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SEALED LITHIUM INORGANIC ELECTROLYTE CELL

GTE Laboratories, Incorporated

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SEALED LITHIUM INORGANIC BATTERY

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The effects of iron impurities in the electrolyte on the lithium anode passivation have been studied further, using both the standard cell hardware and the isolated cell components within a glass cell arrangement. Cold rolled steel was found stable in the inorganic electrolyte if it was held strictly at the potential of lithium. The slightest deviation of the potential from this value resulted in severe corrosion, generated iron species in the solution, and caused the passivation effects described in previous reports. A large area of cold rolled steel cans, exposed to the

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electrolyte in the present type of cell construction at an appreciable distance from the contact with lithium, appears to form dissolution sites generating more than tolerable amounts of iron in the solution. For this reason, the idea of building the large cells with cold rolled steel hardware will have to be abandoned. Attention was gradually switched to studies of stainless steel as the cell hardware.

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SUMMARY

The effects of iron impurities in the electrolyte on the lithium anode passivation have been studied further, using both the standard cell hardware and the isolated cell components within a glass cell arrangement. Cold rolled steel was found stable in the inorganic electrolyte if it was held strictly at the potential of lithium. The slightest deviation of the potential from this value resulted in severe corrosion, generated iron species in the solution, and caused the passivation effects described in previous reports. A large area of cold rolled steel cans, exposed to the electrolyte in the present type of cell construction at an appreciable distance from the contact with lithium, appears to form dissolution sites generating more than tolerable amounts of iron in the solution. For this reason, the idea of building the large cells with cold rolled steel hardware will have to be abandoned. Attention was gradually switched to studies of stainless steel as the cell hardware.

1. INTRODUCTION

The tests carried out in this program have demonstrated that it is not yet possible to construct lithium cells that would be capable of high discharge rates after a prolonged storage without a time delay in reaching a stable operating voltage. It has been established that the cause of the delay is the build-up of a passivating film on the lithium anode.[1] The effect this film has on the discharge characteristics has been well documented by this and other laboratories.[2]

It has been previously reported[3] that iron species introduced into the electrolyte via $AlCl_3$ or through corrosion of cell hardware produces the strongest negative effect on the passivation of lithium anodes. This effect of iron warranted further study of the contribution of the cell hardware to the amount of iron present in the cell.

Once the source of iron was identified, it was of interest to establish the distribution of iron within the active components of the cell; bearing in mind that the lithium anodes would, most likely, act as scavangers for the dissolved iron species. It would also be of interest to establish the role of the iron deposits on the anode in the formation of the passivating film and on the possible loss of lithium due to the galvanic corrosion.

Experiments were designed to determine the iron contribution from cold rolled steel cans to the electrolyte and Li anode, while holding the cold rolled steel potentiostatically at the lithium potential in a specially designed cell and by analyzing the electrolyte periodically for iron.

Assuming great differences in the rate of iron generation between the cold rolled steel and the stainless steel, when each of these materials was held in contact with lithium, one should be able to demonstrate the difference in the voltage delay after storage at elevated temperatures with the cell made with each of the two hardware materials. This comparison is described later in the report. Our past experience with the stainless steel used in the construction of small cells did not warrant an immediate investigation of the corrosion of this material, although it was thought that the pertinent data should be generated at some later date. The investigation into other hardware materials might be warranted should the test with the stainless steel show a similar effect, since the negative effect of the iron species has been satisfactorily documented in the earlier tests with completed cells. The tests with

finished cells were designed to include the discharge capacities as well, in an effort to establish whether the various hardware materials might be of consequence to a possible loss of lithium by the local galvanic corrosion.

In the continuing effort to gather more storage data, several cells made in cold rolled steel cans were taken from storage after three months at room temperature. The delays and capacities will be checked. This data will also give a better indication of cold rolled steel's compatibility with the system.
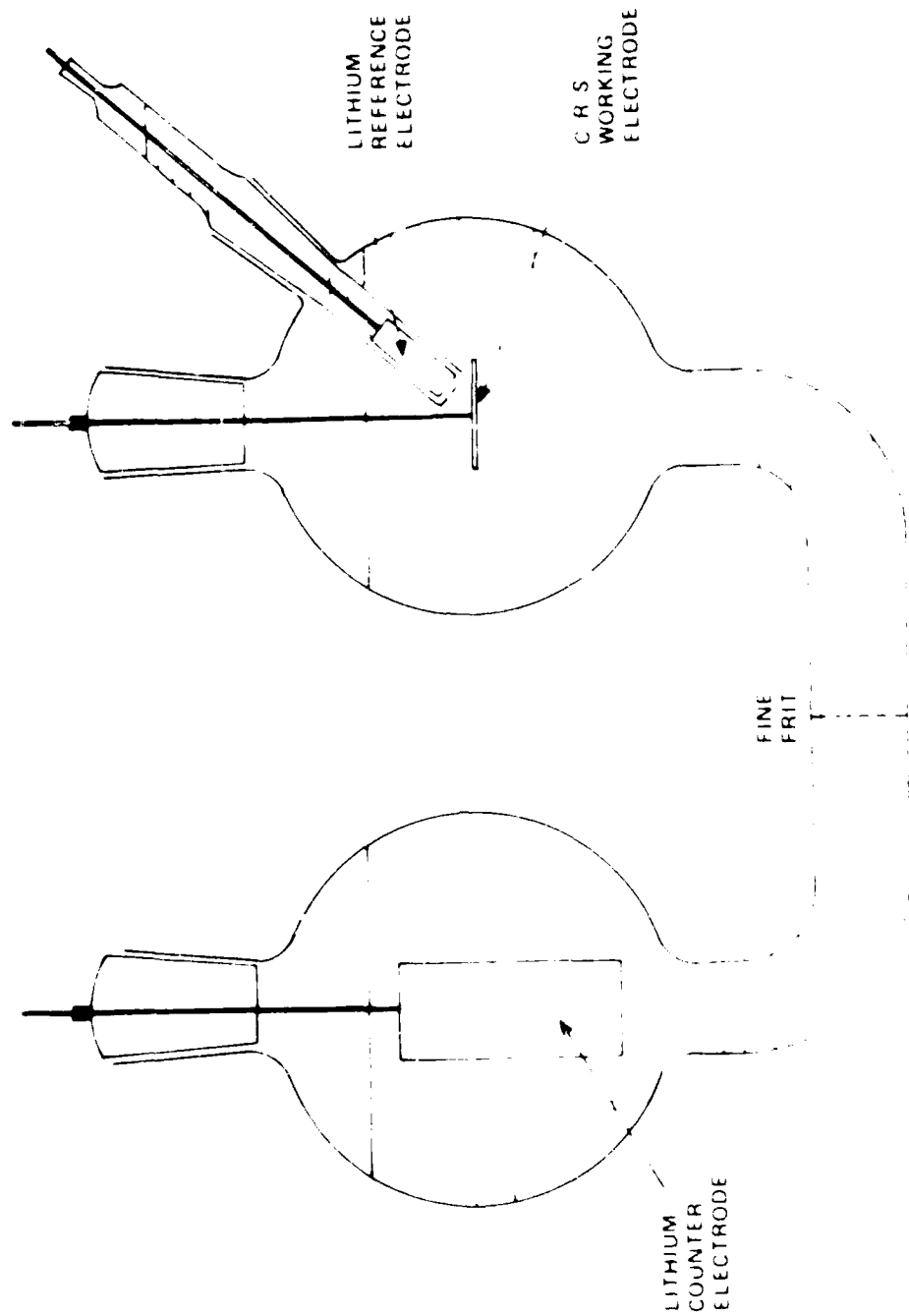
LITHIUM
REFERENCE
ELECTRODE

C R S
WORKING
ELECTRODE

LITHIUM
COUNTER
ELECTRODE

FINE
FRIT

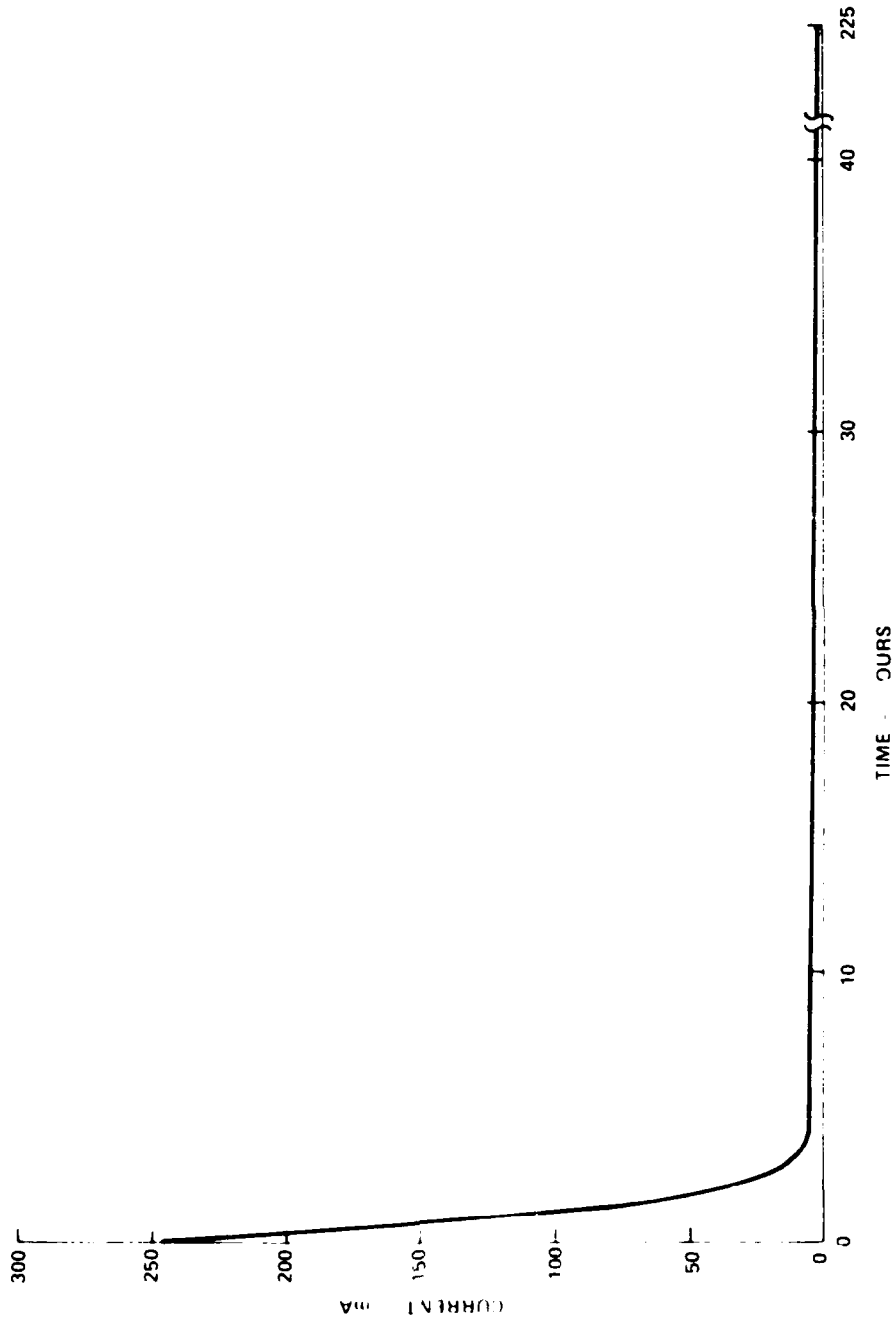Figure 1. Glass Cell Arrangement for Corrosion Studies

Figure 2. Self Discharge on Cold Rolled Steel

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2. CORROSION OF COLD ROLLED STEEL IN CONTACT WITH LITHIUM

It has been the contention of the authors of this report that there would be no hardware corrosion in cold rolled steel cans, due to cathodic protection of the cans by the lithium. Recent data[4] showed that thionyl chloride is reduced very slowly on the surface of nickel and stainless steel, when these materials were in contact with a lithium anode in the thionyl chloride electrolyte. This study also showed that a passivating film was formed on these materials. It was assumed that similar films would be formed on the surface of cold rolled steel protecting it from corrosion in storage. It was also assumed that the galvanic corrosion of the anode would not result in a significant loss of lithium before the bare surface of the cold rolled steel was completely passivated and the reduction of the thionyl chloride discontinued.

The following experiment was done to confirm the fact that the cold rolled steel, when held potentiostatically at the lithium potential, does not corrode. This experiment was done concurrently with an in situ test reported in the next section.

2.1 EXPERIMENTAL

A glass cell was constructed as shown in Figure 1. A fine frit divided the two chambers. A second joint was built into one of the chambers to accommodate a lithium reference electrode. The tube holding the reference electrode was separated from the bulk of the solution by a fine frit and a slight positive pressure was maintained by overfilling the reference tube. The entire cell was immersed in an oil bath at 72°C and brought to equilibrium. The working electrode was a round disk of cold rolled steel 11.28 cm^2 (one side). The Li reference electrode was placed directly above the back side of the working electrode in very close proximity. Each chamber contained 200 cm^3 of electrolyte. The working electrode was kept at the potential of the lithium reference electrode, while the current was monitored between the working and the lithium counter electrode.

2.2 RESULTS

Upon initiation of the test, there was a strong cathodic current indicating the reduction of thionyl chloride on the surface of the working electrode (cold rolled steel). The current was 260 mA, or 23 mA/cm^2 of electrode area. As shown in Figure 2, the current decayed in a few hours to 6 mA, after 25 hours to 4 mA and leveled off at

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1.3 mA at 45 hours and remained there until completion of the experiment. This decay is attributed to the forming of a passivating film of reduction products deposited on the surface of the working electrode.

Calculating the amount of electrolyte lost to reduction over the course of one month on this surface area, using an average of 130 mA for the first 2 1/2 hours and 1.3 mA for the next 717 hours yields:

$$(130 \text{ mA}) \ (2.5 \text{ hours}) \quad - \quad 325 \text{ mA hours}$$
$$(1.3 \text{ mA}) \ (717 \text{ hours}) \quad - \quad \underline{932 \text{ mA hours}}$$
$$1.257 \quad \text{A hours}$$

Using a figure of 1.66 g of electrolyte per Ah of capacity, the total electrolyte loss in one month due to this reaction is:

$$(1.257 \text{ Ah}) \ (1.66 \text{ g/Ah}) \ - \ 2.09 \text{ g}$$

This loss took place on a surface area of 11.2^c cm^2, so the loss is 0.185 g/cm^2 of exposed surface area over the course of one month at $72°C$.

The main objective of this experiment was to determine if there was any corrosion of the cold rolled steel at the lithium potential. Ten milliliter aliquots were drawn periodically and analyzed for iron by Atomic Absorption. Table 1 shows the results of these analyses:

TABLE 1

IRON CONCENTRATION IN THE ELECTROLYTE AT 72°C

TIME HOURS	Fe IN ELECTROLYTE (ppm)
0	9
19	9.8
91	13
163	17
225	20

The results are also represented graphically in Figure 3, showing a linear dependence on time.

The loss of $SOCl_2$ by reduction on the surface of cold rolled steel is accompanied by an equivalent loss of lithium, amounting to 0.325 g.

2.3   CONCLUSION

One of the first conclusions to be drawn from this experiment would be a relatively insignificant rate of thionyl chloride consumption as far as the cell capacity is concerned, due to a low surface area of the cold rolled steel directly exposed to the electrolyte in actual cells.

Second, this experiment shows a minimal corrosion of the hardware when the cold rolled steel is held at the lithium potential. If this is true in the case of a battery, the inexpensive cold rolled steel would be the preferred material of construction.

In the next section, this same experiment is done in situ in a cold rolled steel can in an effort to determine if the case of the battery, when connected to the lithium anode, is actually held at the lithium potential, thus preventing the corrosion of the can as the above experiment suggested.
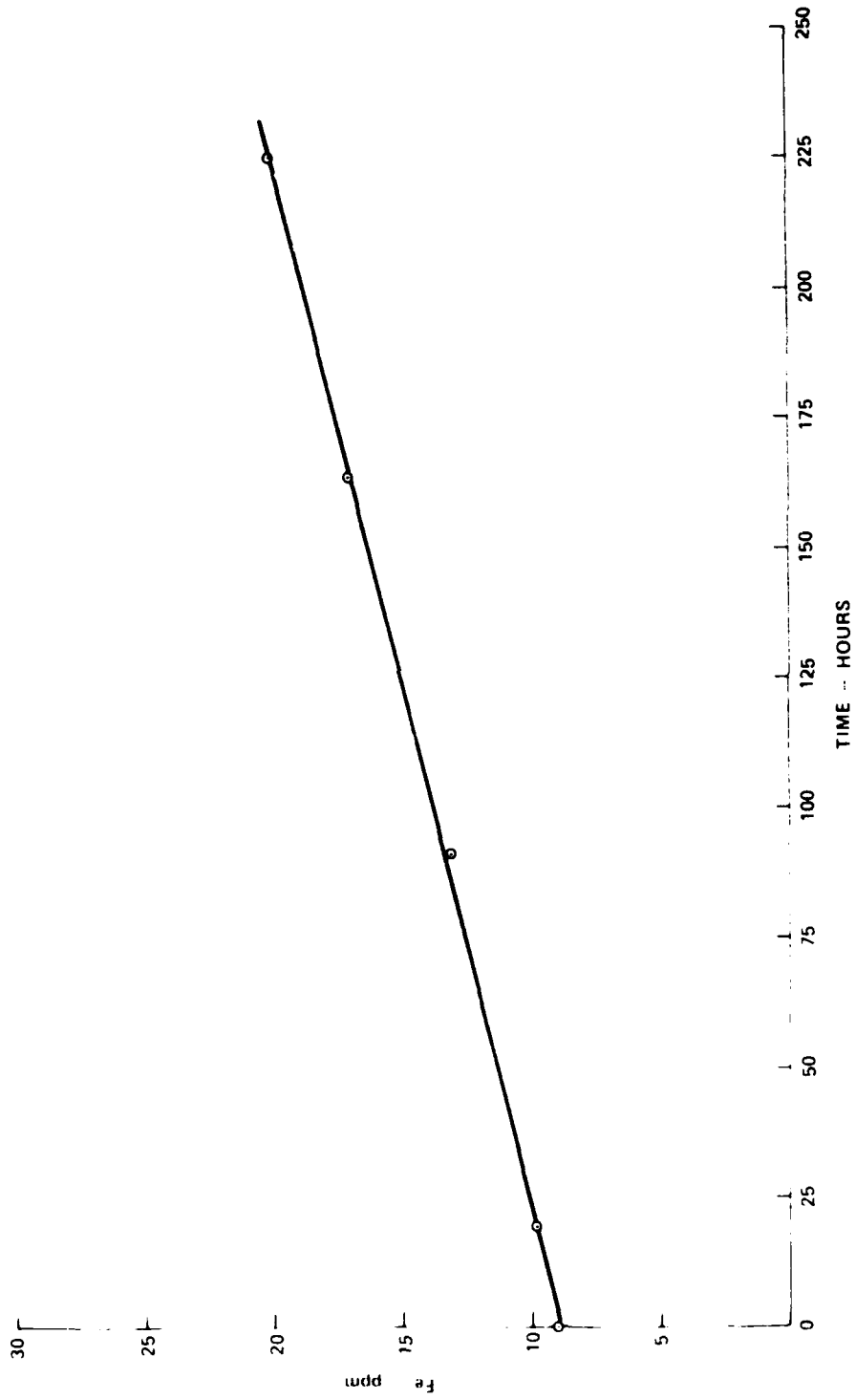
Figure 3. Iron Corrosion at Lithium Potential

3. CORROSION TESTS WITH COLD ROLLED STEEL CANS

A comparison should be made of the corrosion results obtained in the glass cell with those obtained using a more realistic experimental arrangement involving the cell hardware. The efficacy of the cathodic protection of the cell hardware by lithium must be tested under the conditions prevailing inside the cell when a large surface area of the cell hardware remains in a direct contact with the electrolyte. Preliminary experiments carried out in the glass cell have indicated that the geometry of the steel-lithium arrangement might be of consequence. Large sections of the cell can (at some distance from the lithium covered section) will show a severe corrosion in spite of being in contact with lithium. Local voltage differences along the surface of the same piece of steel might be generated and they would be responsible for the corrosion observed. Preliminary experiments of this kind are described below.

3.1   EXPERIMENTAL

Lithium foil was rolled against the interior surface of the C size cans, in the same way in which lithium anodes are formed in the assemblage of the cells with the concentrical arrangement of electrodes (high energy type cells). The cans were filled with the electrolyte and closed without cathodes or separators. They were all stored at 72°C and analyzed at various time intervals for the iron content of the electrolyte by the Atomic Absorption Spectrophotometry (Perkin Elmer Model 460). The can was then emptied of the rest of the electrolyte and washed several times with tetrahydrofuran. The can with the lithium was then plunged into 1600 cm^3 of distilled water. The solution was acidified with HCl to oxidize all the iron that might be present. This solution was boiled down to 250 cm^3 and analyzed by Atomic Absorption to determine if the lithium was taking up iron and at what rate.

3.2   RESULTS

The results obtained in these experiments are presented in Table 2.

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TABLE 2
IRON FOUND IN ELECTROLYTE AND IN THE ANODE
AFTER STORAGE AT 72°C

CELL NO.	DAYS STORED	Fe ELECTROLYTE (ppm)	Fe ON LITHIUM (ppm)	TOTAL mg
1	5	6	27	0.249
2	10	146	8	5.410
3	13	256	15	9.420
4	19	252	4	9.328
5	26	180	16	6.676
6	31	39	35	1.478

The table shows a gradual increase of the iron concentration in the electrolyte with the time of storage for approximately the first two weeks, followed by a gradual decrease over the next two weeks. The iron content of the lithium anode was found to be extremely erratic, suggesting, among other things, that the analytical procedure used may not have been appropriate. The total amount of iron formed by corrosion is shown in the last column of Table 2. Obviously, it shows inadequacies either in the analytical procedures or in the experimental arrangements, since the iron corroded must be found either in the electrolyte or on the anode.

3.3   CONCLUSIONS

The results of this experiment are inconclusive. It was theorized that initially, if there were any corrosion, it should be quite rapid because the can surface is clean and active. As reduction products build up on the surface of the steel the corrosion should decline to a very small rate. It was further assumed that the concentration of iron in electrolyte should increase to a maximum and then decrease to some unknown value due to a plating out on lithium.

Figure 4 graphically depicts the data from Table 2. The graph correlates to the theory as previously explained. There is a gradual rise in Fe concentration, a
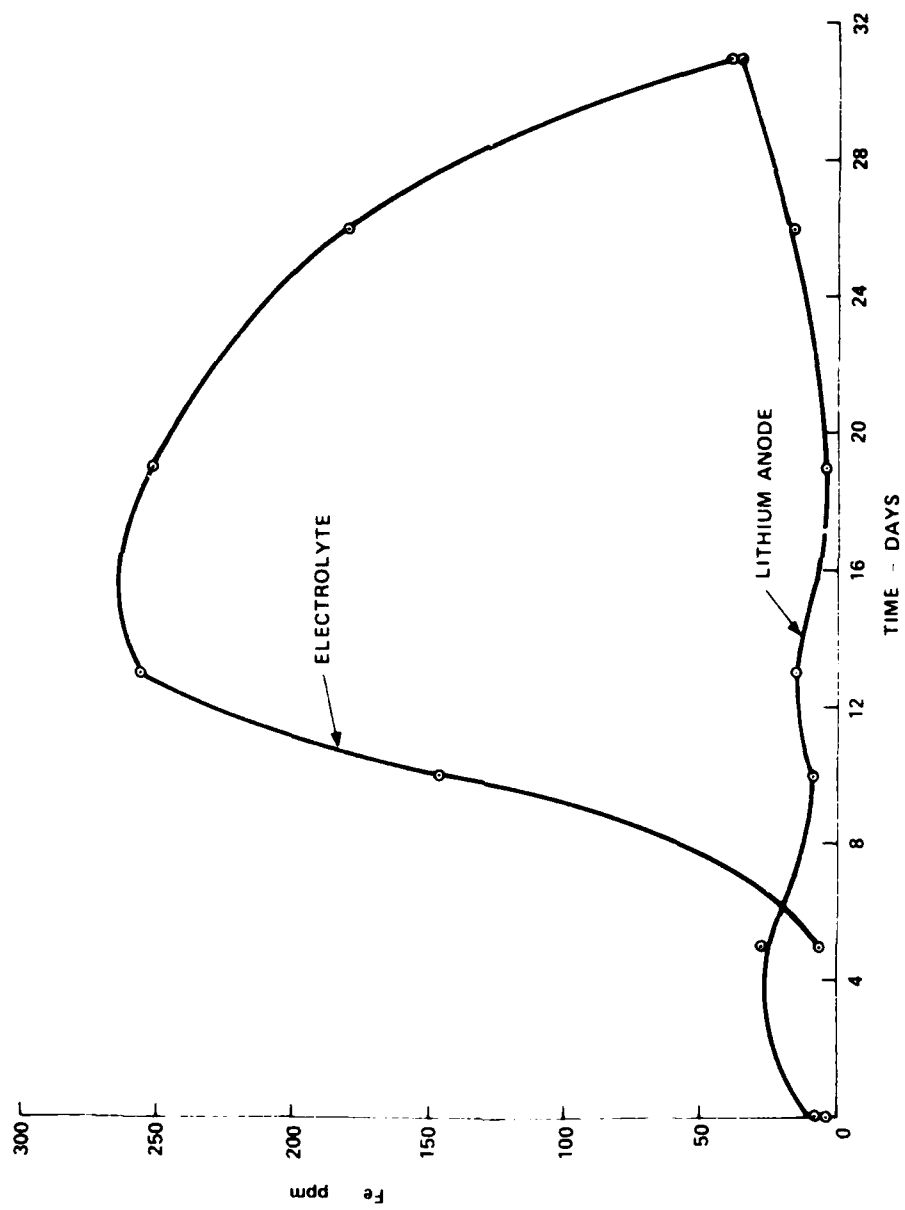
Figure 4. Distribution of Iron Generated by Corrosion in Standard Size D Cells

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leveling out and then a slow decline, all as predicted. The confusion arises when the lithium is analyzed as described in Section 3.1. There is not a significant rise in the Fe concentration on lithium. A blank of lithium will yield an iron concentration of approximately 5 to 10 ppm. As the electrolyte iron concentration was decreasing it was assumed lithium was picking it up, but the lithium analysis did not prove that. One possible explanation may be in the washing technique. After the electrolyte was taken out of the can, it was gently sprayed with THF to wash out any extraneous electrolyte, since it would, in contact with water, attack the can and form more iron in the solution. The iron which plates out on lithium may not be adhering very well and the spraying with THF may be washing it off. This possibility is now being investigated and will be discussed in the next report.

4. THE EFFECTS OF HARDWARE MATERIALS ON THE VOLTAGE DELAY AND THE CELL CAPACITY

An attempt was made to correlate the results of the corrosion studies obtained in the glass cell with the effects of hardware materials on the performance of finished cells. Two sets of cells were built for the purpose, one with stainless steel and one with cold rolled steel hardware and stored at room temperature, 55°C and 72°C. The voltage delay after storage was expected to show a direct influence of the iron present in the solution and also of the rate at which lithium was dissolved in the galvanic corrosion involving the reduction of $SOCl_2$ on the surface of cell hardware. A high rate of the galvanic corrosion involving lithium dissolution should result in a lower value of the voltage delay, since that would keep the surface of lithium free of the passivating film Another consequence of that would be a significant loss in the cell capacity due to the loss of lithium. A high rate of hardware corrosion would have a negative effect on the voltage delay due to the effects of iron plated out on the lithium surface. Another consequence of that would be a better preservation of the cell capacity (lower lithium losses on storage), at least at low discharge rates. A combined effect of both factors should be seen in the discharge of the cells after storage.

4.1 CONSTRUCTION OF CELLS

Twelve cells were constructed for this part of the project; six in cold rolled steel cans and six in stainless steel cans. The wound electrode structure was constructed with computer aided calculations to maximize the cell capacity for a particular thickness of cathodes used.[5]

The component dimensions were as follows:

	LENGTH-IN.	THICKNESS-IN.	WIDTH-IN.
Cathode	14.0	0.033	1.875
Anode	15.4	0.020	1.75
Separator	35.0	0.005	2.00

All cells were hermetically sealed and filled with electrolyte by pre-evacuation.

4.2   TEST DATA

As previously mentioned, six cold rolled steel cans were stored, two each at the three different temperatures, along with six stainless steel cans. After one month they were taken out of storage and allowed to equilibrate at room temperature for one half day. The delays were measured using a current of 340 mA or approximately 1 mA / cm^2 (both sides of cathode). Delays were measured using a Varian Aerograph Model A-5 high speed strip chart recorder and are listed in Table 3. As in previous reports, the delay is defined as any time the voltage of the cell stays below 2.0 volts during the initial phase of discharge. After the delay tests the cells were completely discharged at a constant current of 100 mA to a 2.0 volt cut-off. The capacities obtained are listed in Table 4. The actual discharge curves are shown in Figures 5 through 7.

4.3   ANALYSIS AND CONCLUSIONS

The voltage delay data clearly show a superior performance of cells made with stainless steel cans. The cold rolled steel cans show delay effects as soon as they are exposed to temperatures higher than the ambient. This probably is the result of the high rate of corrosion of cold rolled steel with the iron plating out on the lithium anode. The average discharge voltages for these cells, listed in Table 5 also suggest the negative influence of the iron on the cell performance. The cold rolled steel cells have a consistently lower average discharge voltage at the same discharge current and at all three temperatures involved. At high temperatures, more iron is being produced from corrosion and plated out on the lithium, thus, increasing the internal impedance of the cell. Since the average voltages in the stainless steel cans are higher than those observed in cold rolled steel, it can be concluded that the stainless steel is subject to less corrosion at the same temperatures. At the storage temperature of 72°C, the cold rolled steel cells are completely passivated. The stainless steel cells at 72°C show somewhat lower capacity and also lower operating voltage then those obtained at 55°C or room temperature. These results suggest that the stainless steel cells might be subject to some degree of corrosion of lithium at 72°C. However, the post mortem analysis showed an excess of lithium to be present in discharge cells. This would mean that the lower cell capacity was not the result of loss of active materials but, rather the effect of an increased cell impedance, probably associated with the original passive film formed during storage. This will be investigated in more detail in the next quarter.

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CELL VOLTAGE V

TIME HOURS

PROJECT	801
CELL NO	1 CRS
TYPE	Wound D
MADE	11 26 75
STORED	1 Month R T
TESTED	11 26 75
TEST	100 mA

Figure 5. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

PROJECT : 801
CELL NO : 2 CHS
TYP : Wound D
MADE : 11 26 75
STORED : 1 Month RT
TESTED : 11 26 75
TEST : 100 mA

CELL VOLTAGE V

TIME HOURS

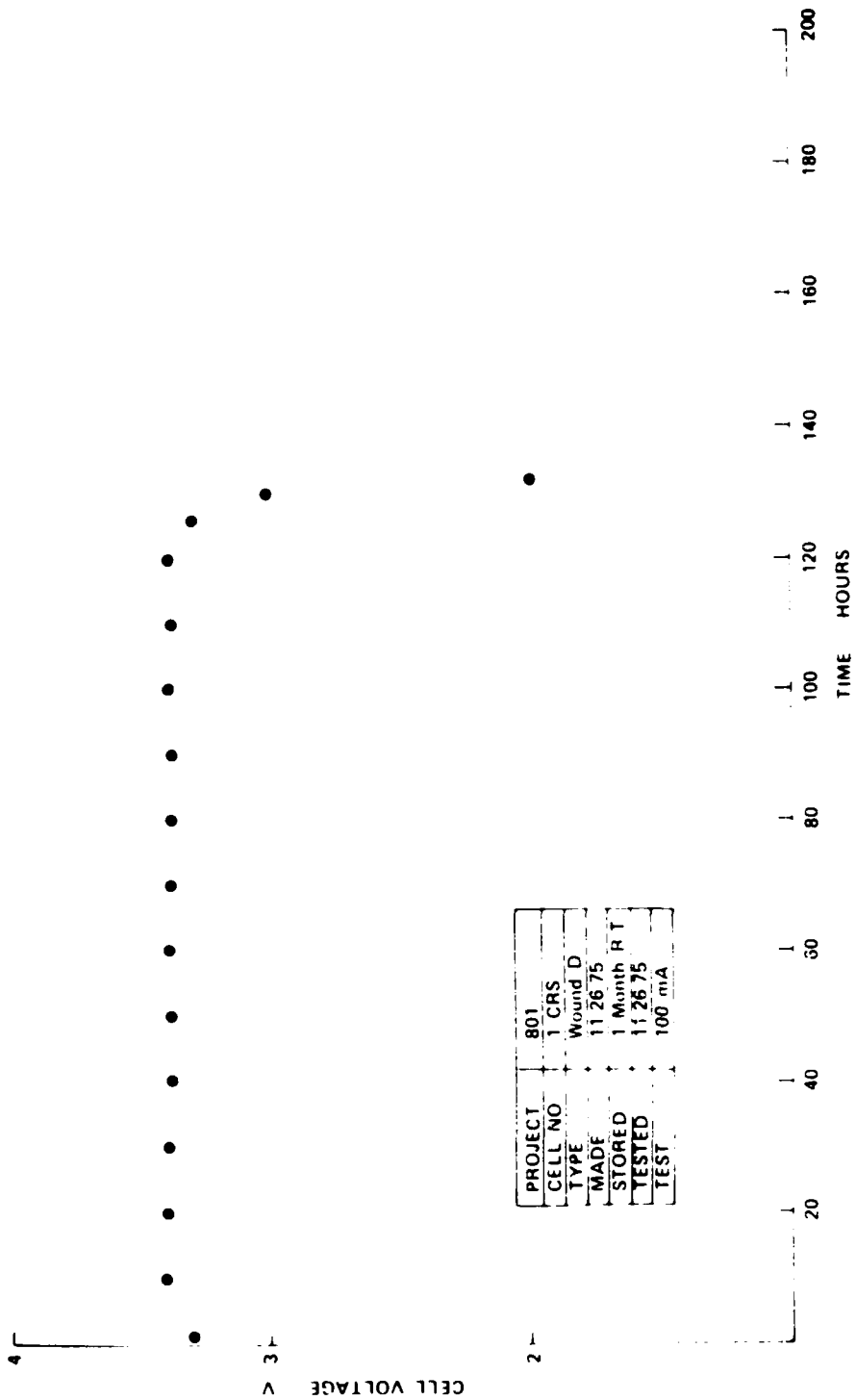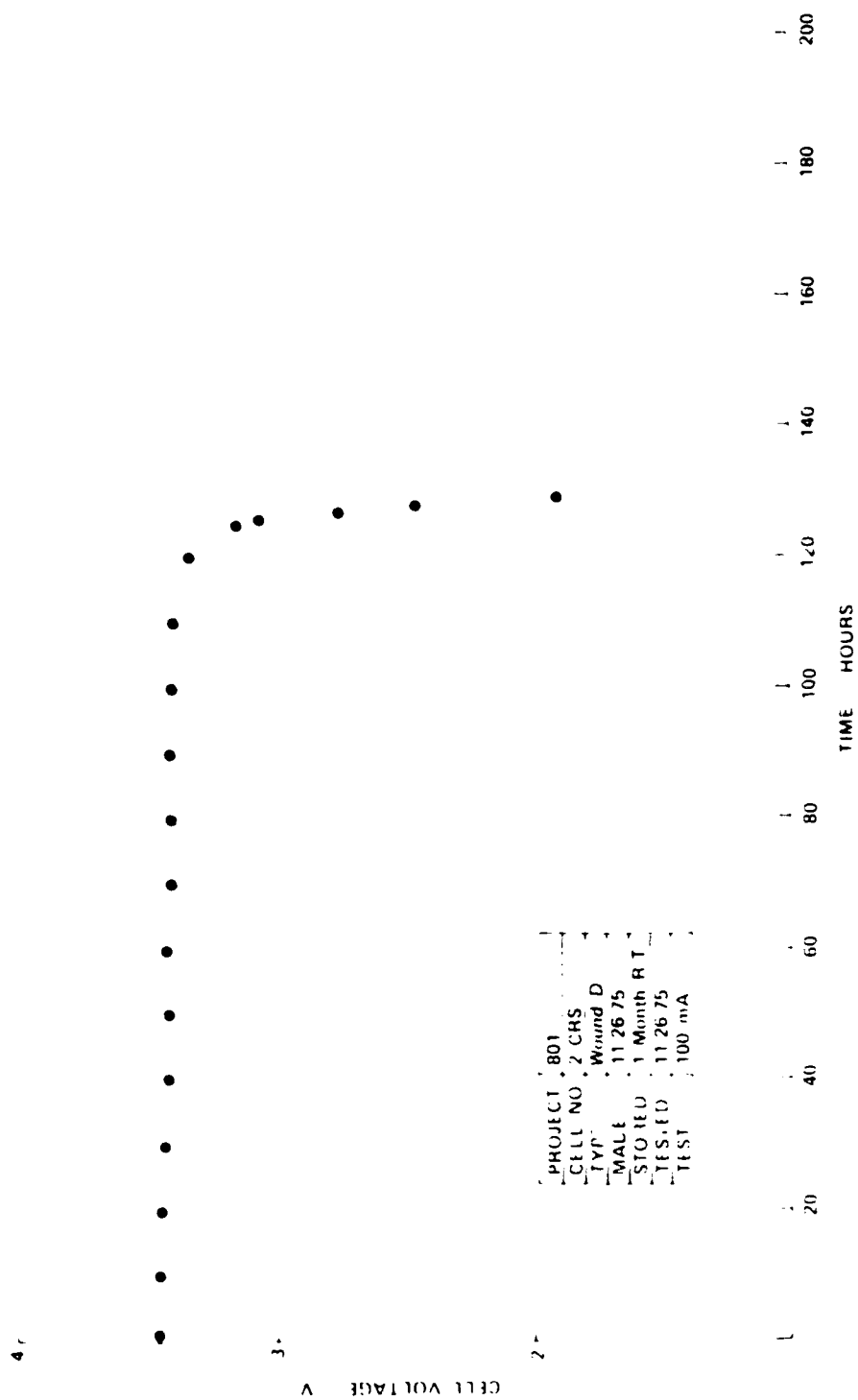20   40   60   80   100   120   140   160   180   200

Figure 6. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures
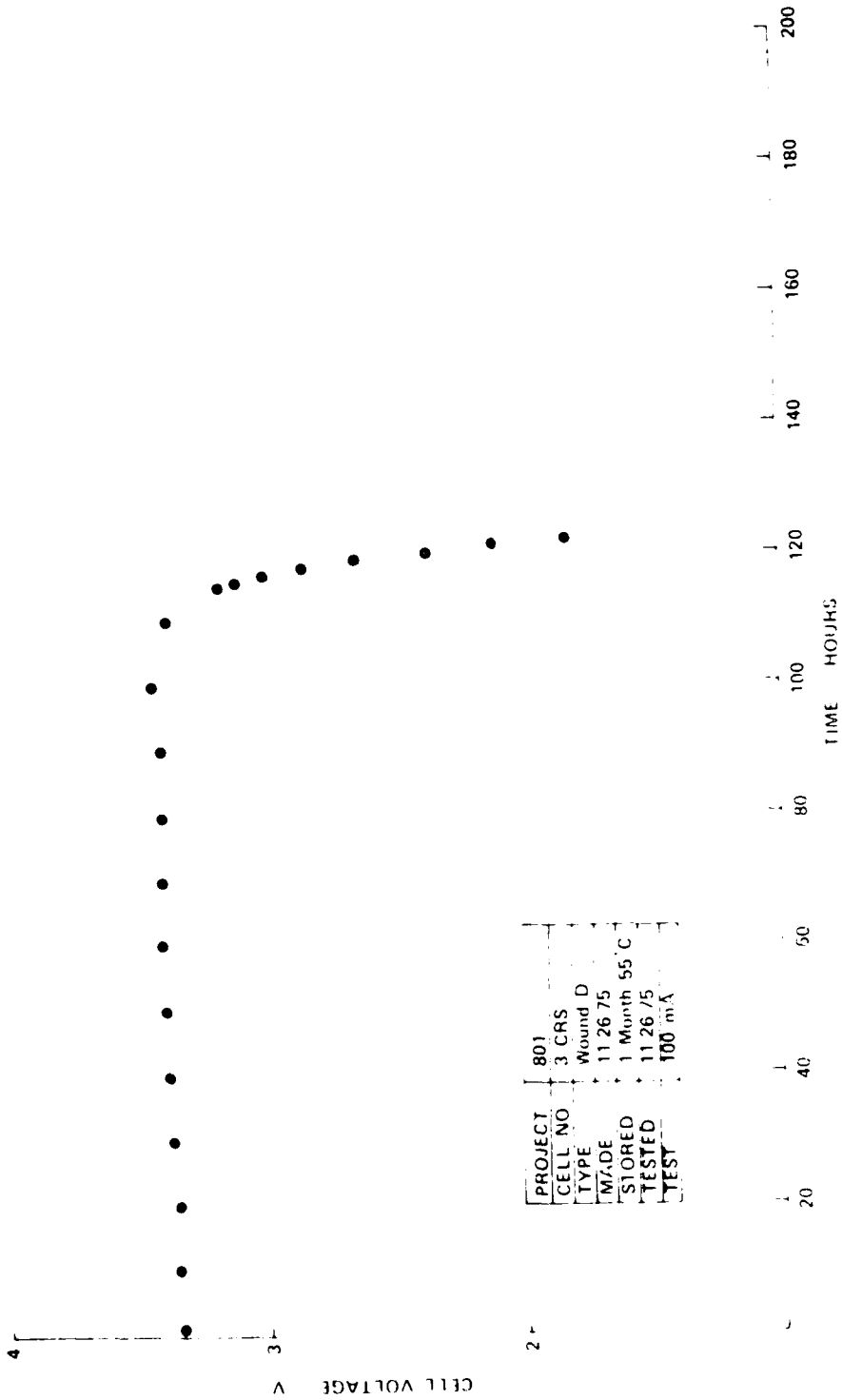
Figure 7. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

TABLE 3

VOLTAGE DELAY AFTER STORAGE FOR 1 MONTH

STORAGE TEMP.	COLD ROLLED STEEL CANS		STAINLESS STEEL CANS	
ROOM TEMP	NO DELAY	NO DELAY	NO DELAY	NO DELAY
55°C	NO RECOVERY AFTER 6 MIN.	15 SEC to 1 V LEVELS OFF AT 1.85V	3.5 MIN	3 SEC
72°C	NO RECOVERY	NO RECOVERY	NO DELAY	NO DELAY

TABLE 4

DISCHARGE CAPACITY AFTER STORAGE FOR ONE MONTH

CELL NO.	CAN TYPE	STORAGE TEMP.	CAPACITIES — Ahr.
1 2	COLD ROLLED STEEL	ROOM TEMP.	13.2 12.8
3 4		55°C	12.2 11.9
5 6		72°C	INOPERATIVE
7 8	STAINLESS STEEL	ROOM TEMP.	13.4 13.2
9 10		55°C	12.8 12.8
11 12		72°C	9.5 9.6

TABLE 5

AVERAGE OPERATING VOLTAGES AT 100 mA AFTER STORAGE

CELL NO.	CAN TYPE	STORAGE TEMP.	VOLTAGE - V
1 2	COLD ROLLED STEEL	ROOM TEMP.	3.390 3.438
3 4		55°C	3.210 3.405
5 6		72°C	INOPERATIVE
7 8	STAINLESS STEEL	ROOM TEMP.	3.438 3.445
9 10		55°C	3.412 3.422
11 12		72°C	3.337 3.340
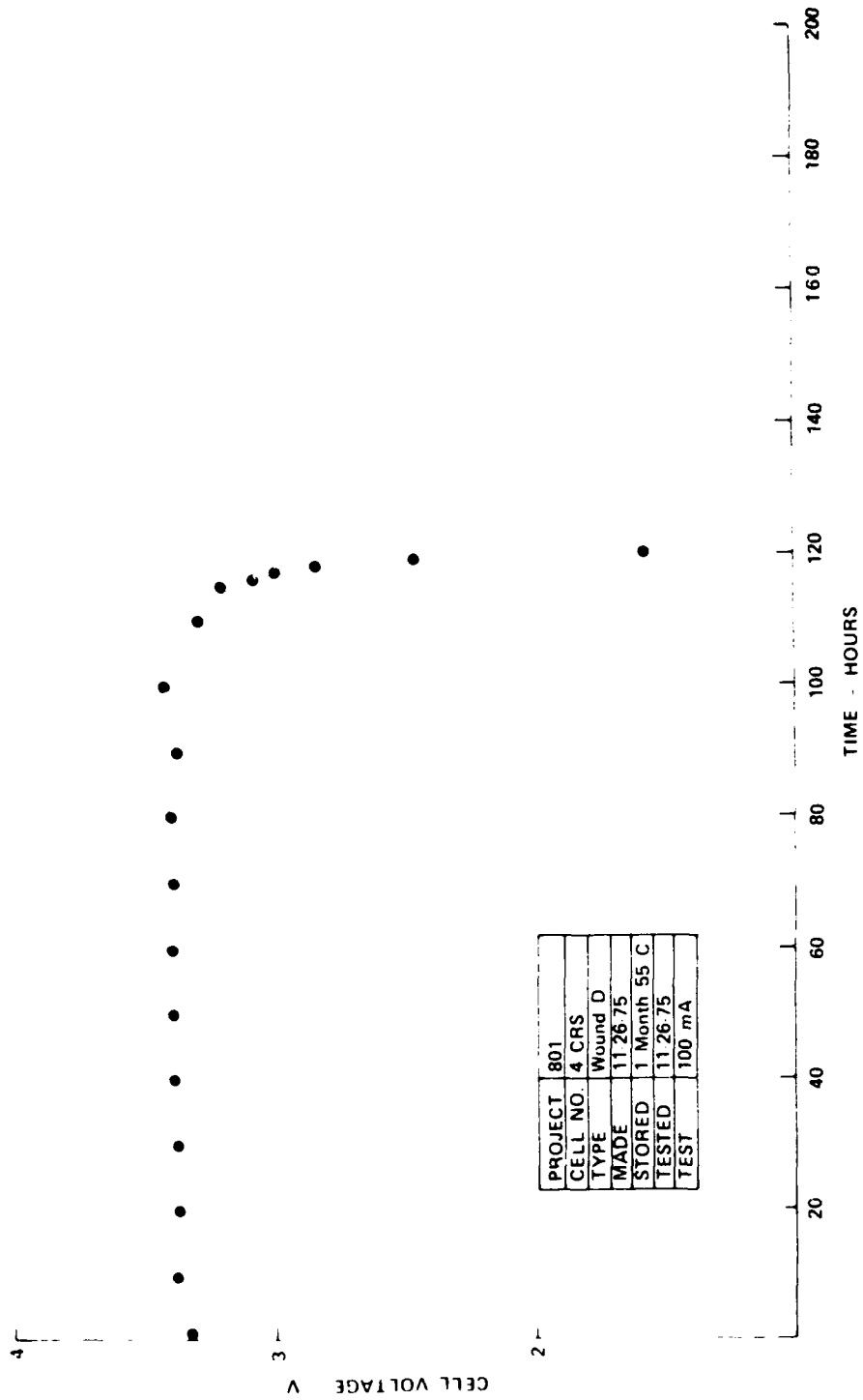
Figure 8. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

PROJECT	801
CELL NO.	4 CRS
TYPE	Wound D
MADE	11 26 75
STORED	1 Month 55 C
TESTED	11 26 75
TEST	100 mA

CELL VOLTAGE V

TIME - HOURS

Figure 9. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

PROJECT	801
CELL NO.	7 S.S.
TYPE	Wound D
MADE	11.26.75
STORED	1 Month R.T.
TESTED	11.26.75
TEST	100 mA

TIME – HOURS

CELL VOLTAGE   V

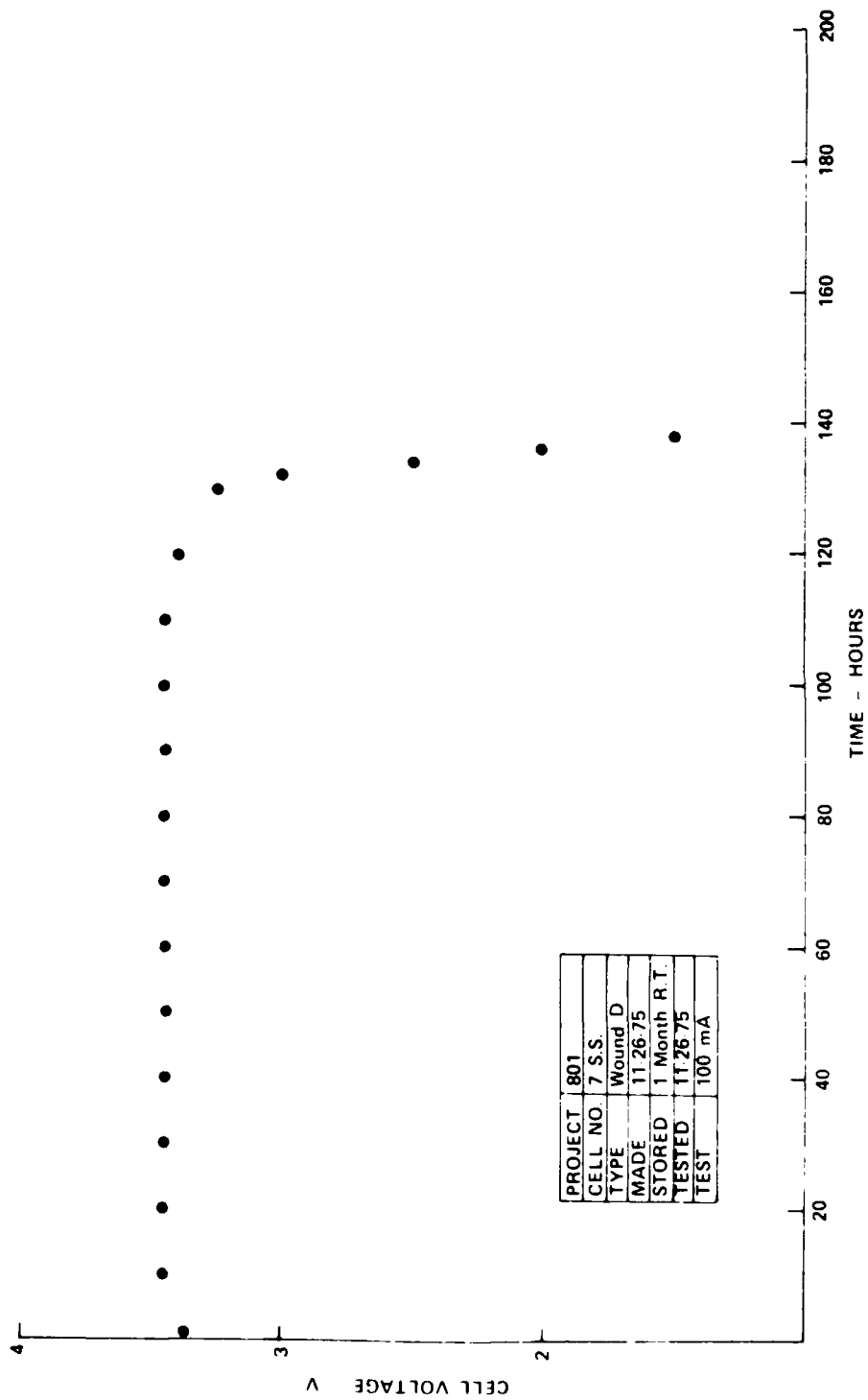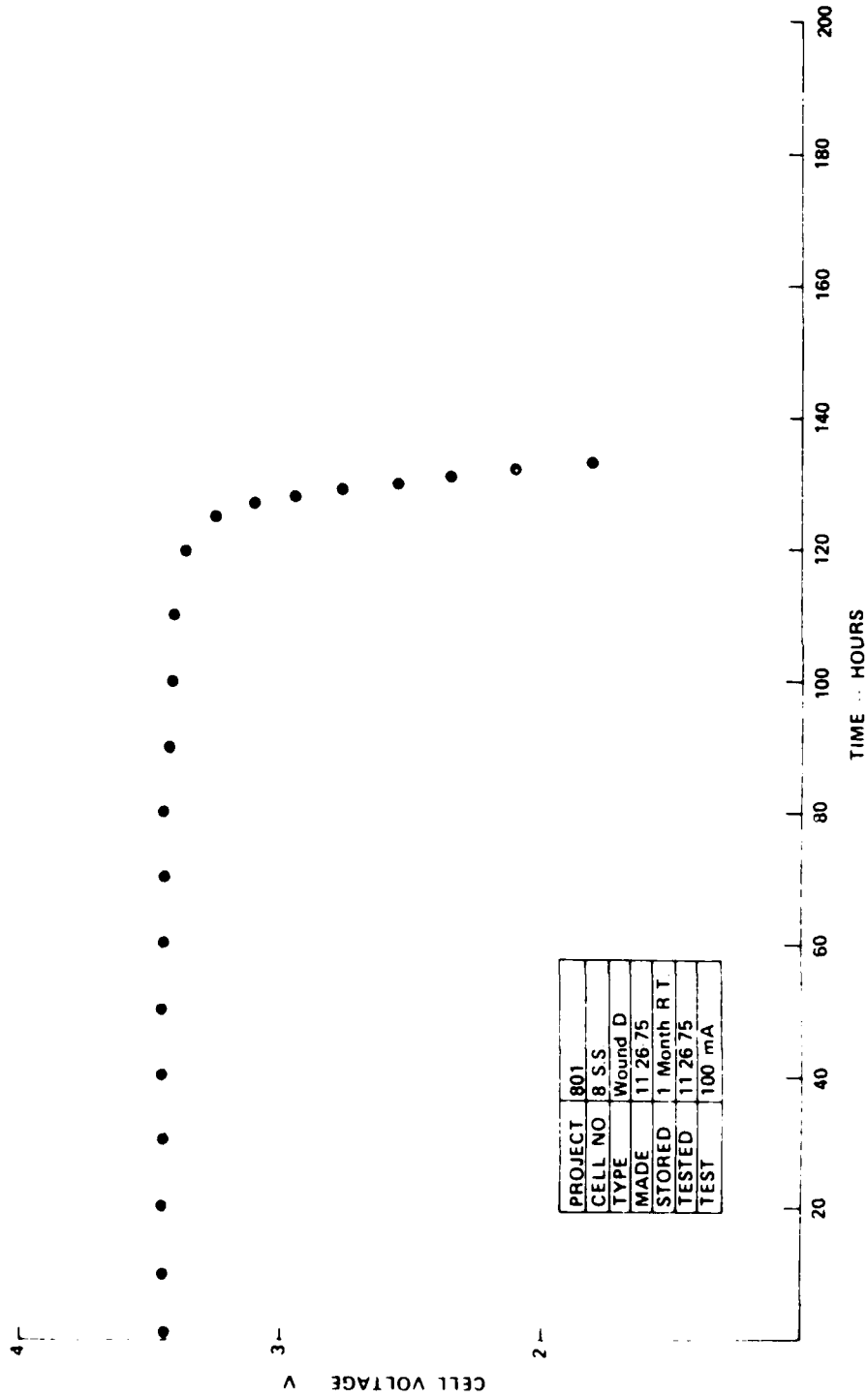|	
PROJECT	801
CELL NO	8 S.S.
TYPE	Wound D
MADE	11.26.75
STORED	1 Month R T
TESTED	11.26.75
TEST	100 mA

TIME — HOURS

CELL VOLTAGE — V

Figure 10. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

Figure 11. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures
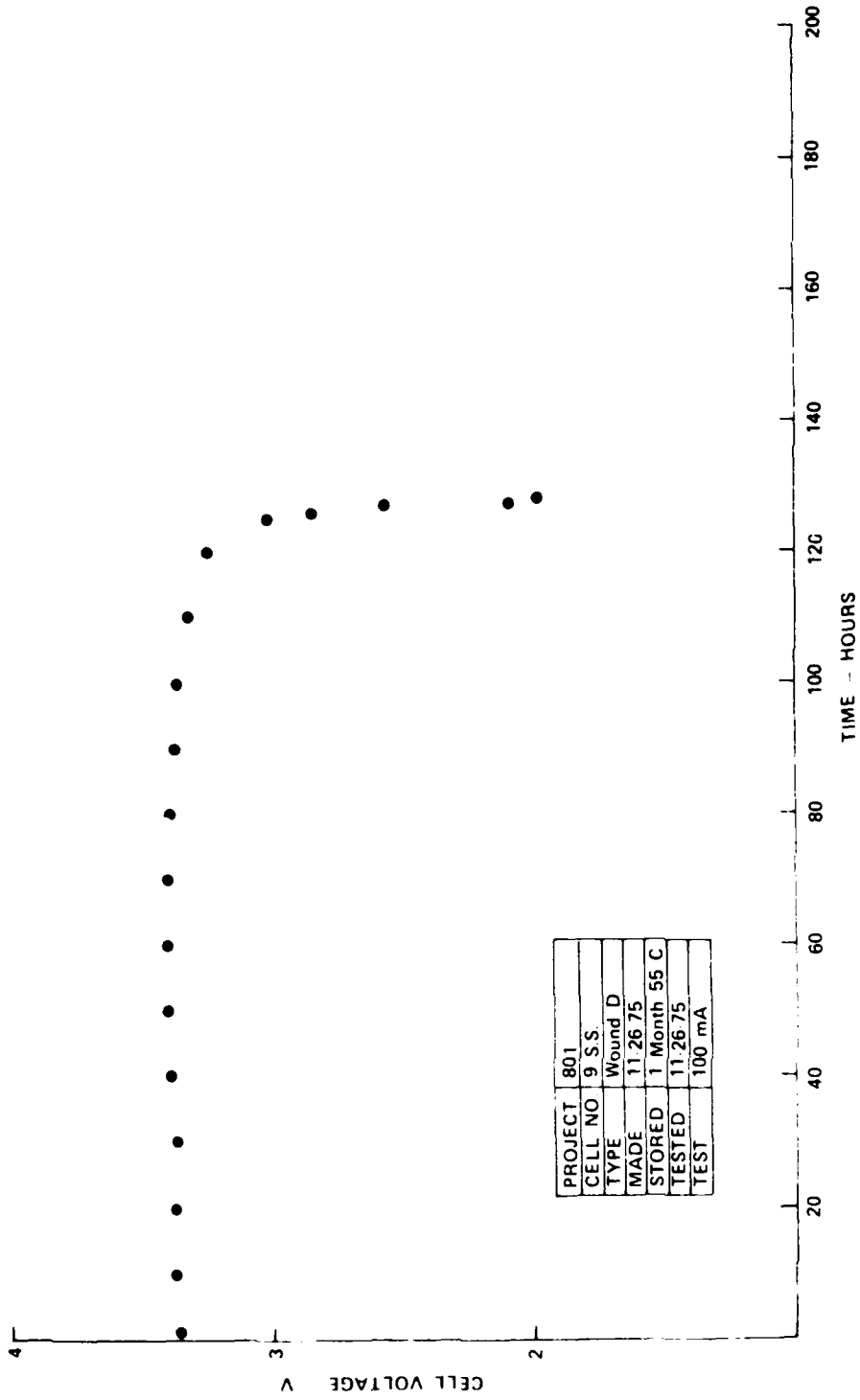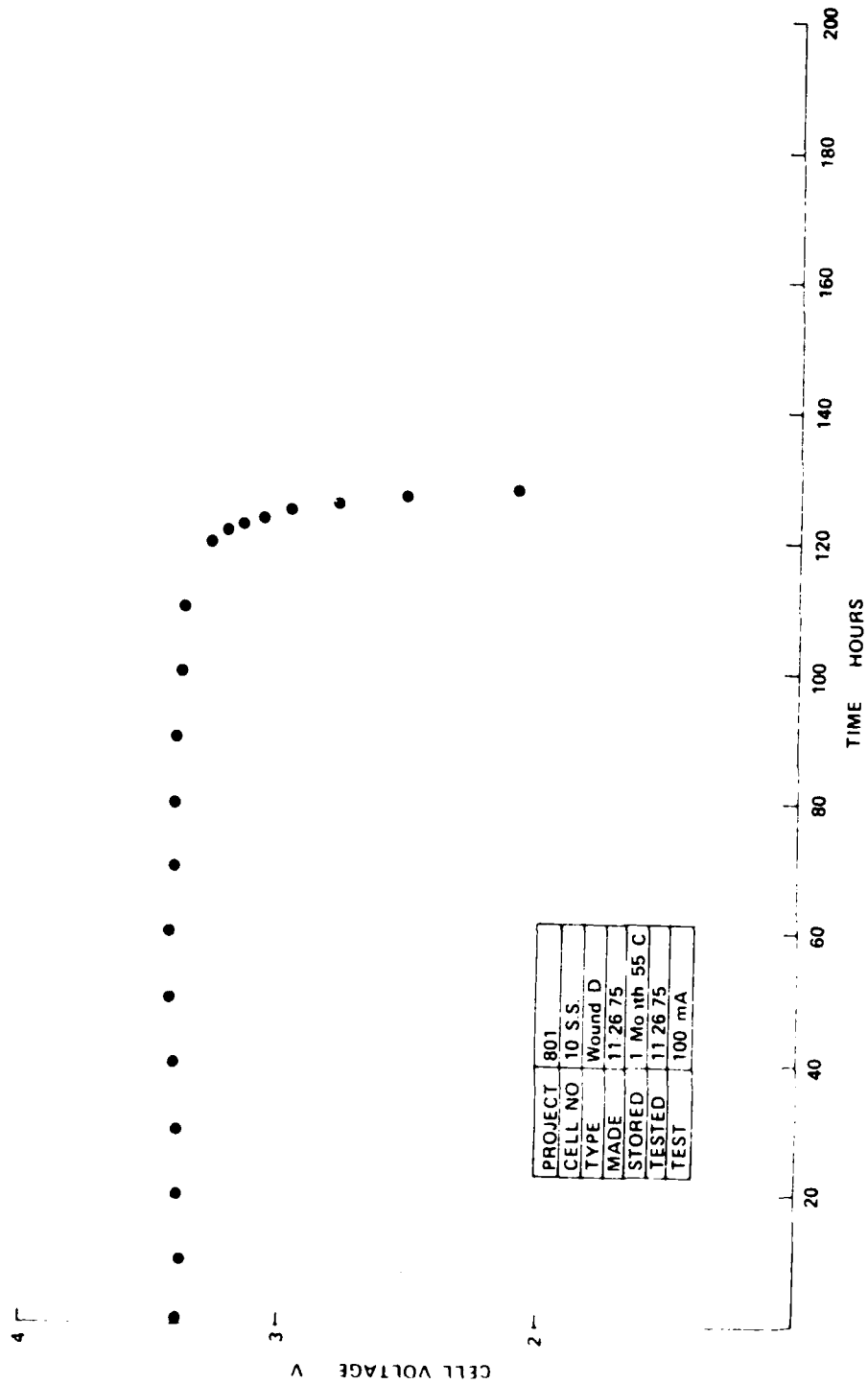
PROJECT	801
CELL NO	9 S.S.
TYPE	Wound D
MADE	11.26.75
STORED	1 Month 55 C
TESTED	11.26.75
TEST	100 mA

CELL VOLTAGE V

TIME — HOURS

TIME HOURS

CELL VOLTAGE V

200 180 160 140 120 100 80 60 40 20

4 3 2

PROJECT	801
CELL NO	10 S.S.
TYPE	Wound D
MADE	11 26 75
STORED	1 Mo th 55 C
TESTED	11 26 75
TEST	100 mA

Figure 12. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

Figure 13. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures
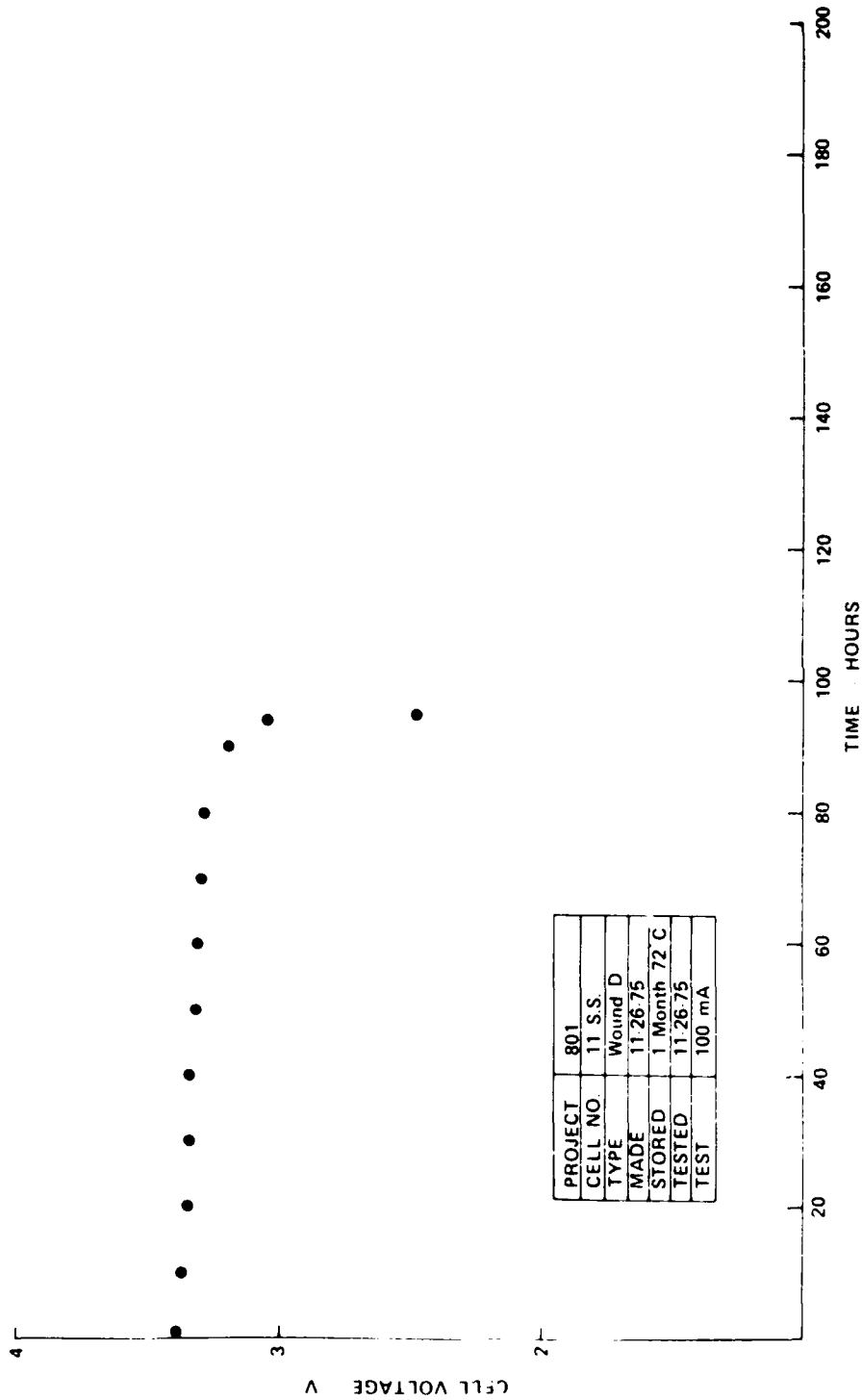
PROJECT	801
CELL NO.	11 S.S.
TYPE	Wound D
MADE	11-26-75
STORED	1 Month 72 C
TESTED	11-26-75
TEST	100 mA

CELL VOLTAGE V

TIME – HOURS

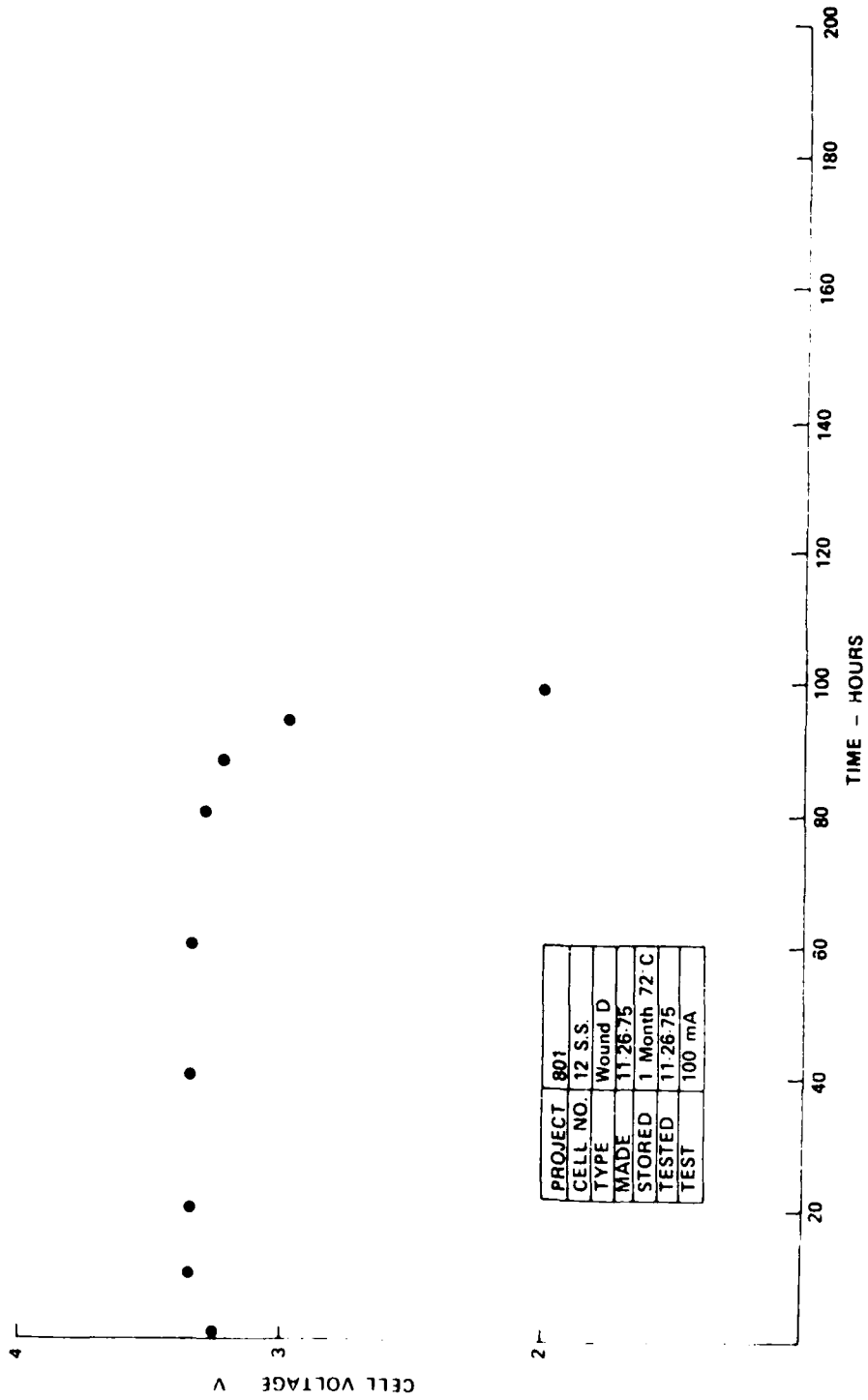| PROJECT	801
CELL NO.	12 S.S.
TYPE	Wound D
MADE	11-26-75
STORED	1 Month 72-C
TESTED	11-26-75
TEST	100 mA

Figure 14. Discharge Curves for Standard Size D Cells after 1 Month Storage at Various Temperatures

5. THREE MONTH STORAGE DATA WITH COLD ROLLED STEEL HARDWARE

Five wound D cells were constructed and stored at room temperature for three months. These cells were not built with the aid of the computer calcuation and, therefore, capacity was not optimized. A capacity of 9 to 10 Ah was expected according to the previous experience with this type of cell after storage.

5.1 DELAY AND CAPACITY

The delay tests were carried out at a current of 300 mA using a high speed strip chart recorder. Of the five cells only one showed a slight delay. All cells retained their full capacity. The results are plotted in Figures 15, 16 and 17 and the capacity listed in Table 6.

TABLE 6

CAPACITY AFTER STORAGE FOR THREE MONTHS

AT ROOM TEMPERATURE USING COLD ROLLED STEEL CANS

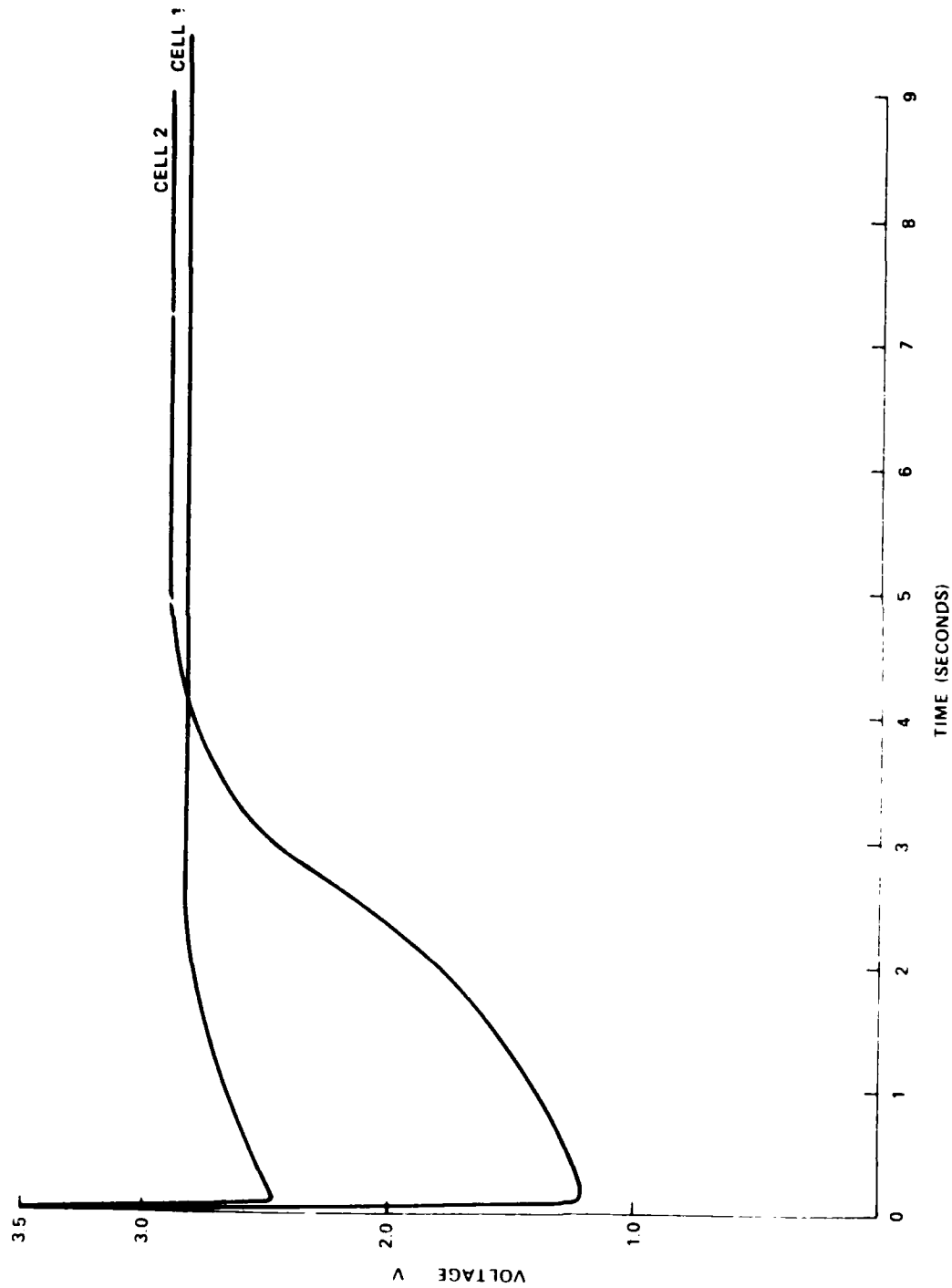| CELL NUMBER	CAPACITY (Ah)
1	8.6
2	9.2
3	9.5
4	9.1
5	9.2

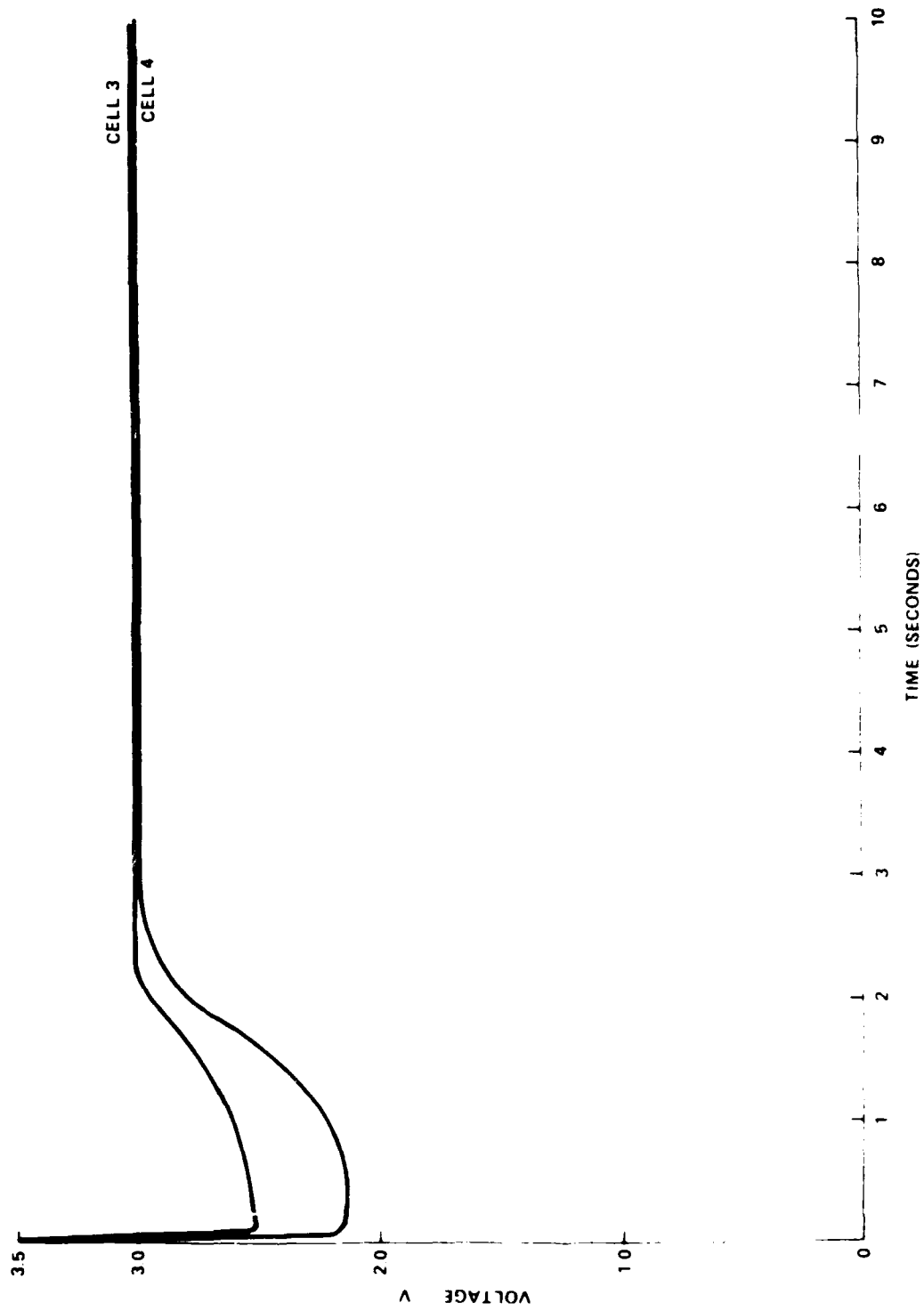Figure 15. Voltage Delay after Storage for 3 Months at Room Temperature

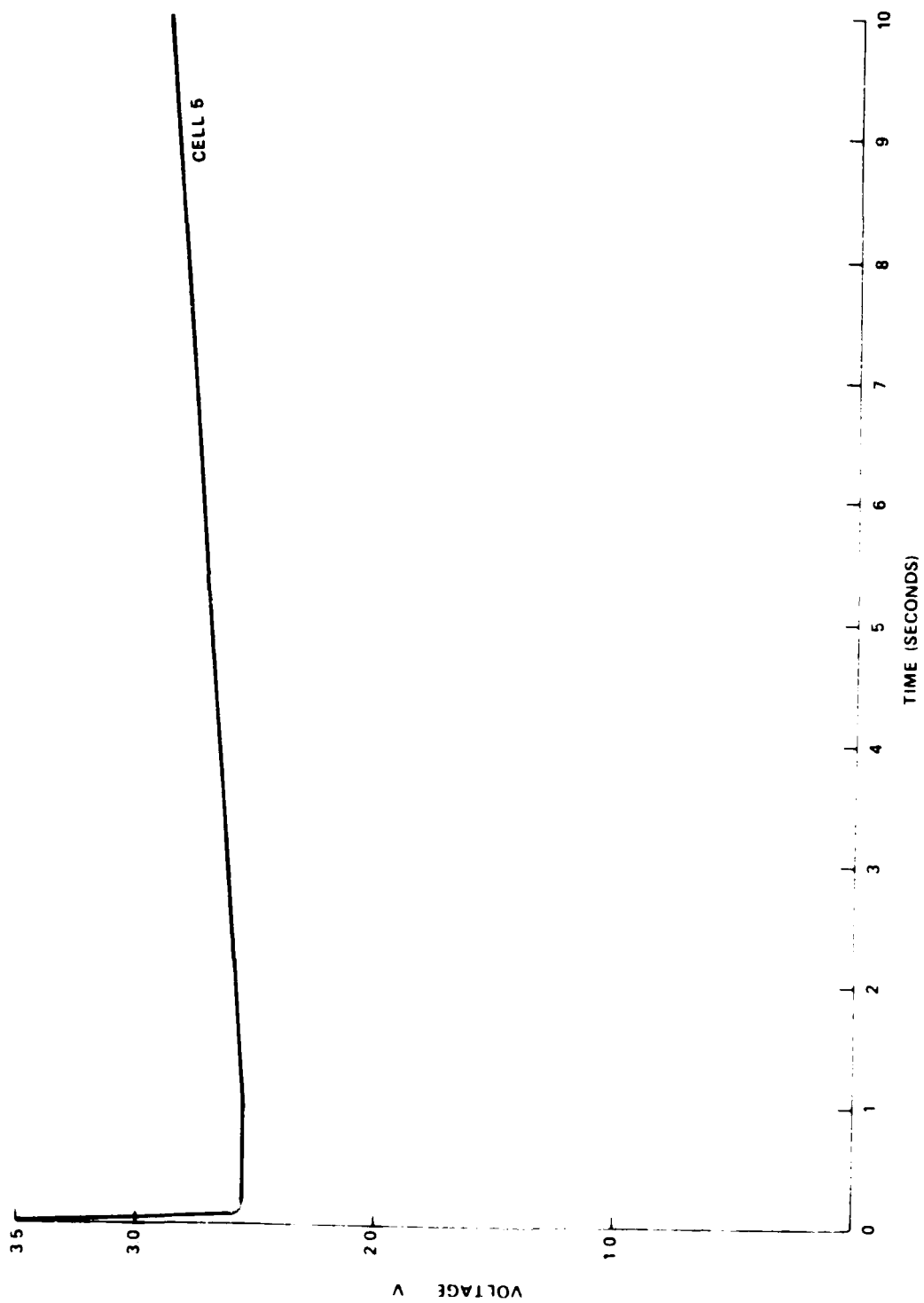Figure 16. Voltage Delay after Storage for 3 Months at Room Temperature

Figure 17. Voltage Delay after Storage for 3 Months at Room Temperature

5.2   CONCLUSIONS

It is obvious from this test that three months storage at room temperature has no ill effects on the voltage delay or the capacity of the cell. The delay is not much different from that of a fresh cell and the entire capacity seems to remain available for discharge. This has generally been the result of most of the tests run at room temperature, including the early tests covering one month storage.

6. NEXT QUARTER PROGRAM

The studies of the corrosion of stainless steel hardware will be continued using both the glass cell set ups as well as the proper cell hardware. In addition, attention will be focused on the possible loss of lithium due to the galvanic corrosion involving the reduction of $SOCl_2$ on the surface of hardware. Completed D size cells will be used and the shelf life of the cell will be tested along with the hermetic cell closure now in progress.

7. REFERENCES

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