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DEVELOPMENT OF A NICKEL CADMIUM STORAGE CELL IMMUNE TO

DAMAGE FROM OVERDISCHARGE AND OVERCHARGE

By A. J. Catotti and M. D. Read

June 21, 1965

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1.0 <u>Introduction</u>

Conventional sealed secondary cells cannot be safely overdischarged because of the high gas pressures which are generated during cell reversal. Experience with fuel cell type of gas electrodes indicates that these gases could be recombined under proper conditions.

The purpose of this contract, therefore, was to develop and build nickel-cadmium cells which would be immune to damage from overdischarge and overcharge. This was to be accomplished by the selection of suitable gas electrodes so that: (1) the onset of overcharge could be detected, and (2) the cell would not be damaged by repeated overdischarges.

2.0 Technical

2.1 Charge Control

Most sealed nickel-cadmium cells are designed so that they are positive limiting on charge. Thus oxygen is liberated at the positive nickel hydroxide electrode after the positive electrode is charged,

 $20H^- \longrightarrow 1/2 \ 0_2 + H_20 + 2e^-$ (positive electrode)

but before the negative electrode is fully charged. As soon as a sufficient oxygen pressure has been generated, oxygen will be reduced at the negative cadmium electrode.

 $1/2 \ 0_2 + H_20 + 2e^- \longrightarrow 20H^-$ (negative electrode)

Thus a conventional sealed cell is capable of continuous overcharge at moderate rates.

Now, if a high performance gas electrode is incorporated into the cell as in Figure 1, it will be held at the cadmium potential when no gas is present.

However, as soon as oxygen is present within the cell, the gas electrode will become an oxygen electrode and will have a positive potential with respect to the cadmium electrode. A potential will thus be detected across resistor R_1 . This potential, or signal, may be used to cut off or regulate the charge current.

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This concept has been recognized for some time (e.g.⁽¹⁾). A number of designs and ratings have been built utilizing this concept and are currently being evaluated on various test programs. These units are often referred to in present-day terminology as third electrode (or 3e⁻) units.

The type, size and placement of the control electrode employed in cells for this contract were based on this previous experience.

2.2 Protection Against Overdischarge

2.2.1 Cell Reversal

When a battery of cells is completely discharged, the lowest capacity cells will be overdischarged and consequently go into reverse, i.e., the normally positive electrode will become negative and vice versa. If the charged state of the positive is such that it is limiting on discharge (i.e., its charged state is less than the negative), hydrogen will be evolved at the nickel hydroxide electrode. If, on the other hand, the negative electrode is limiting on discharge, oxygen will be evolved on the cadmium electrode during cell reversal. The cell potential during reversal when only one electrode has reversed will be negative 0.1 to 0.2 volts depending on rate of overdischarge. If both electrodes are reversed, both hydrogen and oxygen will be evolved and the cell potential will be negative 1.5 volts or more.

Consequently, by regulating the relative states of charge of the two electrodes, it is possible to predetermine which electrode will be limiting on discharge and what gas will be present.

2.2.2 Overdischarge Protection Using an Oxygen Cycle

As noted above, it is possible to design a cell which would be negative limiting on discharge so that on overdischarge oxygen will be evolved on the cadmium (normal negative) electrode. In this case an auxiliary electrode would be connected to the nickel hydroxide (normal positive) electrode. During overdischarge the following reactions would then occur:

 $20H^{-} \longrightarrow 1/2 \ 0_{2} + H_{2}0 + 2e^{-}$,

at the cadmium, and the reverse reaction at the auxiliary,

⁽¹⁾W. N. Carson, Jr. and J. M. McQuade, "The Use of Auxiliary Electrodes in Sealed Cells," Extended Abstract No. 10, Battery Division, Electrochemical Society, New York, October 1963.

$$1/2 \ 0_2 + H_2 0 + 2e^- \longrightarrow 20H^-$$
,

with the electrons flowing through the external circuit. The disadvantages of this scheme are that some oxygen will be evolved on the auxiliary during charging, resulting in (1) a loss of charge efficiency; (2) the possibility of a premature oxygen signal at the control electrode; and (3) a degradation of the gas electrode which is not designed for oxygen evolution. Further, the Niedrach-Alford electrodes planned for these cells are generally better hydrogen electrodes than they are oxygen electrodes. Consequently, it was decided to design the units around a hydrogen cycle.

2.2.3 Overdischarge Protection Using a Hydrogen Cycle

As indicated above, when a positive limiting cell is overdischarged, the normally positive electrode will become negative until it reaches the hydrogen potential and starts to evolve hydrogen,

 $2H_2O + 2e^- \longrightarrow H_2 + 2OH^-$.

It is possible to recombine this hydrogen by connecting an auxiliary gas electrode to the normally negative electrode as in Figure 1. It is readily apparent that this is the same configuration used for overcharge control. The essential differences are (1) that a smaller resistor is used to make this connection and (2), large gas electrodes are employed.

When sufficient hydrogen is present, the auxiliary (hydrogen) electrode will become more negative than the cadmium electrode and hydrogen will be oxidized in preference to the cadmium; that is,

$$H_2 + 20H^- \longrightarrow 2H_20 + 2e^-$$
,

and not

$$Cd + 2OH \longrightarrow Cd(OH)_2 + 2e$$
.

At low hydrogen pressures, the recombination will be slow since the only driving force will be (1) the difference between the $E^{O's}$ of about 20 mv and (2), the difference between the polarization voltages. Further, it is recognized that R_1 must be very low since any voltage drop will reduce the driving force.

During initial planning, it was proposed to use a diode in place of R_1 . It was found, however, that the instantaneous forward voltages of available diodes would be too high to permit the reaction to proceed at reasonable rates. One reason for proposing a diode here was to prevent the formation of hydrogen during the charging mode. As will be demonstrated subsequently, this concern was unwarranted.

Since the recombination rate is proportional to the difference in voltage between the cadmium electrode and the auxiliary and the total resistance in this loop, it is preferable to have the cadmium electrode become completely discharged shortly after the nickel hydroxide electrode. Thus as the cadmium electrode becomes more positive, approaching the oxygen potential, the recombination reaction will increase dramatically.

3.0 Experimental

3.1 Gas Electrode Selection

It was anticipated that we would use Niedrach-Alford type gas electrodes in these cells. When these electrodes are used in fuel cell applications, one side is supplied with gas at a constant pressure with the opposite face continuously contacting the electrolyte. Under these conditions, they are capable of operating at very high current densities--up to 1000 mA/cm^2 . As used in sealed cells, a gas face is provided by using a corrugated, perforated plastic against one side. The other side faces the electrolytesaturated porous separator.

In order to test the suitability of materials and construction, polarization runs were made with test cells to simulate normal operating conditions.

3.1.1 Polarization Data

The various gas electrodes were tested by building a simple test cell which consisted of a sandwich of a gas electrode and plate held between two blocks of Lucite as shown in Figure 2.

The separator was saturated with 31% potassium hydroxide. The cell (or cells) were then placed in a vacuum chamber equipped with electrical lead-throughs, a vacuum pulled and the system backfilled with the desired atmosphere.

Several types of polarization runs were made. The more significant data is given in Tables 1 - 4.

In Table 1, data is given for three standard types of electrodes in oxygen at room temperature. The discharge of the gas electrodes was driven against a partially charged nickel hydroxide electrode. In this case, the voltages given include the polarization voltage (although small) of charging the nickel hydroxide electrode. Throughout this run the current was generally held constant at a particular pressure, allowing the voltage to stabilize. The pressure was then reduced, while holding the current constant. This was repeated a number of times down to a pressure of about 1 psia, at which time the pressure was increased to 13 psia. The current was then incrementally increased and the whole process repeated. The results showed the three materials to be quite comparable as oxygen electrodes under the conditions of test. Similar data are given in Table 2 for three samples of gas electrodes prepared on General Electric sintered nickel. These preliminary samples were not as good as the previous ones tested.

One sample from the previous group was tested as a hydrogen electrode. In this case, the hydrogen electrode was discharged against a charged positive nickel hydroxide electrode. Data are given in Table 3. In the first set of data, the current was held constant and the pressure reduced. In the second set of data, the pressure was maintained and the current increased in steps. As a hydrogen electrode, this material appeared to be severely limited at low gas pressures.

In another series of tests, a Niedrach-Alford type electrode made by the Advanced Technology Laboratories of the General Electric Company in Schenectady was tested as a hydrogen and oxygen electrode at various temperatures. The results are given in Table 4. Whereas results reported above were essentially steady state readings in that the current was held constant for a number of minutes at each point, in this case the total current sweep from zero to 100 mA/cm^2 and back to zero was done within a few minutes.

The chamber was held at either 40° C, 25° C or 0° C, a polarization run made, and the pressure reduced for the next run. This was done for three pressures of hydrogen and oxygen.

In the case of the hydrogen polarization runs, the gas electrode was discharged against a charged positive electrode. In the case of the oxygen polarization runs, the gas electrode discharge was driven against a partially charged positive electrode.

The results were as expected. Better performance was obtained at higher temperatures and only a small difference at the various gas pressures.

3.1.2 Durability Test

In order to determine the feasibility of operating these electrodes over long periods of time under the expected operating conditions, sealed test cells were made with the gas electrode facing a porous sintered nickel counter-electrode using normal separator between them. Two cells were so constructed--one with the preferred material from the Advanced Technology Laboratories (A.T.L.), and the other with activated sintered nickel material. The cells were evacuated and backfilled with two atmospheres of hydrogen. They were operated by making the nickel counterelectrode cathodic, evolving hydrogen, which recombined on the anodic gas electrode. The current densities in the two cells were initially 11 and 25 mA/cm². Some typical cell voltages and pressures are:

	G	- 3	AT •	- 4
Initial	0.17 VDC	28 psia	0.14 VDC	29 psia
24 Hours	0.48	25	0.25	27
120	0.58	21	0.28	20
570	0.59	23	0.28	14

The current densities were increased at 570 hours to 22 and 50 mA/cm² and some typical results are:

570 hou rs	0.71 VDC	23 psia	0.38 VDC	14 psia
858	0.73	27	0.39	11
1266	0.97	29	0.42	9
2058	0.86	30	0.46	7.5

Clearly, the A.T.L. material had better electrochemical capabilities as a hydrogen electrode and this type of material was selected for use in subsequent cell work.

3.2 Design Requirements

The cells were to be nominal six ampere-hour aerospace quality hermetically sealed units protected from damage by overdischarge or overcharge.

3.2.1 Overdischarge Tolerance

In order to test the ability of the cells to withstand repeated overdischarge, the following cycle was specified:

(a)	Charge at	1.615 amps for 65 minutes	(105 A-min)
(b)	Discharge	at 3.0 amps for 35 minutes	(105 A-min)

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- (c) Charge at 1.615 amps for 65 minutes (105 A-min)
- (d) Discharge at 3 amps for 50 minutes (150 A-min)

Thus the cells are to be discharged more than they are charged. The cells are to be capable of withstanding 200 repetitive complete cycles at 45° C, 25° C and 5° C.

3.2.2 Overcharge Signal

The cells are to be equipped with a control electrode which makes it possible, by means of circuitry external to the cell, to detect the onset of overcharge and to use the detected signal to limit or stop the charging process. The cells are to be capable of performing in this manner at charge rates from C/10 to 2C (10 hour rate to 30 minute rate) and at temperatures from 0°C to 50°C.

3.3 Experimental Cells

3.3.1 Preliminary Tests

A preliminary experiment was conducted to demonstrate that an auxiliary electrode could be used for reversal protection. Of particular interest was a knowledge of the relative electrode potentials during the period of inversion.

A 1.5 AH cell was constructed in a styrene-epoxy case with two positive plates and three negative plates. An auxiliary electrode of 10 cm² area was faced opposite the one edge of the plate stack. The cell was equipped with a Ni/NiOOH, Ni(OH)₂ reference electrode, pressure gage and stopcock.

The cell was fully charged, evacuated and sealed. After discharging the cell through a 1/4 ohm resistor, it was driven through inversion for 24 hours at 100 mA. At the end of this time the following conditions existed:

Total cell voltage	-0.34 VDC
Positive Electrode vs. Ref.	-1.4 VDC
Negative Electrode vs. Ref.	-1.08 VDC
Current through Auxiliary	93 mA
Pressure	10 psia

From these data it was apparent that hydrogen was being evolved at the positive and that over 90% of it was being consumed at the auxiliary. Further, the negative electrode was only slightly positive with respect to a normal cadmium electrode. Following the above, the cell was discharged further for 19 hours at 53 mA.

Total Voltage	-0.18 VDC
Positive Electrode vs. Ref.	-1.36 VDC
Negative Electrode vs. Ref.	-1.18 VDC
Current through Auxiliary	45 mA
Pressure	12.5 psia

The cell was then discharged for 3 hours at an increased rate of 194 mA. Voltages became more negative as follows:

Total Voltage	-0.96 VDC
Positive Electrode vs. Ref.	-1.38 VDC
Negative Electrode vs. Ref.	-0.42 VDC
Current through Auxiliary	184 mA
Pressure	13.5 psia

The cell thus had a total inversion of 4.25 AH without developing a pressure of over 1 atmosphere.

The same cell was then continuously overcharged for 64 hours at 200 mA. At the end of this charge the pressure was 3 psia. 178 of the 200 mA of overcharge was now recombining oxygen on the auxiliary electrode.

3.3.2 First Prototype Cells

Having demonstrated the basic principles were sound and the available materials were suitable, two 6 AH prototype units were constructed in plastic cases and potted with epoxy. Standard aerospace processed plates were used. The ten negative and nine positives measured 5.5 x 5 cm. Two auxiliary electrodes measuring 4.5 x 6 cm were used--one auxiliary facing each outside negative. Access of gas to the back side of the auxiliary was provided by using corrugated, perforated PVC sheets. At the time these cells were constructed, the only available Niedrach-Alford type electrode was of the type prepared by the Direct Energy Conversion Operation (DECO) of the General Electric Company, West Lynn, Massachusetts, for use in the Gemini fuel cell. This was used in one cell. The other cell employed the specially prepared electrodes on a sintered nickel substrate. Both cells contained a Ni/NiOOH, $Ni(OH)_2$ reference electrode and a gas electrode. The gas electrode would normally be used for charge control. In the present case, it was used as a gas reference.

A few cycles according to paragraph 3.2.1 were run on each cell to determine performance on overdischarge. It was found necessary to give the negative plates a supplementary charge to insure that the cells were positive limiting on discharge. This was accomplished by filling the cells with hydrogen and charging the cadmium electrode (negative) against the auxiliary (positive).

With these cells, best results were obtained with the auxiliary electrode connected directly to the negative through an ammeter (total resistance of about 0.1 ohm).

It was demonstrated at this time that the cells could be charged with the auxiliary electrode connected directly to the cadmium electrode. Some hydrogen is formed on charge; however, when a sufficient hydrogen pressure is formed, the auxiliary electrode becomes more negative in potential than the cadmium electrode, so that further evolution of hydrogen is minimized.

These cells did not perform as well as anticipated so that extensive testing was not performed. Possible contributing factors were the massiveness of potting resulting in poor heat transfer and adequacy of the electrical connection with regard to resistance.

3.3.3 Second Prototype Cells

3.3.3.1 Construction

Two new hermetically sealed cells were built in metal cans. These cells were standard 6 AH aerospace cells that were modified to provide two additional terminals. This was accomplished by using two additional welded stainless steel fill-tubes through which insulated leads for the control electrode and reference were taken and then the entire lead and tube assembly potted in epoxy using a glass tube as a mold.

In order to fit the case, one set of plates was removed, leaving eight positives and nine negatives. The cells delivered 5.4 AH at the 3 ampere rate. A 4.5 x 5 cm auxiliary gas electrode faced each of the broad sides of the plate stack. These were welded to the case and connected externally to the negative terminal through an ammeter. The control electrode consisted of the same material as the auxiliary, measured 1.5 x 5 cm, and was held on one narrow face of the plate stack. A 1 x 4 cm piece of positive plate was placed against the other narrow face and served as the reference electrode.

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The two cells both employed Niedrach-Alford type electrodes made by A.T.L.; however, a different catalyst mixture was used in each cell. A further difference, which was not considered significant to performance, was that the reference in one cell (64NC339-1) was shorted during assembly to the auxiliary electrode. This made analysis of individual cell voltages difficult.

3.3.3.2 Performance of Prototype Cells

Overdischarge Test

The two prototype cells were tested for overdischarge tolerance according to 3.2.1. The circuit employed is shown in Figure 3. Cell 64NC339-1 and 64NC339-2 received approximately 200 complete cycles.

Some observations made from recording of voltage during these tests are given in Table 5. The performance of the two cells was not identical. It would appear that the negative electrode in Cell #1 has a considerably greater anti-polar mass, which could account for its relative slowness to recover from overdischarge. The final reverse voltage of Cell #1 indicates that oxygen is produced on the negative during final stages of reversal. The low value of the auxiliary recombination current (negative to auxiliary) is further indication that hydrogen is not electrochemically combining as effectively on this auxiliary electrode. However, the absence of excessive pressure indicates that hydrogen and oxygen may be combining chemically on the catalyst surface.

The overdischarge cycle tests were continued for a total of 181 and 201 cycles for Cell 64NC339-1 and 64NC339-2 respectively. The hydrogen pressures at that time were cycling as follows:

#1	13 psi	to	46 psi
#2	28 psi	to	71 psi

Change Control Room Temperature

Without any intervening time period, the normal charge of 1.65 amps was continued until both cells had a positive oxygen signal between the control electrode and the negative. This occurred in 4 hours, 55 minutes for Cell #2 (30 minutes sooner for Cell #1). When this occurred, the pressure dropped rapidly (about 40 minutes) from +49 psig to -20 inches of mercury.

The cells were then discharged at 6.0 amps to 1.0 volt/cell. Cell #1 delivered 40 minutes to 1.0 VDC; Cell #2 delivered 64 minutes.

Cell #2 was charged again the next day at 1.65 amps to a signal of 0.9 VDC and required 5 hours and 4 minutes. During charge there was a slight build-up of hydrogen pressure (to about 3 psig). By end of charge the pressure had dropped back to -20 inches of mercury. The cell gave 130 minutes to 1 VDC when discharged at 3 amps. Cell #1 was tested similiarly and gave only 92 minutes to 1.0 VDC on discharge.

The same day, the cells were given 2 cycles of charge at 12 amps to a charge control shut-off followed by a discharge at 12 amps to 1.0 VDC.

Cell #1 took 12 and 11 minutes of charge and gave 11 minutes on discharge. Cell #2 took 19.5 and 18.3 minutes of charge and gave 18 and 17 minutes on discharge.

Both cells were then charged 16 hours at 0.6 amps. Cell #1 gave a signal of 0.8 VDC after 3 hours. Cell#2 gave a signal of 0.5 VDC (control to negative) after 10.7 hours. Cell #1 gave 72 minutes to 1.0 VDC and Cell #2 gave 111 minutes when discharged at 3.0 amps. At this point tests on Cell #1 were discontinued.

Tentative conclusions drawn from these tests were that the prototype cells met the overdischarge requirements to a large extent, in that pressures were only slightly beyond the requirements. If the internal free-volume were larger, a lesser pressure would have been noted.

The cell (after the cycling test, 3.2.1) can be fully charged with the charge controlled by the control electrode at rates up to C/3 but becomes progressively worse at higher rates. (On subsequent tests, after testing at various temperatures, the cell was charged at 2 amps to 0.71 VDC signal and delivered 59.5 minutes to 1.0 VDC at 6 amps.)

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Capacity at 0°C and 50°C

Cell #2 was charged overnight at 0.6 amps (after weekend storage at 0° C). It delivered 94 minutes to 1.0 VDC at 3 amps. This was repeated again and had a control electrode signal of 0.52 VDC after 16 hours, and gave 85 minutes to 1.0 VDC at 3 amps.

When charged at 12 amps, a 0.82 volt signal was obtained in 11 minutes. The cell gave only 21 minutes to 1.0 VDC on discharge at 6 amps. Similar results were obtained on a second trial.

The next day the cell was charged at 0.6 amps at 50°C. A signal of 0.52 was observed after 16 hours. The cell gave less than 50 minutes to 1.0 VDC at 3.0 amps. This was repeated again with similar results.

When charged at 12 amps and 50° C, a signal of 0.85 VDC was obtained in 18 minutes and the cell gave 55 minutes to 1.0 VDC at 3 amps.

Tentative conclusions drawn from these tests were that the performance was considerably worse at the high and low temperatures. The control electrode would, however, function to prevent excessive overcharge. The performance noted was probably attributable to the positive electrode being inefficient after repetitive overdischarges.

Overdischarge Tolerance at +5°C and +45°C

The cell was then cycled for 16 cycles on the overdischarge cycle at +45°C. At the end of this time the pressure was up to 76 psig. The cell was still performing according to design principles, except that the positive was not accepting charge as fully and thus was inverting sooner.

One full cycle was given at +5°C and the pressure reached 110 psig. The first cycle did not have a full charge cycle and this may have accounted in part for the high pressures. Tests with these prototype cells were not continued, since work had begun on the next generation of cells.

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3.4 Finalized Design Cells

A meeting was held on October 8, 1964, with J. Sherfey at NASA/Goddard to discuss performance to date and various design concepts. It was agreed that the cells should approximate normal 6 ampere-hour size even though the capacity would be somewhat less; that is, the final design would follow essentially that of prototype cells (3.3.3.1).

3.4.1 Fourth Electrode Cell Design

The design goal of the 4th electrode cell was to approximate the size and nominal electrical capacity of a standard prismatic 6 ampere-hour aerospace cell. With this in mind, it was decided to use a case of the same cross section as our standard 6 AH cell, 42B006AB03, and extend the length slightly if necessary. This would enable us to use standard ceramic seal covers, and eliminate the ordinarily long delay experienced when obtaining covers of a unique design.

In order to accomodate the additional electrodes, it was decided to build the cell with two "covers" containing 4 insulated terminals. The cell was designed utilizing the two insulated terminals on the top cover for positive and negative electrodes, one bottom terminal for the 3rd electrode (charge control), and the other insulated bottom terminal for the reference electrode. The can was used as the 4th electrode terminal. In order to allow for additional terminal space at the bottom of the can and still utilize the full plate length, the can was lengthened by 0.25 inches over the standard 6 AH size. The bottom fill tube was pinched off before filling the cell with electrolyte, while the top fill tube was fitted with the required plumbing after addition of electrolyte. An outline drawing of the finished cell is shown in Figure 4.

The internal construction of the cell is shown schematically in Figure 5. It contains 9 negative plates and 8 positives. These were welded to the top insulated terminals and wrapped with non-woven nylon separator. A third (charge control) electrode was located on one edge of the pack with a piece of separator between the pack and the electrode. A section of corrugated PVC was placed between the electrode and the cell case for insulation and to provide a gas space. The reference electrode, consisting of a small piece of partially charged positive plate, was placed on the bottom of the pack assembly completely enclosed with a wrap of separator. Fourth electrodes were assembled on both broad faces of the plate pack, with a layer of separator between the electrode and the adjacent negative plate. Gas space was provided between the can and the electrode by a piece of corrugated PVC.

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The entire plate and auxiliary electrode pack was enclosed in a polypropylene wrapper, and inserted into the can and the top cover welded in place. The fourth electrodes were spot welded to the can walls, the third electrode lead welded to one bottom terminal and the reference electrode lead welded to the other bottom terminal. Following this, the bottom cover was welded on and 18 cc of 31% K O H was introduced into the cell. A pressure gage was assembled to each cell and after a 24 hour soak period the cells were ready for testing.

3.4.2 Preliminary Trial Cells

Sufficient plate material was selected to build the required number of cells plus spares and given the normal aerospace processing. Since there was to be a short delay in obtaining all the cell components, it was decided to build two preliminary cells using a can fabricated in our pilot shop.

The cells were given a number of formation cycles. The state of charge was set by driving the cells into reverse and venting the gasses generated.

The cells were given seven full overdischarge cycles according to (3.2.1) at room temperature.

They were charged at room temperature at 6.0 amps to a signal cut-off and gave 63 to 64 minutes when discharged at 6.0 amps.

One cell was given 16 overdischarge cycles according to (3.2.1) at 0°C. The pressure range during the 16th cycle was from 35 to 66 psig.

Both cells were given three cycles at 0° C of charge at 6.0 amps to a signal cut-off and discharge at 6.0 amps. They ran from 54 to 60 minutes on discharge.

Both cells were given three similar cycles of charge and discharge at 6.0 amps at 40° C. They ran from 50 to 62 minutes.

The two cells were given 15 overdischarge cycles at 45° C according to (3.2.1). On the 15th cycle the pressure ranged from -25'' of Hg to +69 psig.

The results from these tests, were considered satisfactory so that the final production cells could be assembled and tested in a straight forward manner as soon as all hardware was received.

3.5 Production Cells

Fifteen cells were assembled. One cell had a defective cover and was not processed further after being leak tested on the mass spectrograph. The assembly formation and testing was done according to General Electric Process Instruction P24A-PB-128 (See Appendix). Test results are given in Table 6.

Throughout all testing a 10 milliohm wire was connected between the auxiliary, (4th) electrode and the negative electrode. The potential across this wire measured with a millvoltmeter, can be related to the recombination current at the auxiliary.

A 10 ohm resistor was connected between the control, (3e⁻), electrode and the negative. The cells were all equipped with pressure gages and pressure relief valve.

3.5.1 Formation Tests (3.0 of P24A-PB-128)

The cells were given 3 formation cycles consisting of 16 hour charge at C/10.

The cells were all seen to be very close to 6 AH at the two hour rate.

The discharge on the second cycle was taken to -1.0 vdc and the cells vented to regulate the state of change.

3.5.2 Acceptance Tests

Voltage Stabilization (4.1 of P24A-PB-128)

The stabilized voltages are given in Table 6. It should be noted that the stabilized voltage is less than the maximum value reached during charge. The maximum voltage was reached after about 13 hours of charge at C/10 and was about 0.02 volts higher.

The reason for this difference is that when the oxygen recombination current becomes equal to the charge rate then the negative cadmium electrode is no longer being charged and the cell potential no longer shows the overvoltage due to charging. Obviously the order of magnitude of this difference will depend on the charging rate.

Internal Impedance (4.2 of P24A-PB-128)

The 60 cycle impedance were measured by determining the AC voltage drop when 100 mA AC is passed between the desired points. The cell impedance were from 2.6 to 4.8 milliohms which is considered normal for a cell of this size. The fourth electrode to negative impedances ranged from 12 to 22 milliohms. The third electrode to negative impedances ranged from 112 to 140 milliohms.

Capacity Discharge (4.3 of P24A-PB-128)

The capacity of the cells ranged from 5.5 to 6 AH at the two hour rate.

Internal Short (4.4 of P24A-PB-128)

All cells passed the internal short tests.

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Overdischarge at 25°C (4.5 of P24A-PB-128)

The cells were given 4 cycles of overdischarge (two on one day and two on the following day). After each of the first two cycles, the cells were vented to further adjust the state of charge. This was the last venting which the cells received. The result of all four cycles are summarized in Table 6.

More detailed results are given in Table 7 for the last two cycles. Cell voltages, reference to negative voltages, auxiliary electrode to negative current and pressure are given. The auxiliary to negative currents were obtained by dividing the voltage drop measured across the 10 milliohm wire by 0.01. These measurements were probably accurate to $\pm 10\%$. It can be noted that the auxiliary to negative current is generally greater when the negative electrode approaches more complete discharge. One cell #14 is seen to perform differently from the others in that the pressure remains at a low value. This may be partially accounted for by the fact that the negative breaks earlier than most others, however, performance on other tests would indicate that the auxiliary electrode in this cell was more active (showed less polarization) for some reason.

Overcharge Control (4.6 and 4.7 of P24A-PB-128)

On the first cycle the cells were charged at the C/2 rate to a signal voltage of 0.5 vdc (control electrode to negative) and then discharged at the two hour rate. The data is summarized in Table 6 and details are given in Table 8. All cells gave greater than 6.3 AH on the first cycle, which, followed the overdischarge tests of 4.5. The high charge rate combined with the previous deep discharge accounts for the high capacities observed.

On the second cycle the cells were charged in the same manner except that the charge at C/2 was continued for a total of 210 minutes, which corresponds to a charge input of 10.5 AH. Results are given in Tables 6 and 8.

The data has been plotted in Figure 6 for cell #2. During charge a hydrogen pressure develops due to evolution of hydrogen on the auxiliary electrode. If it were considered desirable to minimize this effect, a somewhat larger resistor could be used to replace the 10 milliohm connection. The charge current being used to generate hydrogen during the early stages of charging at this rate is estimated to be about 60 mA or about 2% of the total. At these charge rates the generation of hydrogen is not serious since the potential of the auxiliary will become more negative as the hydrogen pressure increases until the auxiliary potential becomes equal to the negative at which time the hydrogen current will be insignificant.

The hydrogen pressure disappears completely as soon as oxygen

starts evolving at a significant rate from the positive plate, i.e., near the end of full charge. (The hydrogen combines chemically on the catalyst surface with the oxygen.) At this time the voltage between the control and negative electrode rises sharply. The data in Table 8 shows that the auxiliary to negative current also increases sharply at this point indicating that the oxygen generated on overcharge is recombining on the auxiliary electrode. The recombination is so efficient that the pressure remains at a very low value during the overcharge period.

The Cell voltage shows an interesting increase at about the same time. The rise in potential is due to the positive electrode and probably is due to the oxygen overvoltage. As soon as the auxiliary starts to recombine oxygen at a significant rate, there is a drop in potential on the negative electrode. This corresponds to the overvoltage of charging the cadmium electrode, i.e., the negative electrode is no longer being charged since the current is now being used for the reduction of oxygen on the auxiliary.

(It is seen in Tables 6 and 8 that cell #14 shows an extreme drop in the potential of the negative electrode at this time. The value of the negative potential on these cycles is at times less than the open circuit potential of the cadmium electrode, which would suggest an error in measurement possibly due to poor voltage or current connection. However, soldered and stack-on connections were used with a terminal board and no single set of conditions can explain the anomalous behavior noted.)

The cells were discharged at the 6 ampere rate (except for #14 which was continued on overcharge for 24 hours). The capacities were somewhat lower than the previous cycle and ranged from 5.9 to 6.6 AH.

3.5.3 <u>Supplemental Testing</u>

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Ten of the fourteen cells were selected and shipped to NASA/ Goddard for additional tests. Two of the remaining four were tested according to paragraph 5.0 of P24A-PB-128. The charge control tests were performed in air ambients of 0°C, 25°C, and 50°C. The overdischarge tests were performed at 5°C, 25° and 45°C.

Overcharge at 0°C (5.1 of P24A-PB-128)

The two cells were charged at the 6 amp rate at 0° C until a signal was obtained between the control electrode and the negative electrode.

The data is given in Table 9 and is plotted in Figure 7 for cell #2.

The results are similar to Figure 6 (C/2 at RT) except that the pressures are a little higher. When discharged at

the 6 amp rate, 5.2 AH were obtained.

Overcharge at 50°C (5.2, 5.3 and 5.4 of P24A-PB-128) .

The cells were given two cycles of charge (to signal cut-off) and discharged at 6.0 amps. The cells gave 4.5 AH on discharge after a charge of 5.6 AH for a charge efficiency of 80%. Detailed data are given in Table 9.

The third cycle at 50° C consisted of a 3 amp charge to a signal and a 6 amp discharge. The cells gave 5.2 and 5.0 AH after a charge of 6.6 AH for a charge efficiency of 78 and 76%.

Overcharge at RT (5.5 and 5.6 of PB24A-PB-128)

The cells were given two cycles of charge and discharge at the 6 amp rate. The cells gave 5.7 and 5.6 AH on the first cycle after a charge of 6.7 AH for a charge efficiency of about 85%. Data are given in Table 9 and plotted in Figure 8 for cell #2 on the first cycle.

Overdischarge Cycle (5.7 of P24A-PB-128)

The two cells were given 20 full overdischarge cycles at 25°C, 10 cycles at 5°C and 10 cycles at 45°C. Tracings were made of the data for the last cycle at each temperature from continuous recordings of the single electrode potentials and auxiliary gas recombination currents. The discharge portions are given in Figures 9A and 9B, 10A and 10B, and 11A and 11B for cell #2.

The positive to reference voltage is very small on a charged plate, since the reference electrode is the same type of electrode. When the positive plate is discharged the potential of the nickel hydroxide electrode becomes negative to the reference. At a negative potential of about 1.2 it begins to evolve hydrogen.

The negative to reference voltage is nearly constant with state of charged and has a negative value of about 1.2 - 1.3. When it is discharged it becomes less negative with respect to the NiOOH, Ni(OH)₂ reference electrode. At a potential more positive than about 0.15 volts it will evolve oxygen.

The cell voltage (which is not given in the recorder tracings) is the algebraic difference between the two single electrode potentials. Thus during discharge in Figure 9A the mid-voltage of the normal discharge (@ 80 minutes) would be about 1.25. At 97 minutes the potentials are equal and the cell voltage is zero. From 98 to 100 minutes the nickel hydroxide

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electrode is more negative than cadmium electrode and the cell potential is negative (about -0.20).

The current is given as the negative to auxiliary current and a positive value corresponds to the hydrogen recombination reaction. Whereas a negative value corresponds to hydrogen evolution on the auxiliary electrode.

Overdischarge cycles at 25°C

Referring again to Figure 9A, (The first discharge of the twentieth cycle at room temperature) the ampere-hours discharged are equal to the previous charge. In this case about 86% of the charge is delivered as useful capacity. At this point the positive electrode is completely discharged and hydrogen is evolved. The negative cadmium electrode is not discharged until the last few minutes and its potential is just breaking as the cells go into the charge cycle. The negative to auxiliary current is small, less than a half an ampere and drops slightly during the discharge, while the hydrogen pressure continues to drop (see Figure 12). The hydrogen being consumed results in increased charge of the cadmium electrode. Near the end of the discharge period the auxiliary current shows an increase due to the increase in hydrogen pressure and due to the negative electrode becoming less negative.

Referring to Figure 12 the hydrogen pressure is seen to have been dropping during the charge and discharge period until the positive electrode reverses at which time the pressure shows a sharp increase.

The pressure then drops slowly during the next charge period, drops more rapidly during the next discharge and then increases rapidly during the overdischarge.

Referring to the second discharge in Figure 9B, the various curves are very similar to those of 9A until the longer overdischarge period. When the negative electrode becomes more positive than previously the auxiliary currents increases to a higher value. Since the recombination current is less than the overdischarge current the pressure continues to increase until the end of the cycle. If the negative electrode had a smaller charge and thus had become more positive, the recombination current would have been much higher.

The maximum pressures during the twenty cycles at 25° C were 68 psia for #2 and 74 psia for #13. The minimum pressures were 16 psia for #2 and 19 psia for #13.

Overdischarge Cycles at 5°C

Results are given in Figure 10A and 10B for the tenth

overdischarge cycle at 5°C. The initial parts of the discharge curves are similar to those obtained at 25°C. The capacity of both the positive and negative are less than at 25°C. Consequently, the pressure (see Figure 12) begins to rise sooner on overdischarge. However the negative electrode breaks sooner and more sharply and consequently on the second discharge the gas recombination current rise to a higher value of almost 2 amperes.

At the start of the next charge after the second overdischarge, the negative to auxiliary current shows a sharp negative spike. This spike is frequently observed when the cadmium electrode has reached a relatively positive value and the direction of current flow in the circuit is instanteously reversed. It may be that the cadmium electrode recovers its normal negative value sooner than the auxiliary with the result that the current flows in the opposite direction until the auxiliary has acquired the same potential. It is unlikely that hydrogen is being evolved from the auxiliary at this potential.

The maximum pressures during the ten cycles of overdischarge at 5°C were 70 psia for cell #2 and 100 for cell #13. The minimum pressures were 30 psia for #2 and 40 psia for #13.

Overdischarge Cycles at 45°C

Results are given in Figures 11A and 11B for the tenth overdischarge cycle at 45°C. The positive electrode shows a pronounced graphitic step which amounts to almost one fourth of the total capacity. It is interesting to note that the total positive charge is about the same as at 25°C, while the capacity to the normal 1.0 vdc is only about 75%.

The cadmium electrode became discharged shortly after the positive electrode and rose rapidly to a less negative potential.

The activity of the gas electrode at the higher temperature is predictably much greater. As a result the pressure is very low (see Figure 12). During the overdischarge, the hydrogen is recombined as rapidly as it is formed and a different pattern is observed from that at lower temperatures. At 45°C the pressure increases during charge to about one atmosphere and drops during discharge, remains essentially constant even during overdischarge and then rises slowly again on charge.

The recombination current approaches the total overdischarge current of 3 amperes. During the overdischarge, the cadmium electrode showed some variability in that it started to recover a more negative potential at times. As a result there were similar variations in the recombination current.

The maximum pressures during the ten overdischarge cycle at 45°C were 14 psia for cell #2 and 29 psia for cell #13. The minimum pressures were 5 psia for cell #2 and 10 psia for cell #13.

4.0 Discussion and Recommendations

It has been demonstrated that hermetically sealed nickel cadmium cells can be built which are immune to damage due to overdischarge or overcharge. This has been accomplished by employing suitable gas electrodes within the cell container in the proper configuration.

Ten hermetically sealed aerospace quality cells were delivered to NASA/Goddard as a consequence of this contract. These cells will be given additional tests to determine their long term performance characteristics.

While the cells were designed primarily to demonstrate the feasibility of using gas electrodes to recombine gases formed during overdischarge, they clearly demonstrate the advantages of using auxiliary electrodes for overcharge protection. These cells are capable of being continuously overcharged at rates as high as the C - rate. Obviously if this is done in practice, provisions must be made for removing the heat generated.

Control electrodes were provided which can be used to determine the presence of oxygen on overcharge. The signal so developed can be used in the conventional manner to regulate the charge. In a sense the control electrode is redundant since the direction of current flow in large auxiliary to negative loop could also be used for the same purpose. Thus a low resistance coil might be used to make this connection and the circuit set up in such a manner that the charge would be terminated at a particular oxygen recombination current.

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TEST A-421

POLARIZATION DATA OF 3 OXYGEN ELECTRODES

AT VARIOUS PRESSURES AND CURRENT DENSITIES AT ROOM TEMPERATURE

Time in Minutes	Pressure, psia	Current Density	Voltage NiOOH to Test Electrode		
	02	mA/cm ^{2*}		Sample	
			AT-1	G. D.	<u>A. C.</u>
$ \begin{array}{c} 1 \\ 2 \\ 6 \\ 7 \\ 9 \\ 11 \\ 12 \\ 17 \\ 18 \\ 21 \\ 24 \\ 25 \\ 27 \\ 29 \\ 30 \\ 32 \\ 34 \\ 40 \\ 45 \\ 49 \\ 50 \\ 52 \\ 55 \\ 57 \\ 58 \\ 60 \\ 61 \\ 63 \\ 64 \\ 72 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 73 \\ 75 \\ $	$ \begin{array}{c} 13.15 \\ \hline 13.15 \\ \hline \hline 7.05 \\ \hline 4.96 \\ \hline \hline 2.95 \\ \hline \hline 1.0 \\ \hline \hline 13.1 \\ \hline \hline 6.9 \\ \hline \hline \hline 3.0 \\ \hline \hline 1.0 \\ \hline \hline 13.0 \\ \hline \hline 13.0 \\ \hline \hline \end{array} $	0 0.25 V 1.0 10.0	$\begin{array}{r} AT-1 \\ 0.190 \\ 0.390 \\ 0.400 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.460 \\ 0.470 \\ 0.470 \\ 0.470 \\ 0.470 \\ 0.470 \\ 0.470 \\ 0.580 \\ $	G. D. G. D. 0.220 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.320 0.400 0.400 0.420 0.560 0.560 0.560 0.570 0.620 0.530 0.620 0.620	A. C. 0.210 0.270 0.280 0.340 0.350 0.350 0.350 0.360 0.380 0.380 0.380 0.390 0.390 0.390 0.410 0.415 0.420 0.360 0.500 0.500 0.510 0.510 0.520 0.550
77			0.620	0.620	0.570

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Time in Minutes	Pressure, psia	Current Density	Noltage NiOOH to Test Electrode		
	o ₂	mA/cm ^{2°}		Sample	
			AT-1	G. D.	A. C.
78 80 82 83 85 86 87 89 90 92 93 100 103 105 106 107 109 110 112 113 113 117 117 119 121 130 133 135 136	$ \begin{array}{c} $	25.0	AT-1 0.640 0.640 0.650 0.650 0.650 0.650 0.650 0.660 0.700 0.700 0.700 0.640 0.700 0.700 0.700 0.700 0.700 0.740 0.740 0.740 0.750 0.750 0.760 0.760 0.820 0.720 0.800 0.800 0.800 0.800	S a m p 1 e G. D. 0.640 0.640 0.640 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.660 0.770 0.780 0.780 0.610 0.620 0.620 0.620 0.740 0.740 0.740 0.760 0.760 0.800 0.940 0.940 0.940 0.740 0.740 0.740 0.740 0.740 0.740 0.740 0.740 0.740 0.740	A. C. 0.590 0.600 0.600 0.610 0.620 0.620 0.630 0.630 0.630 0.660 0.670 0.610 0.610 0.610 0.610 0.700 0.700 0.710 0.710 0.710 0.730 0.730 0.830 0.830 0.680 0.760 0.760 0.780
140 140 142 142 145 145	$ \begin{array}{r} $		0.810 0.820 0.820 0.830 0.830 0.830	0.770 0.790 0.790 0.840 0.840 1.050	0.780 0.780 0.800 0.800 0.950
148	↓ <u> </u>		0.890	1.050	0.950

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(continued)

Time in Pressure, Minutes psia		Current Densiţy	Voltage NiOOH to Test Electrode		
	02	mA/cm ²		Sample	
			AT-1	G. D.	A. C.
155	13.0	75.0	0.790	0.720	0.740
180		<u>v</u>	0.790	0.720	0.740
184		100.0	0.840	0.760	0.800
189			0.840	0.780	0.820
195	<u> </u>		<u> </u>	0.780	0.820
195	6.85		0.880	0.800	0.830
198			0.880	0.800	0.830
198	5.0		0.890	0.830	0.840
201			0.890	0.830	0.840
202	3.0		0.920	0.890	0.870
207			0.920	0.870	0.860
210	V		0.920	0.870	0.860
210	1.0				
211			1.000	1.580	1.030
216	V		1.000	1.580	1.030
224	13.0		0.800	0.720	0.780
228			0.800	0.720	0.780
232			0.800	0,720	0.780
235			0.800	0.720	0.780
242		₩	0.800	0.720	0.780
244	V	OCV	0	0.340	0.300
	1	}			

TEST C-512

POLARIZATION DATA OF 3 OXYGEN ELECTRODES

AT VARIOUS PRESSURES AND CURRENT DENSITIES AT ROOM TEMPERATURE

Time in Minutes	Pressure, psia	Current Density	Voltage NiOOH to Test Electrode		
	⁰ 2	mA/cm ²	·	<u>Sample</u>	
			G - 1	<u> </u>	<u>G - 4</u>
0	13.0		0.30	0.30	0.29
3	1.2.0	5.0	0.58	0.50	0.51
5	5.0	5.0	0.50	0.51	0.56
7			0.61	0.51	0.56
8	i k		0.62	0.50	0.56
10	↓		0.60	0.50	0.56
12	3.0		0.62	0.54	0.58
13	↓		0.62	0,54	0.58
14	13.0	<u> </u>	0.60	0.51	0.55
15		10.0	0.72	0.56	0.64
18			0.72	0.58	0.64
20	<u> </u>		0.72	0.58	0.64
20	5.0		0.72	0.60	0.66
23	<u> </u>		0.72	0.60	0.66
23	3.0		0.73	0.62	0.67
25	· · · ·		0.73	0.62	0,67
25	1.0		0.74	0.64	0.69
27	<u> </u>		0.74	0,64	0.69
28	13.0	<u> </u>	0.70	0.60	0.64
28		15.0	0.76	0.65	0.70
31	<u> </u>		0.76	0.65	0.70
31	5,0	ļ	0.78	0.66	0.72
34	·		0.78	0.66	
35	3.0		0.78	0.68	0.73
39	<u> </u>		0.78		0.75
29 /1			0.79	0.70	0.75
4L //2		↓	0.79		0.70
42	1 12.0	20.0	0.74	0.64	0.76
42		20.0		0.00	0.74
45			0.80	0.69	0.74
47	5.0		0.82	0.70	0.76
50	V		0.82	0.70	0.76
50	3.0		0.82	0,71	0.77
53	↓		0.82	0.71	0.77
	·				
}		▼]		

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(continued)

Time in Minutes	Pressure, psia	Current Density	Voltage NiOOH to Test Electrode		
	0 ₂	mA/dm ²		Sample	
			G - 1	G - 3	G - 4
53	• 1.0	20.0	0.86	0.74	0.80
55	<u> </u>		0.86	0.74	0.80
55	13.0	<u> </u>	0.77	0.68	0.74
55		25.0	0.84	0.71	0.78
58	<u> </u>		0.84	0.71	0.78
58	5.0		0.85	0.72	0.80
60	<u> </u>		0.85	0.72	0.80
60	3.0		0.85	0.73	0.80
63	<u></u>		0.85	0.73	0.80
63	1.0		0.89	0.78	0.84
65	<u></u>	J J	0.92	0.80	0.84
65	13.0		0.79	0.70	0.77
70		30.0	0.94	0.7/	0 92
			0.04	0.74	0.02
			0.84	0.74	0.84
	J.U		0.80	0.76	0.84
80	3.0		0.87	0.76	0.85
83	يان ا		0.87	0.76	0.85
83			0.92	0.82	0.88
85	1		0.92	0.82	0.88
85	13.0		0.82	0.72	0.80
86		50.0	0.97	0.82	0.96
89			0.97	0.82	0.90
89	5.0		0,99	0.84	0.98
92	¥		0.99	0.84	0.98
92	3.0		1.00	0.86	0.99
95	<u> </u>		1.00	0.86	0.99
95	1.0		1.40	1.24	1.08
98	↓ <u> </u>		1.40	1.20	1.08
98	13.0	<u> </u>	0,94	0.78	0.94
98		100.0	1.25	1.00	1.30
100	5.0		1.25	1.00	1.30
105			1,25		1.30
105	3.0		-1.30	1.08	2.20
110	<u> </u>		2.30	2.10	2.20
	1				

TEST G-528

HYDROGEN POLARIZATION DATA ON SAMPLE G-3

(Voltage NiOOH to Hydrogen Electrode)

.

CURRENT CONSTANT WHILE PRESSURE VARIED

Hydrogen	Current Density mA/cm ²					
Pressure psia	0	2	3	5	10	15
13	1.27	1.25	1.21	1.20	1.09	1.03
7		1.21	1.20	1.15	1.05	0.98
3		1.20	1.15	1.10	0.99	0.86
1		1.10	1.08	1.0	0,83	-0.32

PRESSURE	CONSTANT	AND	CURRENT	VARI ED
		_		

Current Density	Voltage NiOOH vs. H2 Electrode				
mA/cm ²	13.0.psia	7.0 psia	3.0 psia		
0	1.28	1.23	1.25		
2	1.26	1.19	1.22		
3	1.24	1.18	1.18		
5	1.21	1.05	1.18		
10	1.12	.96	.95		
15	1.12	.91	.80		
25	.97	.81	.82		
35	.87	.72	.46		
50	.58	.62	46		
75	.60				
100	.52	60			
150	.36				
	1				

TEST D-519

POLARIZATION DATA OF PREFERRED ELECTRODE MATERIAL

AT VARIOUS TEMPERATURES AND PRESSURES OF H2 AND 02

(Voltage: NiOOH to Gas Electrode. Data from X - Y recordings)

Test Condition	Current Density	OCV	50 mA/cm ²	100 mA/cm ²
42°C	Initial	1.34	1.25	1.14
13.0 psia H ₂	Return		1.155	1.08
41°C	Initial	1.32	1.255	1.17
7 psia H ₂	Return	1.255	1.16	1.10
40°C.	Initial	1.32	1.23	1.135
3 psia H ₂	Return	1.205	1.105	1.02
Room Temperature	Initial	1.365	1.18	0.965
13.0 psia H ₂	Return	1.28	1.06	0.915
Room Te mperature	Initial	1.34	1.15	0.90
7.0 psia H ₂	Return	1.25	1.035	0.88
Room Temperature	Initial	1.34	1.21	1.06
3.0 psia H ₂	Return	1.25	1.09	0.99
0°C	Initial	1.38	1.14	0.92
13.0 psia H ₂	Return	1.28	0.98	0.82
0°C	Initial	1.39	1.14	0.91
7.0 psia H ₂	Return	1.25	0.91	0.68
0°C	Initial	1.38	1.15	0.93
3.0 psia H ₂	Return	1.22	0.915	0.67

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(Continu	ed)
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Test Condition	Current Density	ocv	50 mA/cm ²	100 mA/cm ²
	Tathtal	0.41	0.64	0.75
13 psia O ₂	Return	0.58	0.76	0.75
41°C	Initial	0.37	0.63	0.76
7.0 psia O ₂	Return	0.58	0.76	0.86
40°C	Initial	0.38	0.67	0.83
3.0 psia O ₂	Return	0.60	0.79	
Room Temperature	Initial	0.20	0.77	0.96
13.0 psia O ₂	Return	0.67	0.91	1.04
Room Temperature	Initial	0.36	0.74	0.94
7.0 psia O ₂	Return	0.26	0.73	-
Room Temperature 3.0 psia O ₂	Initial Return	-	-	1.05 -
0°C	Initial	0.32	0.74	1.08
13.0 psia O ₂	Return	0.67	1.06	
0°C	Initial	0.24	0.90	1.11*
7.0 psia O ₂	Return	0.67	1.05	-
0°C	Initial	0.37	0.81	1.15**
3.0 psia 0 ₂	Return	0.66	1.13	

*80 mA/cm²

- 32 -

39-2	Cycle #161	Similar to cycle #33	Similar to cycle #33	Similar to cycle #13	Similar to cycle #13	Mailmum auriliary current 1.8 amps Preseure range: 23-52 pai
(MIC)	Gycle #33	Immediate recovery. Normal voltaga 65 min.	29 min. discharge. Fositive inverta at 29 min. Begative inverta at 35 min.	Immediate recovery Normal voltage 65 min.	33 min. normal discharge Positive inverts at 33 min. Megative begins to invert at 34 min. Final cell voltage 1.26 VDC.	Mariama auriliary current 2.35 ampa
	Cycle #140	22 min. for negative to recover. Has two steps 15 min. and 7 min. 43 min. normal charge.	20 min. discharge. Fositive inverts at 20 minutes. Negative inverts at 28 minutes.	15 minutes for negative to recover. Normal charge 50 minutes.	25 min. normel discharge Positive inverts at 25 min. Negative inverts at 30 min. Final cell voltages 1.66 VDC	Maximum suxiliary current 0.3 amp Press range: 15-43 pei
<u>648C339-1</u>	Cycle #40	<pre>11 min. for negative to recover normel voltage. 54 min. normel charge.</pre>	25 min. normal discharge. Positive inverts at 25 min. Negative inverts at 30 min.	Positive recovers immedi- ately. Begative recovers in 5 min. 60 minutes normal charge.	29 min. normal discharge. Positive inverts at 29 min. Regative inverts at 33 min. Final cell voltage1.66 VDC	Maximum auxiliary current 1.4 amp
	Operation (per 3.2.1)	<pre>i) Charge 1.62 amps for 65 minutes</pre>	o) Discharge 3 amps for 35 min.	c) Charge 1.62 amps for 65 min.	 Discharge 3 amps for 50 min. 	

RESULTS ON OVERDISCHARCE CTCLE (PARACHARE 3.2.1)

TABLE 5

	kaf. Tast M	1.1 I. First Charge Dischar 1.1 2.1 3. 1	1.2 II 2nd Fo 4th Ell 3rd 10 11.1 1.1 1.1 1.1 1.4 1.4 1.4 1.4 1.4 1.	3.3 III 3rd 70 Clarge Dische 1. 3. 5.	4.1 IV Voltat Charge 1 4.2 2. 4.3 3.	4.4 Y. Inter Short Charg
	amber Test Description	formation Cycle, C/10 - 16 hours Ege C/2 - 1.0 Volt Egel Carrge Voltage End Carrge Fressure Macharge Time to 1.0 V in minutes	rmation Cycle concode shorteed to magative ohn to magative C/10 16 hours C/10 10 volt vented fit -1.0 volt to Charge Voltage End Charge Voltage End Charge remeature Discharge rism to +1.0 volt Pressure at -1.0 volt before vent	rmation Cycle - C/10 - 16 hours - C/10 - 16 hours End Charge Voitage End Charge Pressure End Charge Inseite c 1.0 V in minutes End Voitage 160 minutes End Pressure 160 minutes	e Stabilization co colo until voitages change 5 w or na coe hour (miaiama 16 houre) Final Voitage Orycia impadance e. Poritive to nagative milliches b. éth to nagative milliches b. eth to nagative milliches bichange c/2 to 1.0 V Tima to 1.0 V, minutes	mai Short : 16 hours : 16 0 S adautes 24 hours :y, at 24 hours min. 1.0 V
	1-1	1.42 -21" 122	1.41 - 21" 122 60 0	1.41 - 25" 129 - 0.15	1.406 3.2 19 130 119	1.23
	-1	1.42 -22" 123	1.41 -22" 117 21\$	1.41 -22" 125 -0.14 -17 "	1.414 4.3 20.5 1137	1.26
	1-3 -1	1.42 -25" 121	1.42 -25" 118	1.42 -25" -1.16 -25"	1.418 3.0 20.5 134 116	1.24
	4-1	1.42 -12" 125	1.41 - 25" 164	1.42 -24" -1.40 -1.40	1.408 2.4 12 140 120	1.22
	1-5	1.42 -26" 125	1.41 - 25" 150	1.41 125" 125" 125"	1,408 3.3 16.5 1138 119	1.23
ŭ	1-7	1.42 -26" 120	1.41 -25" 121 -21"	1.42 -26" 125 -1.50	1.416 4.0 13.4 136 120	1.22
.11	1-8	1.43 0 121	1.42 -26" 47 4	1.43 -19" 121 -1.45 -24"	1.420 3.5 19.7 134 115	1.26
	6-1	1.42 -4" 121	1.41 -16" -16"	1.42 -11" 118 -1.46 -1.46	1.414 2.7 21.5 112 111	1.23
	1-10	1.42 -16" 118	1.41 -16" 116 -9"	1.42 -16" -116 -1.44	1.418 4.8 21 126 114	1.23
	11-1	1.42 -26" 119		1,43 -26" 125 -1.51 -25"	1.422 3.0 20 138 115	1.22
ŀ	7-17	-24"	1,4,1 	-20" 122 -20" -20" -20" -20" -21"	1.426 2.6 15.5 1132 115	1.2
F	1-1	1.42 -19" 118	1.41 1.41 1.14 1.16 -10 ⁻	-19" -19" -19" -1145 -11"	1.414 2.9 22 136 112	1.12
	91-1	1.44 -25" 120	1. 24* -24" 1	1.42 -26" 120 -1.48 -25"	1.418 3.8 15.5 119 115	1.24
ſ	1-15	- 24" - 24"	1.41 -22" 119	1.42 -26" 122 -1.44 -25"	1.412 2.5 14.3 113 116	1.22
	Coments		41-14 mot charged		Actual Stabilization Time 18 hours	

TARIA 6 TARIA 6 COMMATION AND ACCEPTING TO ACCOUNTING TO G.E., PROGES, STRUTTICATION TO ACCOUNTING TO

Commit	All calls vented B ₂ to 5 paig Vented B ₂ to + 2 paig	
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• •		62 155 1.48 1.48 191
		74 155 70 136 130
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		86 145 - 26" 128
	1	87 149 1.78 1.47 1.23 123
	- •¥4¥¥¥ ¥÷1988 + ¥84¥¥4 *¥493	86 155 1.48 132
	Test Restand: Shartey Orela A Tr Pour Manuel: Shartey Orela A Charge 65 atlantes 0.1613 and C Charge 55 atlantes 0.1613 and C Charge 55 atlantes 0.1613 and C C Charge 55 atlantes 0.1613 and C C Charge 55 atlantes 0.1613 and C C C C C C C C C C C C C C C C C C C	 24. Rad Sad Disch. 412 Oylia Yoliaga 27. Discharge to 3rd to Magazive Signal 27. Discharge to 1.0 volts @ 2⁵C 1. Risan voltaga 2. Rignal voltaga 3. End charge voltaga 4. Bad charge prosents 5. Then disch. to 1.0 volt, unastee
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TANKE 6 (Continued)

	Comments	014 mot diacharged but laft on overcharge for 24 hours at C/2
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	1-14	138
	1-13	147 . 69 1. 606 - 19"
	1-12	142 .83 60 .1.412 -21"
	1-11	142 . 706 61 -25
	1-10	147 59 - 149 - 23"
	1-9	147
	1-8	138
•	1-7	142 .70 1.416 -26"
	1-5	147 - 75 1.410 - 26"
	1-4	147 - 76 1.414 - 22"
	1-1	142 . 614 - 26''
	1-2	147 73 61 -22"
	1-1	147 . 83 64 42 - 25"
	Test Mumber Test Description	WIII Overcharge - Charge C/2 to Signal and 90 minutes bygood signal Discharge C - Ants 1 Time to Signal 2 Signal Voltage 3 End Overcharge - 1.0 V - minutes 4 Time Discharge to 1.0 V - minutes 5 Freenure End Overcharge 6 Fill Voltage b. Call Presence 1 Feasure
Lef.	Para.	

TARE 6 (boutimed)

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Test Number	Test Description	>	ġ	-	Bure	▶	¥.	-	e la	╞	ž	-	<u>z</u>	,		1		-		<u>ا</u>	+-	+		-	-	┢		┢	
 Jrd Cycle	**											· · · ·													• • • • •				
Charge 1.615 amps for 40 min 60 min 64 min	r 65 minutes	1.39	899	000	*	£ 9 9	0.1 1.30	9.7.0		1.39	8.88	•••	'n	1.38	222	<u> </u>		1 66. 1 66.	នុនុន្	8,00 1	<u>ннн</u> ђ	8.6.6 111	<u> </u>		<u></u>	<u></u> 888	222	800	1
Diecharge 3.0 amps fr 5 min 26 min 30 min 32 min 33 min 35 min	or 35 mimtee	1.28 17 19 20	88888	28888	- 10" - 10" + 54 + 194	-11 -11 -12 -13	<u>ສສສສ</u>	85888	2013	17 17 18 18 18	1.26	82233	-15" -10" -10"	- 17 - 17 - 18 - 18 - 18 - 18 - 18	กุฎสุสุส	82833	100000	29 1 16 1 18 1 18 1 18 1	ถุมุมชุม	<u>77+77</u> 88938	Loso	1181.	ສຸສຸສຸສຸສຸ	17+77 19988	<u></u>	211111 211111	มมมมม	28388	'n0355
Charge 1.615 mps for 32 min 38 min 64 min	r 65 minutes	1.39	1.30	000	+25# +24#	8,9,9 8,9,9	8.5.5	000	1126	1.37	1.30	000	+15#	1.39	888			37 1	8,8,8	8.00	111	1 65. 1 65.	888	7+ Roo	<u>. न न न</u> हे8	<u></u>	888	<u>++</u> 8.00	33
Diacharge 3.0 amps fr 1 min 19 min 23 min 33 min 36 min 41 min 43 min 50 min 50 min	or 50 milautes	1.31 1.22 1.22 1.22 1.22 1.22 1.22 1.22	427777777777 4272727777777 68.	8.0.01.05.944 9.01.01.05.944	- 2" - 8" +11# +27# +40# +52#	745 28 18 1.1 18 2.3 1 . 7 5 2 8 18 1.1 18 2.3 1	22222222	4.0 0 % 9 % 9 % 9 % 9 % 9 % 9 % 9 % 9 % 9	11.0		1.128	8.3.8.2.5.8.5.4		237 248 258 258 258 258 258 258 257 258 257 257 257 257 257 257 257 257 257 257	*2222448663	2. • • • • • • • • • • • • • • • • • • •	61.688338		82222222222 1112	8:5:0:5:6:5:6:4 +++++	11111111111 22222222	11111111111111111111111111111111111111	80.00 80.000	300838844	 5555555	<u></u>	<u> </u>		
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	st Mumber Test Description	charge 3.0 amps for 30 minutes a min 30 min 15 min 14 min 14 min	urge 1.615 ampe for 65 minutes 8 min 18 min 18 min 18 min 18 min 5 min	ciarrys 3.0 amps for 30 minutes 3 min 5 min 5 min 7 min 7 min 3 min 9 min 0 min	
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TABLE 7 (continued)

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Para	Test Numbe	er Test Description							-		<u> </u>																	
	A. Charge 1. 40 min 50 min	615 amps for 65 minutes	1.39	1.30	8,00	<u> </u>	1111 1111	888			1.1 89 1.1 80 1.1 80	- <u> </u>	°		0 1.3 0 1.3 0 1.3	000	فة 	.	1.2	000	. 50 1	1.39	1.37	<u></u> <u>8</u> 00	-12,	36. 1 1.39 1.39	1.30	
	64 min B. Discharge 5 min 30 min 32 min 32 min	a 3.0 anga for 35 minutes	1.29 17 18 18 18	<u>ສ</u> ສສສສ	88383		29 19 18 18 18 18 18	****	0 0 9 0 9 1 1 + + + +		169 1.1 16 1.1 18 1.2 18 1.2	<u> </u>	- 19 - 19 - 19 - 13 - 19 - 19 - 19 - 19 - 19 - 19 - 19 - 19		88869	<u> </u>	00 - 15	<u> </u>	5555 11111			N N N N N	1.25		51-1-1 1-1-1-1 1-1-1-1 1-1-1-1 1-1-1-1 1-1-1-1 1-1-1-1 1-1-1-1 1-1-1-1-1 1-	1.29 16 18 18 18	2.1 2.1 2.1 2.1 2.1	
	C. Charge 1. 32 min 38 min 64 min	.615 amps for 65 minutes	1.37 1.39 1.39	1.30	8.00	110 110	1 66.	នុន្តន	0 0 0 +17	 **	39 1	888	20 +174	<u> </u>	55 55 55 55 55 55 55 55 55 55 55 55 55	<u>888</u>	119		1.2		3 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1.4	1.33	•••	** **	1.37	1.30	N 00
	D. Discharge 1 min 28 min 28 min 35 min 35 min 41 min 45 min 56 min 50 min	a 3.0 maps for 50 minutes		1.28 1.22 1.22 1.22 1.22 1.23 1.16 1.16 1.16 1.28	4.1 2.1 2.1 2.1 2.1 2.1 2.1 2.1 2.1 2.1 2		1111111 1111111 1111111111111111111111	22222222222222222222222222222222222222	3202380000		88119111	<u> </u>	4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4		22222222222	22222222222	\$2355550 +++++++		1111111 12222222				0000111111111111		2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2			
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TABLE 7 (Continued)

l-15 haf.to 4th e Pres-Met. I sure *** 37 + + + + + 00009928644 38283 0000 2222222228 1.26 1.26 1.26 8.1.1. II » 1.32 1.23 1.18 -.17 21.12 11.15 a Prea - 28" - 28" - 28" - 28" + 6 - 15 - 21 80.99.5 1-14 Not. 1 4th Not. 1 8,000 2.0022.00 22228 1.29 Cell 22125 ¥.1.35 e Pres-114 134 -18" -18" -18" -118" -118" 0088898144 Cell Laf. to Ath -V Meg. I ខ្ខន្តនុន្ត 2222 1.29 22211222 1.32 1.18 1.1 1.18 ¥.1 8.1 1.39 811.128446. #11+ #6 + a Pres-13 1 1 1 + + + + + -15° +18° +27° +27° +46° 32883 1-12 Maf.to/db Neg. 1.27 1.26 1.26 1.26 1.30 113 11.58 11.18 1.32 1.23 1.18 1.16 -.16 1.35 ** 30.48 Cell Maf.to 4th a Prea-V Neg I aure + ++ + + 22,8888,880 **** 0080 1.26 1.8.9.1 1.15 1.13 1.14 1.15 ¥.1.36 1-10 Laf.to4th e Pres-Neg. I sure * - - * • 22.4.1.1.80.200 **** 22.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28.1.1.28. 22.1.28 8.1.3 Cell 22.1 1.1 1.1 1.1 1.1 ¥.11.36 Ath e Pres-0.4 I H ** + + 32883 1000 <u>_</u> 22.122.1 22.1 22.1 31.1 191.1 191.1 191.1 8.1.3 Ju i 8911919191 Cell P 1.36 Test Description winutes minutes ai mutas 2 3 3 for for for Ì i i 3.0 Test Numbe ۰ Ŀ. . Raf.

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		Test Bescription		Charge 3.0 amps to signal		110 44	120	128 min	135 min	I40 min	143 mtn		149 ain 155 ai n	Discharge 3.0 amps to 1.0 wolt	Capacity	Charge 3.0 amps	33 min	82 min		127	138	142 min				200 mtn 200 mtn	210 min	Discharge 6.0 amps to 1.0 wolt	Capacity	
		i i	1.15																											

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DETAILED DATA OF CEMERE CONTACL AT NOON (A A and A.7 of F2A4-27-128)

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TABLE 8 (Continued)

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		r. : rai Test Wimber Test Description		Charge 3.0 amps to signal	60 min	93 min		120 min	128 mdn	135 min	140 min	143 min	145 min	149 min	155 min		Discharge 3.0 amps to 1.0 volt	Capacity	Charten 3.0 ampe	33 mto	82 mfn	105 mdm			142 min	147 min			200	210 mdn - All celle off encept cell #14	Discharge 6.0 amps to 1.0 volt Canacity		#14 after approximately 24 hours overcharge at 3.0 amps	 	
	Ŀ	2 4	L			_																												 	

TAKLE 8 (Continued) Page 43

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6 TIRI

OVERCEARCE TESTS AT 0°C, 25°C and 50°C (5.1 to 5.6 of F24A-FB-128)

Ref	ľ	Description of Test	Time,			1-2					1-13		
Para.	υ		Min.	Cell VDC	Ref. to Neg.	4th e I	3rd Neg.	Pressure	Cell VDC	Ref. to Neg.	¢th ● Ι	3rd Weg.	Pressure
5.1	0	Charge 6.0 A	0.	1.27	1.28		, 8	-20"	1.27	1.27		8	-26"
			4 I	 	1.36	0.09	8	-11.	1.46	1.33	90.06	8	-20
			ង	1.47	1.36	0.08	8.8	5	1.48	1.33	8.6	ŝs	-19"
			2 2	9-1 1-51	1.37	58	3.6	. ۲. ۱۱	1.52	1.36	0.06	6	-11-
			54	1.53	1.37	0.095	80.	# # +	1.53	1.36	0.075	68	- 6 -
			94 24 24	1.56	1.39	11.0	60 ·	# # + +	1.57	1.38	28.0 78.0	s =	, i , i
			22	1.60	1. 1.	121	1	3	1.61	1.38	4E.0	1:	5
			55 75	1.61	1.39	0.36	.14	-10"	1.62 1.61	1.35	1.2	.11	.
			58.5	1.59	1.37	5.5	.38	-15"	99.1	1.34	1.8	.18	-2 <u>-</u>
			62 62	1.59	1.37 1.36	3.0	8C.	-15"	1.59	1.32	2.8 7.8	3.U)	រុង
			5.5 2.5	1.56	1.35	3.4	67.	-14"	1.58	1.31	3.2	8°5.	22-
		Off charge Discharge 6.0 Å	65		5.2	- HE						2 AB	
5.2	20	Charge 6.0 A	2 17	1.34	1.29 1.29	0.25 0.12	8.2	-111	1.34	1.28	0.20 0.11	5 5	-20"
			16.99	1.41	1.29 1.29	0.70	30. 32. 24.	.11-	1.41	1.28	0.65 1.0 2.1	ຍ່ອ່ອ	- 24"
		#1-2 Off charge	ននះ						1.41	.1.27	•	11.	
			358							1.2	0.4	ંડ્રંગ	-17"
		Off charge	59									_	
·		Discharge 6.0 Å			4.2	¥,					-	2 A B	
								=					
				-									

ſ	-																 		 	-1
		Pressur	10, 11, 11,	-14 -15	-22"	-22"	<u></u>	-22" -16"	÷÷	-16"	-21"	-26"		-24"	-24" -24"		 	<u>. </u>		
		3rd Neg.	.02	8 S		.54	АН	.02 01	1 <u>0</u> 10	10.0	583	8.2.5	.23	.30	.38 .51	АН				
	1-13	4th e I	.15	.28	. 6 .	. æ.	4.5	81.	• •	00	500	. 4 . 4		1.9 2.1	2.0	5.0				
		Ref. to Neg.	1.28 1.28	1.28	1.27	1.27		1.28	1.27	1.27	1.27	1.26	1.26 1.26	1.26 1.25	1.25 1.25					
		Cell VDC	1.35 1.39	1.41	1.42	1.42 1.42		1.33	1.36	1.41	1.41	1.42	1.41 1.41	1.41	1.41 1.41		 			
		Pressure	ۍ ه د	0	-12"	-12"		-11"		1	- 12"	-15"		-15"	-15-					
		3rd Neg.	.03 .03	5.2	8 ŝ;	. 75 27.		5.0	55	55	<u>5</u> 5	8 [.]	.38	.50	22.	·	 			
	1-2	4th e I	.20	.20	2.2	3.0	AH	81.	200	0	00		1.6		2.3	AB				
		Ref. to Neg.	1.29 1.29	1.29	1.29	1.29	4.5	1.29	1.29	1.29	1.29	1.28	1.78	1.28	1.28	5.2				
TABLE 9 Continued		Cell VDC	1.35 1.39	1.41	1.42	1.42 1.42		1.33	1.37	1.41	1.41	1.42 1.42	1.42	1.41	1.41				 	
		Min.	7 19	8:	\$ \$	5 52	65	4	185	29 29 29	87 100	118 120	123	127	វិគីគី	132				
		Description of Test	Charge 6.0 A				Off charge Discharge 6.0 A	Charge 3.0 A								Off Charge			 	
		L	50					50									 		 	
	ſ	Ref. Pare.	5.3					5.4												

Pressure -20" -12" - 7" - 6" -16" 23811166988989889388 Srd Neg HZ . AA 5.6 5.5 • 82558 4.0.5 114 Ref. to Neg. $\begin{array}{c} 1.29\\ 1.29\\ 1.29\\ 1.29\\ 1.29\\ 1.22\\$ $\begin{array}{c} 1.28\\ 1.28\\ 1.29\\ 1.29\\ 1.29\\ 1.29\\ 1.29\\ 1.29\\ 1.25\\$ ĕ $\begin{array}{c} \textbf{1.39}\\ \textbf{1.41}\\ \textbf{1.45}\\ \textbf{1.45}\\ \textbf{1.45}\\ \textbf{1.55}\\ \textbf{1.5$ Cell Pressure 888335588758888005 +++++ 3rd Neg. 882288888868586858688 88675833333 886758333333 • 201102 1-2 4th I 1.4 3.8 4.6 Ŧ EV 3 5.7 5.5 Neg. Ref. Cell VDC 1.39 1.44 1.44 1.45 1.45 1.48 1.48 1.50 1.50 1.51 1.51 1.49 Time, Min. Description of Test Off charge Discharge 6.0 A Off charge Discharge 6.0 A Charge 6.0 A Charge 6.0 A ង ង Ref. Para. 5.5 5.6

TABLE 9 (Continued)





Schematic of Auxiliary Electrode Cell

- 47 -



- Lucite blocks
 Corrugated perforate plastic
 Test Electrode 3 cm x 3.3 cm
- 4. Non-woven nylon separator
- 5. Partially charged positive plate 4.5 x 5.5 cm
 6. Container

- 7. Tape 8. 31% KOH

Figure 2

Gas Electrode Test Cells

- 48 -

CYCLE: 215 MIN. TM Contact (min.)

65	1 00	165	215	
0	65	õ	165	
l	I	I	I	
Open	Closed	Open	Closed	



Automatic Cycle Circuit

Figure 3



Outline Drawing of General Electric Cell Cat. No. 42B006XB02

- 50 -



Figure 5

Schematic Drawing of 4th Electrode Cell Construction

- 51 -







- 54 -











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APPENDIX

GENERAL ELECTRIC COMPANY PROCESS

INSTRUCTION P24A-PB-128

(Paragraphs 3.0, 4.0 and 5.0)

- 3.0 FORMATION Before starting formation, the 3rd electrode is attached to the negative with a 10 ohm resistance, and the 4th electrodes are shorted to the negative terminal with 10 milliohms resistance. Cells are wired this way in all subsequent testing.
 - 3.1 First Cycle

Follwing a 16 - 24 hour stand, cells are charged for 16 hours at .60 amps and discharged at 3.0 amps to 1.0 volt/cell. Measure and record end of charge voltage and pressure, and time to discharge to 1.0 volt.

3.2 Second Cycle

Cells are again charged at .60 amps for 16 hours and discharged at 3.0 amps to 1.0 volt. Maximum charge voltage and pressure, and time to discharge to 1.0 volt are recorded. Discharge is continued to -1.0 volt and cells are vented to the atmosphere. Pressure before venting is recorded.

3.3 Third Cycle

Cells are charged at .60 amps for 16 hours and discharged at 3.0 amps to 1.0 volt. Data is recorded as before.

4.0 ACCEPTANCE TESTS

NOTE: Temperature is 25°C ‡ 3°C unless otherwise noted.

4.1 Voltage Stabilization

Cells are charged at .60 amps until voltage stabilizes as indicated by less than 50 millivolt change in one hour. Stabilized voltage and time on charge are recorded.

4.2 Internal Impedance

Internal impedance of each cell is measured, positive to negative, 3rd electrode to negative, and 4th electrode to negative. Record impedances in milliohms. 4.3 Capacity Discharge

Discharge each cell at a 3.0 amp rate to 1.0 volt/cell. Measure and record time to 1.0 volt.

4.4 Internal Short

A completely discharged cell shall be short-circuited for 16 hours and be charged at .750 amps for 5 minutes. Open circuit voltage after 24 hours shall be at least 1.16 volts to be acceptable.

4.5 <u>Overdischarge ("Sherfy Cycle") at 25°C</u> Cells are given four cycles to demonstrate overdischarge control principle as follows:

a. Charge at 1.615 amps for 65 minutes

b. Discharge at 3.0 amps for 35 minutes

c. Charge at 1.615 amps for 65 minutes

d. Discharge at 3.0 amps for 50 minutes

Measure and record cell voltages and pressure at end of each step for each cycle.

4.6 Overcharge Control - Step 1

Charge each cell at 3.0 amp rate until 3rd electrode-negative signal reaches at least 0.50 volts. Measure and record time to signal, 3rd electrode signal and cell voltages. Discharge cell at 3.0 amp rate to 1.0 volt/cell and record time to 1.0 volt.

- 4.7 <u>Overcharge Control Step 2</u> Charge each cell at 3.0 amp rate until 3rd electrode to negative signal reaches at least 0.50 volts. Continue charge for 90 minutes past onset of signal. Record signal voltage, and cell voltage and pressure at end of overcharge. Discharge cells at 6.0 amp rate to 1.0 volt/cell and record time to 1.0 volt.
- 5.0 <u>QUALIFICATION TESTS</u> Select two cells at random from the lot and subject to the following tests:

5.1 Overcharge at 0°C

Cells are placed in chamber at 0° C and after 4 hours minimum stabilization, are charged at a 6.0 amp rate until 3^e to negative signal reaches at least 0.50 volts. Cells are discharged at 6.0 amp rate to 1.0 volt/cell. Measure and record charge time, 3^e signal at termination of charge, cell voltage and pressure at end of charge, and time to discharge to 1.0 volt.

5.2 Overcharge at 50°C

Cells are stabilized at 50°C for 4 hours minimum and charged at 6.0 amp rate until 3rd electrode to negative signal reaches at least 0.50 volts. Cells are then discharged at 6.0 amp rate to 1.0 volt/cell. Measure and record charge time, 3^e signal at end of charge, end of charge cell voltage and pressure, and time to discharge to 1.0 volts.

- 5.3 <u>Overcharge at 50°C 2nd Cycle</u> Repeat test of Paragraph 5.2.
- 5.4 Overcharge at 50°C 3rd Cycle Charge cells at 3.0 amp rate until 3rd electrode to negative signal reaches at leat 0.50 volts. Cells are discharged at 6.0 amps to 1.0 volt/cell. Record charge time; cell voltage, 3^e to negative voltage and cell pressure at end of charge, and time to discharge to 1.0 volt.
- 5.5 Overcharge at 25°C

After a minimum 4 hours stabilization at 25° C, cells are charged at 6.0 amp rate to a 3^{e} signal voltage of at least 0.50 volts. Discharge cells at 6.0 amps to 1.0 volt. Measure and record charge time, cell voltage, pressure, and 3^{e} signal voltage, and time to discharge to 1.0 volt.

- 5.6 Overcharge at 25°C 2nd Cycle Repeat test of Paragraph 5.5.
- 5.7 Overdischarge Cycle

Cells shall be continuously cycled until performance has stabilized (24 hours minimum) at each temperature of 5°C, 25°C, 45°C through the overdischarge cycle of Paragraph 4.5. Measure cell pressures during each temperature cycle and record when operation has stabilized.

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