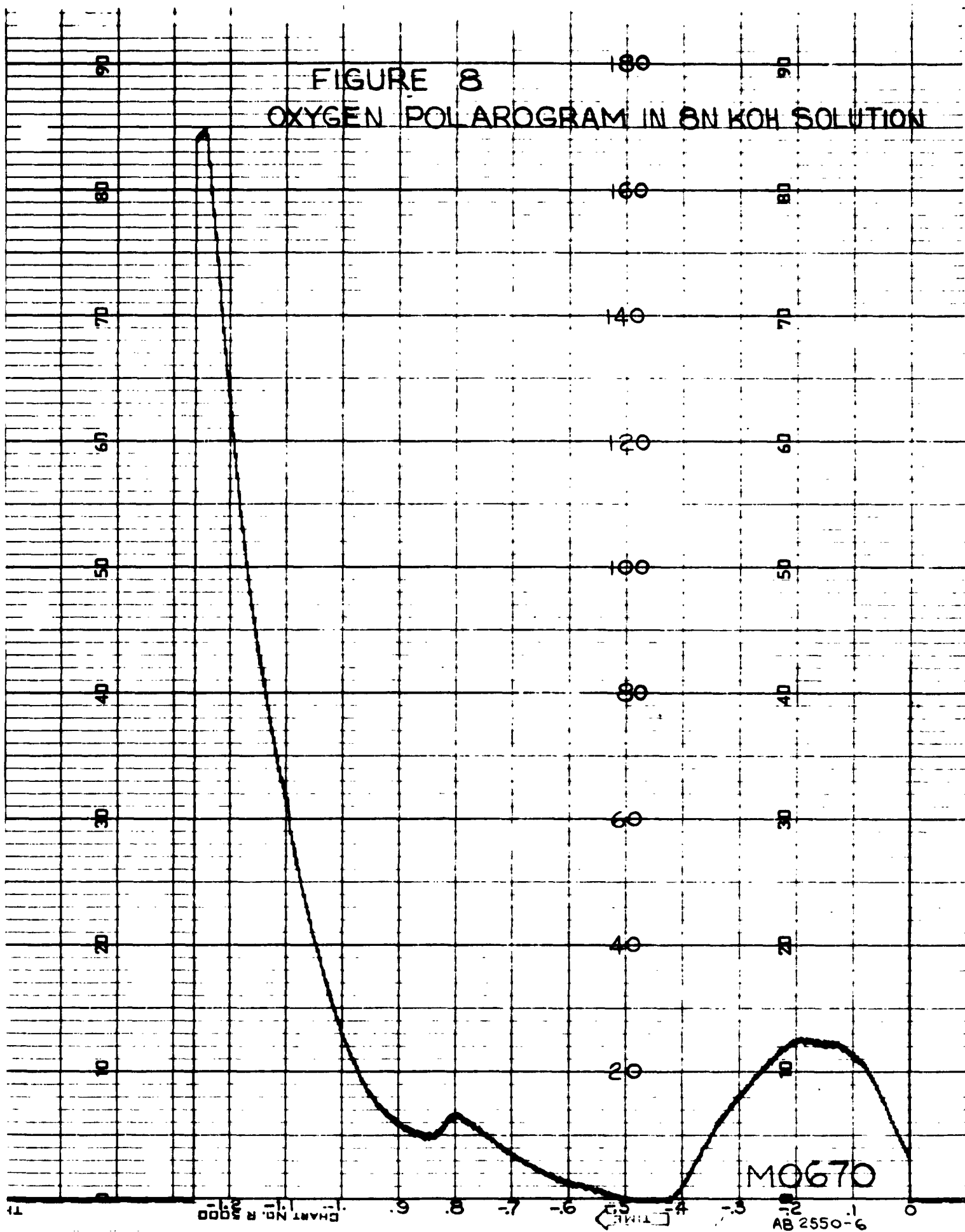
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FIGURE 7
OXYGEN POLAROGRAPH IN 2N KOH SOLUTION



M0669

FIGURE 8
OXYGEN POLAROGRAM IN 8N KOH SOLUTION



AD	Accession No	UNCLASSIFIED
Gulton Industries, Inc., Metuchen, New Jersey Development of Improved Sealed NICKEL-CADMIUM BATTERIES H. N. Seliger, R. C. Shair Sixth Quarterly Report, p October 1961 to 31 December 1961 48 pp., 8 illus., 15 Tables Signal Corps Contract DA-36-039 SC-85390 DA Project No. 3A990002, Unclassified Report		1. Batteries - sealed 2. Batteries - Nickel - Cadmium Contract DA-36-039 SC-85390
Screening tests were started on three new separator materials, (1) modified polypropylene from RA1, (2) a nonwoven nylon paper from Eaton Dileman, and (3) a modified nonwoven polyimide from Pellon. The Pellon material is the only one of these three which appears satisfactory for continued testing.		
Laboratory cells of the VO-6 type were prepared with two kinds of polypropylene separator and with a nonwoven polyimide material. The pressure during a C/10 overcharge of the cells with polypropylene material reached over 150 PSIG. They were vented to the atmosphere and the gas was found to contain hydrogen as well as oxygen. The voltages during this overcharge averaged about 1.56 volts. The cells with the polyimide separator averaged 45 PSIG and 1.43 volts.		
Hermetically sealed cells, VO-6HS, were charged for 8 hours at 1.0 amperes. The capacity obtained appears to indicate insufficient charge. An overcharge versus discharge capacity curve at room temperature indicates that about a 20% overcharge is required.		
A format on study was instituted. The results indicate that capacity, voltage and pressures subsequent to formation are not significantly dependent upon the mode of formation charge (fast or slow), the mode of discharge (fast or slow), nor does it depend upon whether the formation discharge is carried to the exhaustion of the negatives.		
The program for the study of the slow self-discharge process is underway. Oxygen analysis were run by the spectrometric procedure. The electrochemical procedure requires a dilution of the electrolyte to obtain reproducible results, but this procedure consequently reduced the sensitivity of the analysis.		

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Hermetically sealed cells, VO-6HS, were charged for 8 hours at 1.0 amperes. The capacity obtained appears to indicate insufficient charge. An overcharge versus discharge capacity curve at room temperature indicates that about a 20% overcharge is required.		
A format on study was instituted. The results indicate that capacity, voltage and pressures subsequent to formation are not significantly dependent upon the mode of formation charge (fast or slow), the mode of discharge (fast or slow), nor does it depend upon whether the formation discharge is carried to the exhaustion of the negatives.		
The program for the study of the slow self-discharge process is underway. Oxygen analysis were run by the spectrometric procedure. The electrochemical procedure requires a dilution of the electrolyte to obtain reproducible results, but this procedure consequently reduced the sensitivity of the analysis.		

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Screening tests were started on three new separator materials, (1) modified polypropylene from RA1, (2) a nonwoven nylon paper from Eaton Dileman, and (3) a modified nonwoven polyimide from Pellon. The Pellon material is the only one of these three which appears satisfactory for continued testing.		
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