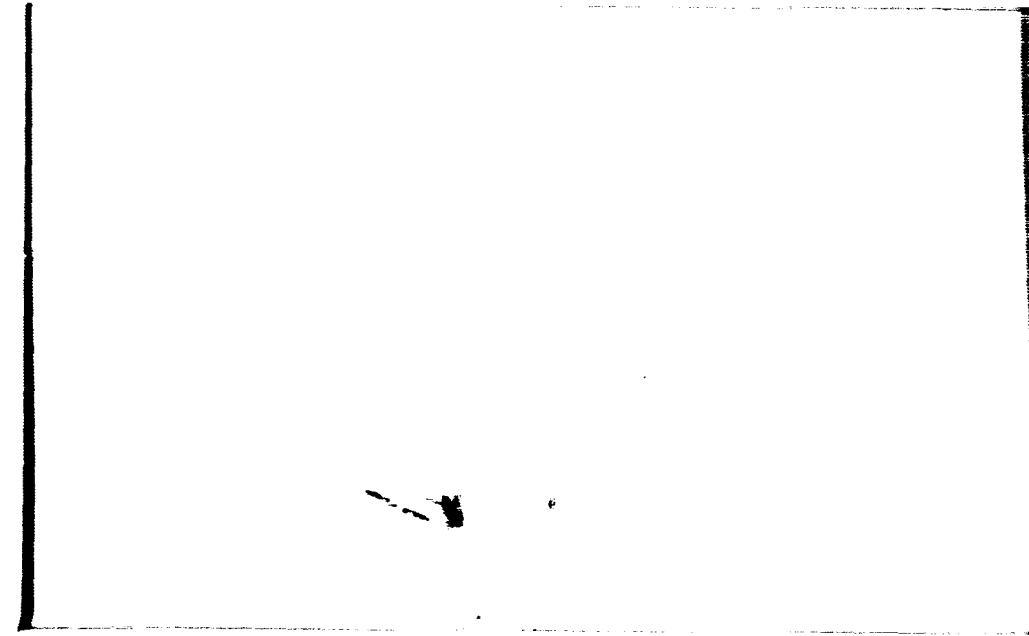


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HYDROGEN-OXYGEN ELECTROLYTIC REGENERATIVE FUEL CELLS

Prepared for  
National Aeronautics and Space Administration  
Lewis Research Center  
21000 Brookpark Road  
Cleveland, Ohio  
Attention: D. G. Soltis

Contract NAS3-2781

EOS Report 4110-ML-27

10 August 1966

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## 1. INTRODUCTION

This report reviews the progress made on the development of a hydrogen-oxygen regenerative fuel cell (secondary battery) under NASA Contract NAS3-2781 during the period of 1 July through 1 August 1966. During this period primary emphasis was placed on the testing of single cells with various electrode matrix structures in order to improve cycle life performance. Single cells tested employed variations of potassium titanate matrices to optimize fabrication techniques and to obtain performance parameters and an understanding of modes of deterioration that exist within cycling cells. The titanate matrices have continually demonstrated superior performance over what has previously been obtained, and the limit of their capability has not yet been determined.

*Author*

## 2. TECHNICAL DISCUSSION

### 2.1 Single-Cell Tests

Eleven single-cell tests were conducted during this report period to evaluate various electrode and potassium titanate matrix structures. Table I is a summary of the test results showing various construction variables. During the preceding report period, testing was started on cell 198. The oxygen electrode of the cell was a gold-plated nickel screen platinized by electrodeposition of platinum ( $25 \text{ mg/cm}^2$ ) on the surface of the screen. The hydrogen electrode was a chemically platinized porous nickel plaque. The matrix was potassium titanate (90 percent by weight) and asbestos (10 percent).

As shown in Fig. 1, the cell was cycled continuously for a period of 952 cycles. During the cycling, there was a gradual increase in the charge voltage and a decrease in discharge voltage. After 952 cycles a short circuit developed within the cell, causing the cell voltage to drop to 0 during charge and discharge. The test was discontinued at this failure.

The load bank used to test the cells was a fixed resistor. Therefore, as the cell voltage during discharge degraded, the discharge current also decreased. In the early cycling phases discharge current ranged from 15 to 18 amperes, but as the voltage began to gradually drop during the latter cycling sequences, the current degraded steadily. At the start of the 952nd discharge cycle the current measured 15 amperes; it dropped during discharge to 10 amperes at the end of the cycle.

TABLE I

## SUMMARY OF SINGLE-CELL TESTS

| Cell No. | <u>O<sub>2</sub> Electrode</u> |                          | <u>H<sub>2</sub> Electrode</u> |                          | Mat         |         | Electrolyte |          | Comments                                | Results                                              |
|----------|--------------------------------|--------------------------|--------------------------------|--------------------------|-------------|---------|-------------|----------|-----------------------------------------|------------------------------------------------------|
|          | No.                            | Catalyst                 | No.                            | Plat.                    | Thick In.   | Dry Wt. | o/o KOH     | Wt. (gm) |                                         |                                                      |
| 198      | Plat. Gold Screen              | 25 mg Pt/cm <sup>2</sup> | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 19.6    | 40.0        | 34.0     |                                         | 952 cycles, gradual degradation                      |
| 199      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 24.5    | 40.15       | 34.0     | O <sub>2</sub> electrodes from cell 196 | Test stopped at 405 cycles, due to poor performance. |
| 202      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 20.09   | 40.0        | 34.0     | O <sub>2</sub> electrodes from cell 195 | 387 cycles, cell developed internal short.           |
| 205      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 25.0    | 40.15       | 34.09    |                                         | 52 cycles, developed slow recombination.             |
| 206      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 20      | 40.0        | 34.0     |                                         | Still on test.                                       |
| 207      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | 90 KT 10 AB | 20      | 40.0        | 34.0     | O <sub>2</sub> electrode from cell 202  | 25 cycles, developed slow recombination.             |
| 208      | "                              | "                        | "                              | "                        | "           | 24.5    | 40.0        | 34.0     | Electrode from 6-cell 110               | 21 cycles. Good performance.                         |
| 209      | Plat. Nickel Plaque            | 20 mg Pt/cm <sup>2</sup> | "                              | "                        | "           | 22.0    | 40.0        | 34.0     | H <sub>2</sub> Concentration cell       | Still on test, gradual increase in voltage.          |
| 210      | Am. Cy.                        | 9 mg Pt/cm <sup>2</sup>  | "                              | "                        | "           | 21.5    | 40.0        | 34.0     | Electrodes from 6-cell 110              | Ran poorly.                                          |

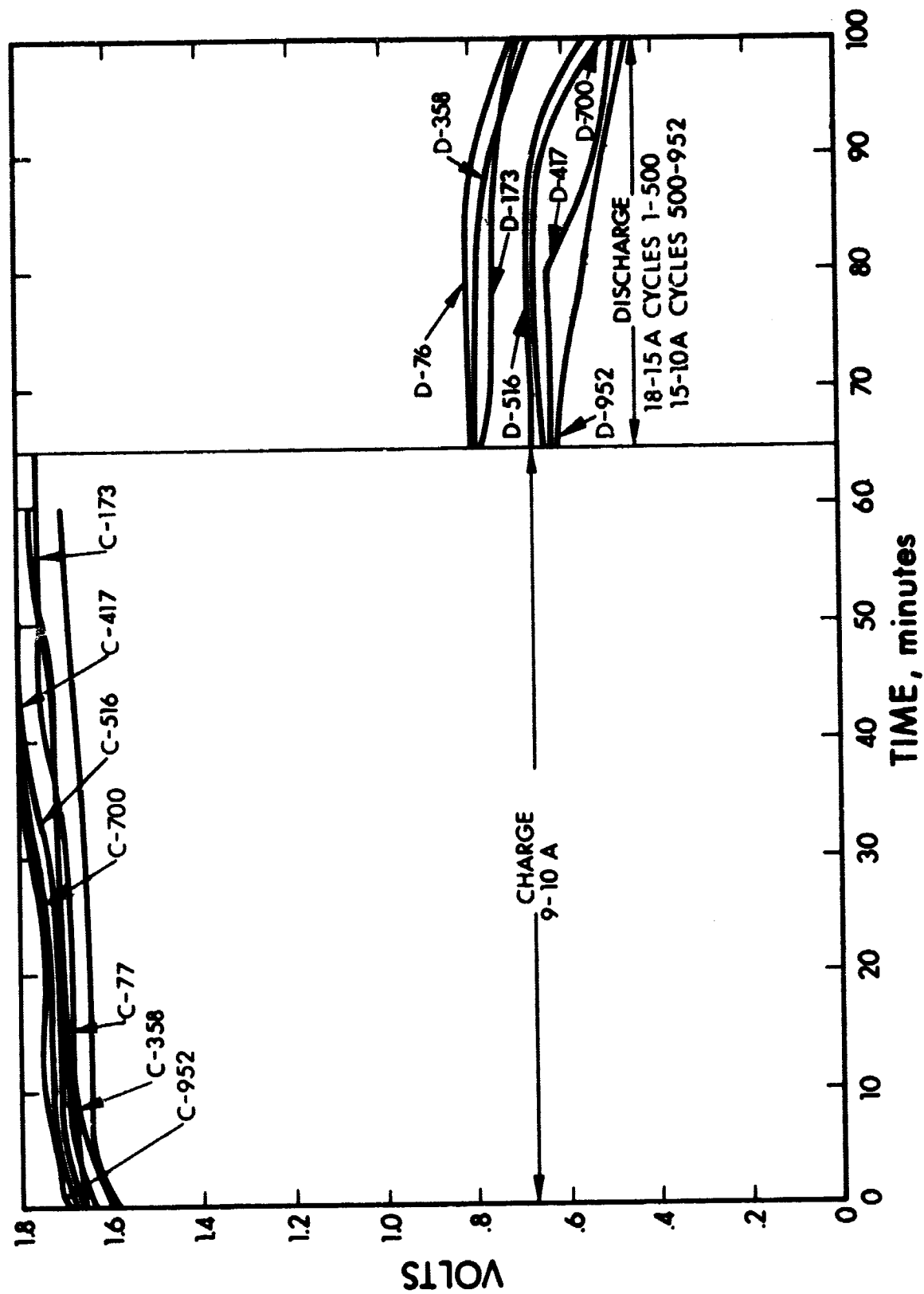


FIG. 1 CYCLING PERFORMANCE OF CELL 198

Since the ampere-hours used during the discharge cycle decreased with voltage and current falloff, the recharging time also reduced. During the charge periods of the latter cycles the pressure switch would cut off when the pressure reached 350 psi, and the cell would experience a no-charge/no-discharge open-circuit condition for 5 to 10 minutes before the next discharge cycle began. This condition occurred in order to maintain a uniform cycling period of 65 minutes for charging and 35 minutes for discharging. Even with the considerations of degradation and reduced performance observed, this cell test represents the best performance level achieved to date. The 952 cycles at 100 min/cycle are equivalent to 1585 hours or a period of 66 days - more than two months of continuous operation.

In examination of a disassembled cell, it was not possible to locate the area where the internal short had developed. The electrodes were stuck to the matrix, and it was necessary to rip part of the electrodes and the matrix in order to separate the cell components. There was black discoloration on the matrix adjacent to each of the electrodes which appeared to be adhered electrode material that had been ripped away when the electrodes were removed. The internal portions of the matrix were slightly gray in color, but showed no substantial difference from mats that had been examined after lesser cycling periods. Analysis of the mat for electrolyte concentration and platinum has not yet been conducted, but will be covered during the next period.

Cell 199, also a cell that was put on test during the last period, consisted of an American Cyanamid 9 mg platinum/cm<sup>2</sup> oxygen electrode and a platinized porous nickel plaque hydrogen electrode. The cell was tested continuously for 405 cycles after which the test was discontinued. Figure 2 shows the performance of this cell. As shown, there was a gradual degradation in performance with cycling. The test was discontinued to examine the internal components, since it appeared no more useful information could be obtained from this cell.

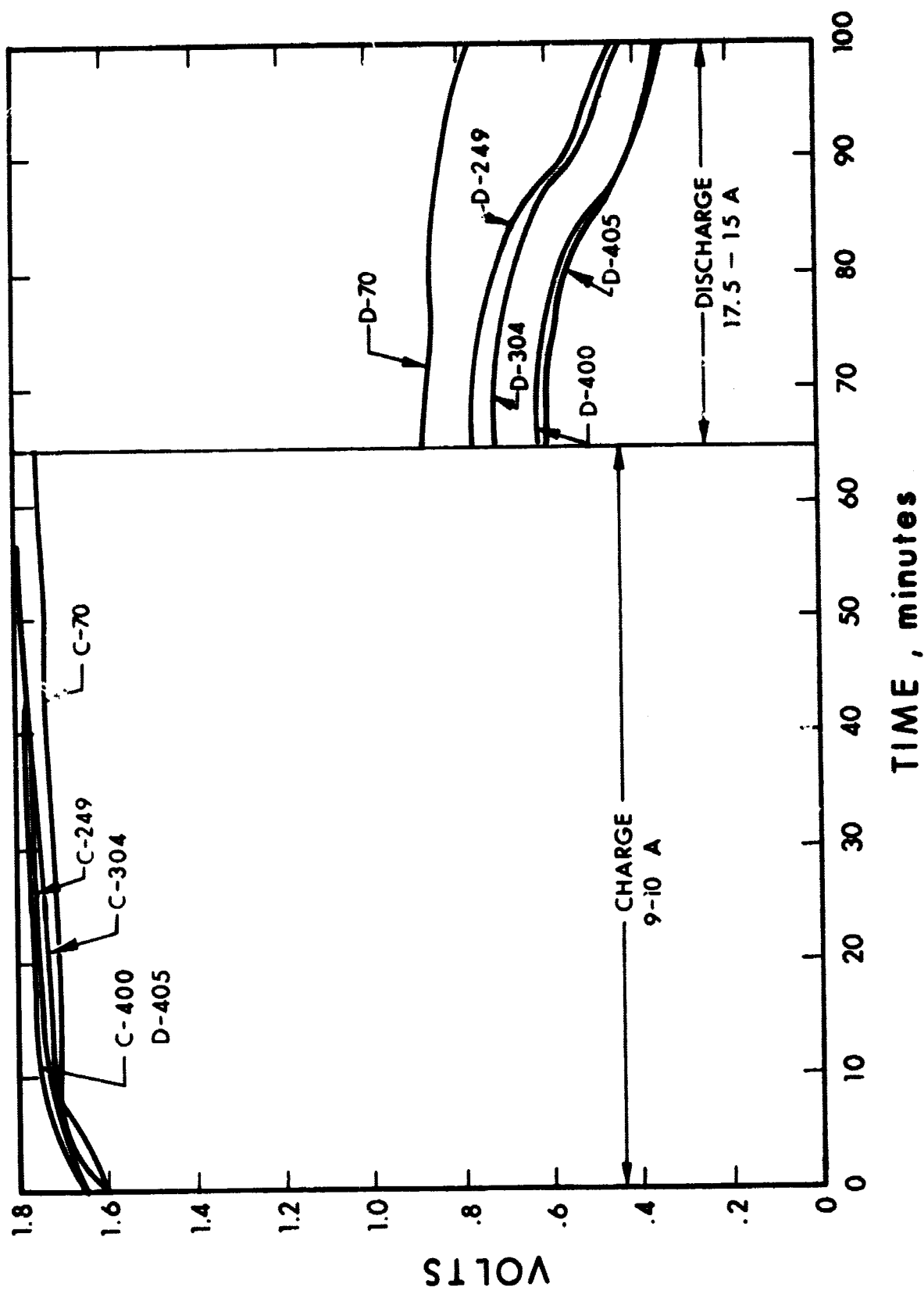


FIG. 2 CYCLING PERFORMANCE OF CELL 199

Cell 202 consisted of an American Cyanamid 9 mg platinum/cm<sup>2</sup> oxygen electrode previously used in cell 195, and a new porous nickel plaque platinized hydrogen electrode on mat. The cell was subjected to the standard test cycle and cycled continuously for 387 cycles at which time an internal short developed within the cell and the test was discontinued. The cycling performance is shown in Fig. 3. The cell was disassembled and the oxygen electrode was recovered in good condition and reassembled with a new hydrogen electrode and matrix and designated cell 207. This cell showed initial good performance, but developed a slow gas recombination on the 27th cycle and the charge pressure did not rise above 150 psi. The test was then discontinued. The performance of cell 207 is shown in Fig. 4. The oxygen electrode of this cell has also previously been employed in cell 195, results of which were described in the eighth quarterly report. That cell had been subjected to 278 cycles, the results of which are shown in Fig. 5. Therefore, the total accumulated number of cycles subjected to the oxygen electrode was 692 cycles. In each case when the cell was reassembled with a new matrix and hydrogen electrode, the initial performance returned to the original level of approximately 0.8 to 0.85 volt on discharge at 17 to 18 amperes. The results of this series of tests seemed to indicate that the cause in mode of deterioration encountered with the titanate matrix type cells, now under study, is centered in the matrix and/or hydrogen electrode and not the oxygen electrode, as previously concluded in asbestos type cells.

Cell 205 consisted of American Cyanamid 9 mg platinum/cm<sup>2</sup> oxygen electrode and a platinized nickel plaque hydrogen electrode with a 90 percent potassium titanate, 10 percent asbestos matrix. The cell was cycled 52 times and showed good initial performance, but at the 53rd cycle, it exhibited a slow recombination, and was unable to recharge the cell above 200 psi; the test was discontinued.

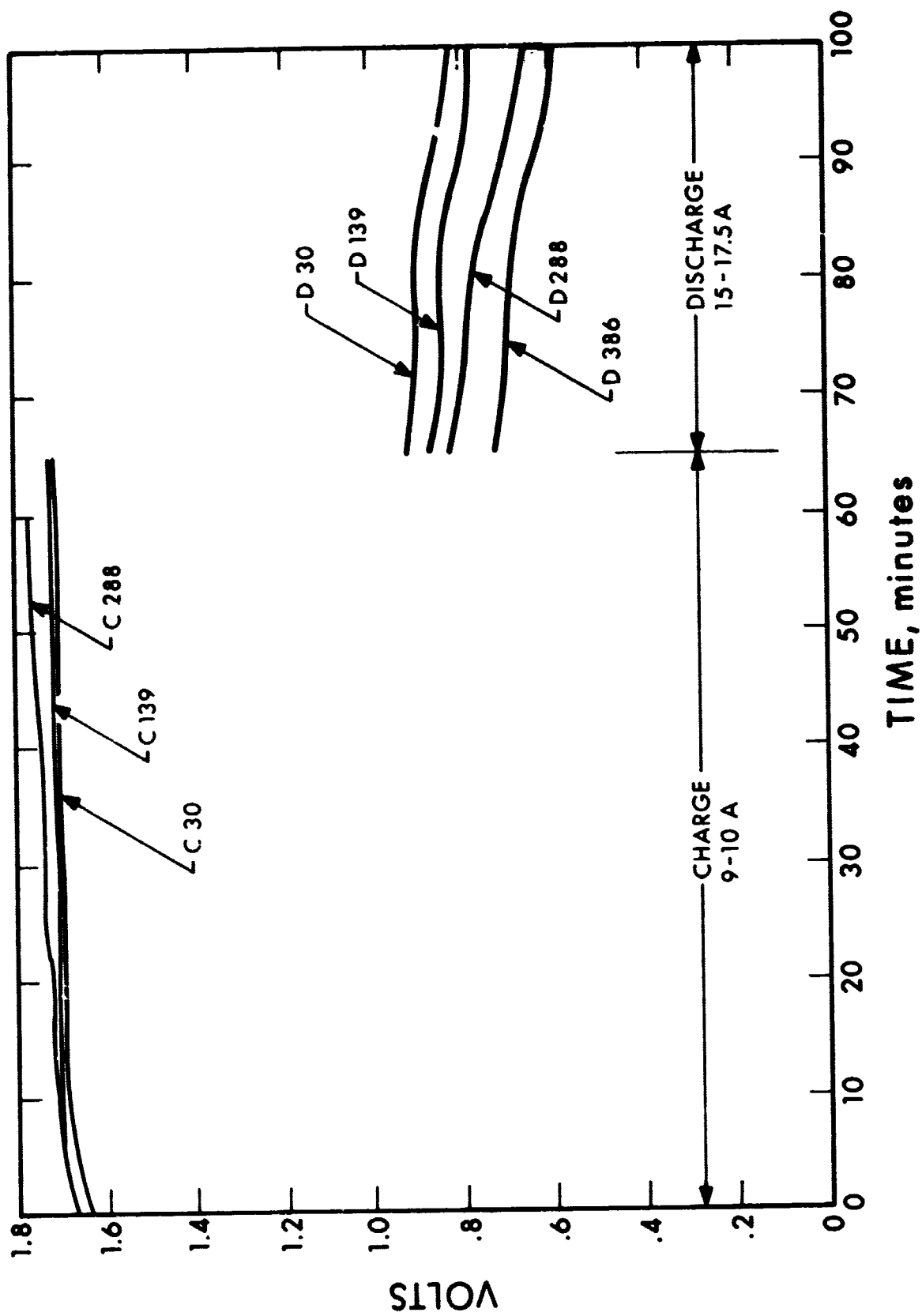


FIG. 3 CYCLING PERFORMANCE OF CELL 202

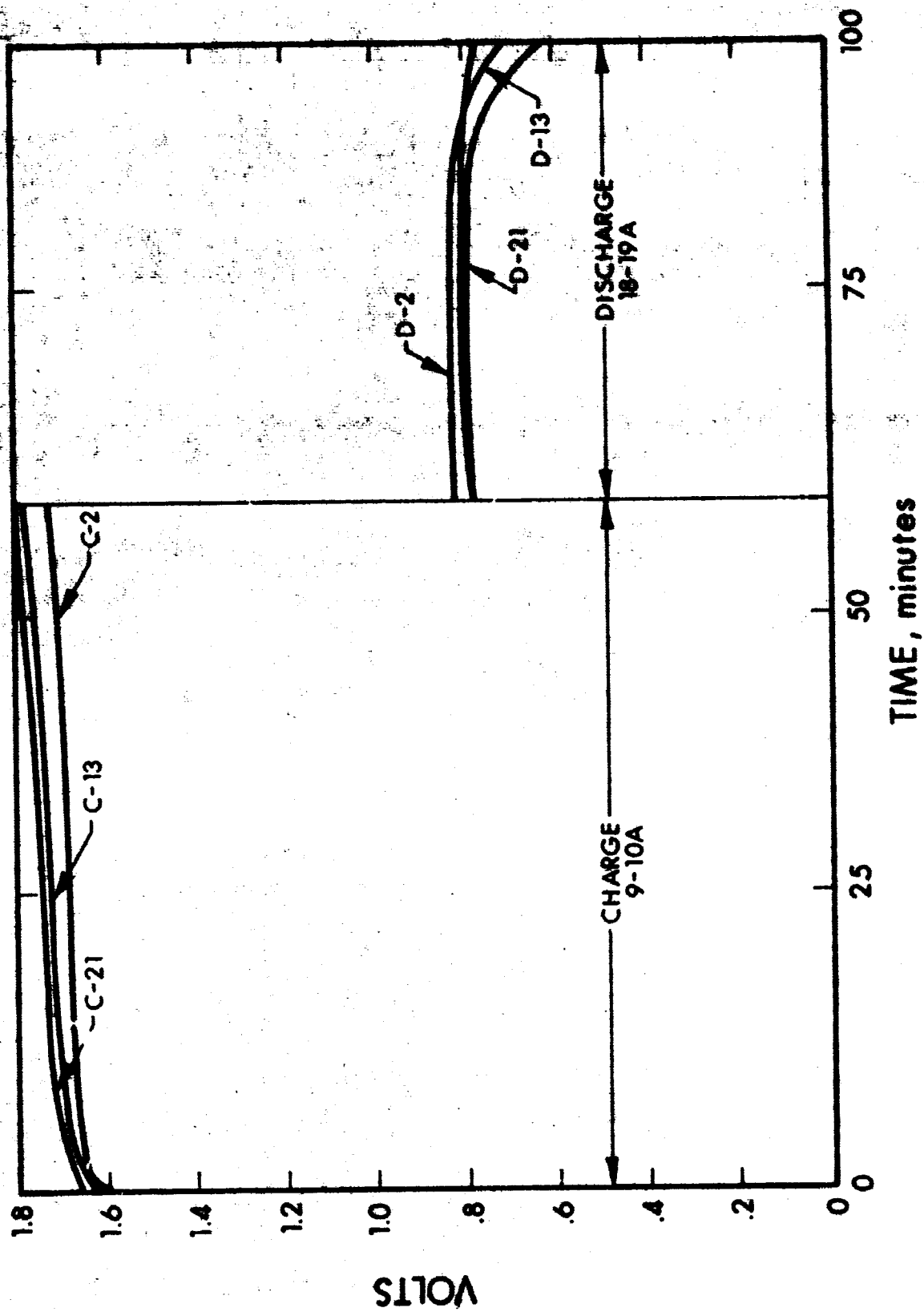


FIG. 4 CYCLING PERFORMANCE OF CELL 207

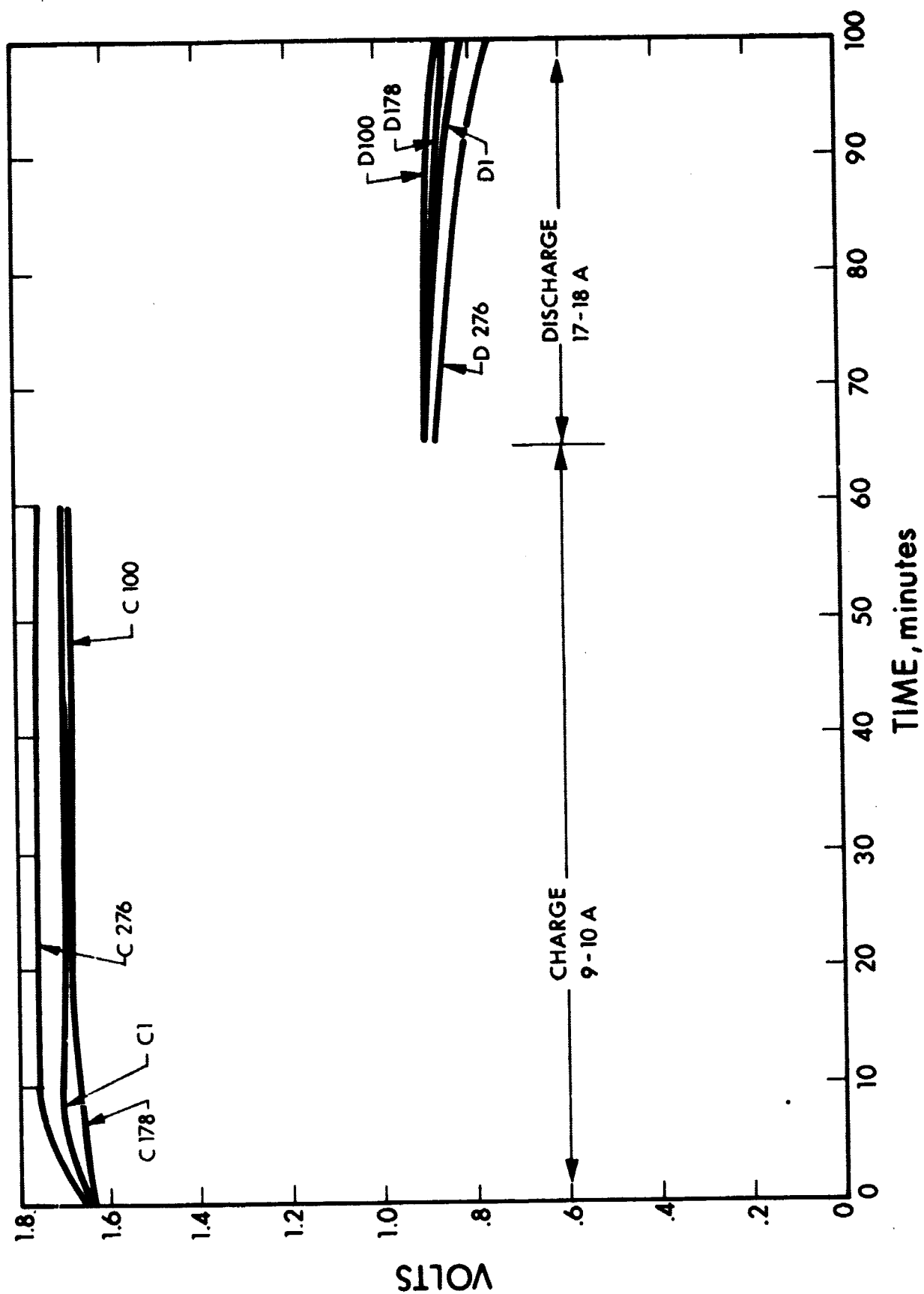


FIG. 5 CYCLING PERFORMANCE OF CELL 195

Cell 206 was of construction similar to cell 205. It is still under test. It has cycled continuously for 400 times at this point and shows a slight gradual degradation in performance with cycling. Performance of this cell is shown in Fig. 6.

Single cells 208 and 210 contain electrodes that have been previously used in 6-cell units 109 and 110 (described in the eighth quarterly report). Both cells contained matrices of the type used in the 6-cell unit (90 percent potassium titanate, and 10 percent asbestos).

Cell 208 was cycled for 21 times and showed initial good performance. At that time the test was discontinued.

Cell 210, containing a different set of electrodes, exhibited poor performance by discharging at 0.6 to 0.7 volt per cell, charging approximately 1.8 volts per cell. This poor performance of cell 210 confirmed the previous observation on 6-cell testing that the electrodes had somehow been poisoned in the initial testing of 6-cell unit 109, and that was the cause for the poor performance of 6-cell unit 110. Apparently, the only possible poison within the unit as previously observed was the corrosion products of the bipolar plates due to the imperfections in plating of the plates utilized in 6-cell unit 109.

Based on some of the previous results indicating that modes of degradation now encountered are caused by the matrix and/or hydrogen electrode, a new hydrogen concentration cell test was set up and designated cell 209. In this case, two platinized porous nickel plaque electrodes were employed. The matrix consisted of 90 percent potassium titanate, 10 percent asbestos by weight. The cell was run in a continuous concentration mode at 18 amperes (equivalent to  $100 \text{ mA/cm}^2$ ). Figure 7 shows the voltage performance of this cell as a function of time. The cell has been run for a period in excess of 500 hours to this point and is still under test. As can be seen, there has been a gradual increase in voltage with time. It seems to have increased

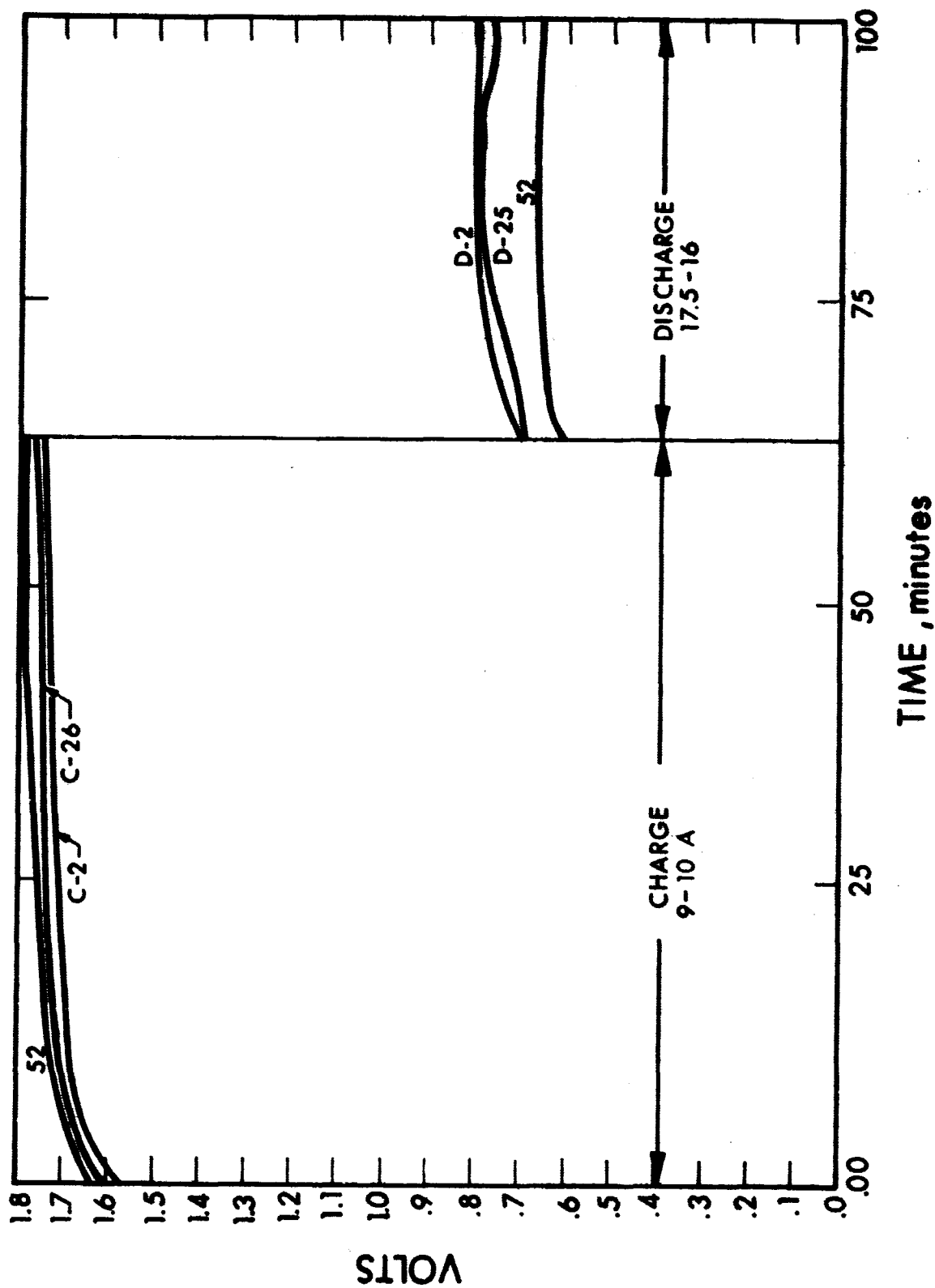


FIG. 6 CYCLING PERFORMANCE OF CELL 205

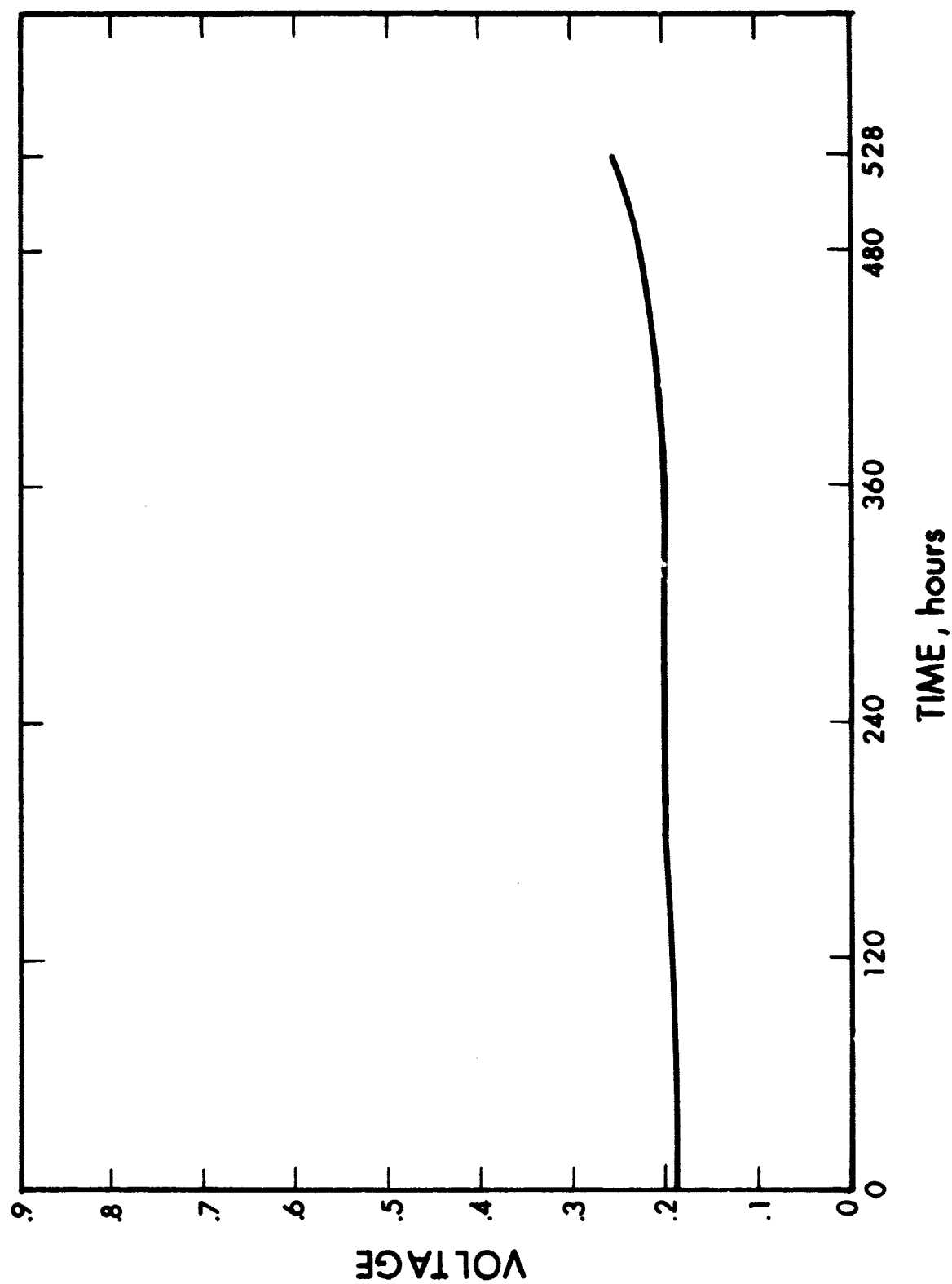


FIG. 7 H<sub>2</sub> CONCENTRATION CELL

in rate over the last 100 hours, which indicates some deterioration is taking place. The test will be continued to determine the extent and rate of deterioration that will be encountered.

## 2.2 Multicell Testing

Based on the satisfactory performance in single cells, of the American Cyanamid oxygen electrode, 90-10 potassium titanate matrix and platinized nickel plaque hydrogen electrode configuration, a new 6-cell unit was built during this period to be subjected to test. However, during the checkout of the cell stack, the unit is subjected to slight differential pressure to determine if any cross-leakage exists. In this checkout, it was found that there was an appreciable gas cross-leakage through the matrices which would have resulted in a slow recombination within the stack; the unit was therefore disassembled. An examination of the matrices revealed no obvious area where the cross-leakage was occurring, except that many of the matrices were found to be thinner in the center than on the edges. A review of the process technique of the matrices revealed that a slightly convex Bunkner funnel was utilized in the filtrate formation of the matrices which would result in less material being in the center of the mat. To alleviate this problem, a new funnel that has a flat inner plate will be utilized to fabricate future mats. A new series of mats will be fabricated and the 6-cell unit reassembled, checked out, and subjected to test in the next period.

## 2.3 500-Watt 34-Cell Unit

In preparation for the ultimate delivery of a 34-cell 500-watt unit, a new set of tankage was ordered. The new tanks which contained a smaller flange than the previous tanks to reduce weight were fabricated of aluminum and nickel-plated to protect the aluminum surface. These tanks were delivered during this period.

### 3. PLANS FOR NEXT PERIOD

Single cell tests will be continued to determine the component that is causing the deterioration in the potassium titanate cell configuration. Matrices will be evaluated containing mixes of potassium titanate and Teflon or polypropylene as substitute for asbestos fibers. The hydrogen concentration cell tests will be continued and repeated if necessary to determine what mode of deterioration occurs during that test condition. A 6-cell unit employing American Cyanamid oxygen electrode 90-10 titanate asbestos mats and platinized nickel electrodes will be assembled and subjected to test. Toward the end of this period a single cell configuration will be frozen and the fabrication of components for the assembly of a 34-cell prototype unit will be initiated.

4. FINANCIAL STATEMENT

Man-hours and dollar expenditure for the period June 24, 1966 through July 24, 1966 were as follows:

|                          |            |
|--------------------------|------------|
| Direct Labor Hours       | 457        |
| Direct Labor Dollars     | \$1,959.00 |
| Purchase and Commitments | \$2,632.00 |
| Total Dollar Expenditure | \$8,390.00 |