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HYDROGEN-OXYGEN ELECTROLYTIC REGENERATIVE FUEL CELLS

by

M. Klein and E. Findl

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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QUARTERLY REPORT

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27 APRIL 1966

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Contract NAS 3-2781

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1. INTRODUCTION

This report reviews the progress made on the development of a regenerative hydrogen-oxygen fuel cell under NASA Contract 3-2781 during the period of 1 January 1966 through 31 March 1966. The program objective is the development of an electrolytically regenerative, hydrogen-oxygen fuel cell that will be superior in performance to currently available rechargeable batteries. The device under development consists of a cell stack that is utilized as an electrolyzer during charge periods and as a fuel cell during discharge periods. Integral gas storage tanks are used to contain hydrogen and oxygen gas that is generated during charge. Such a device offers advantages in the area of watt-hours-per-pound, high ambient temperature operation, and greater cycle life than that which can be obtained from existing secondary batteries. Phase I consisted of a design development and testing of a nominal 75-watt, 44-watt-hour, 6-cell unit to demonstrate the feasibility of a multicell regenerative device. This phase has been completed. Phase II (as modified) consists of the investigation of the asbestos matrix, electrode combinations to improve cycle life, and the design and development of a 500-watt, 600-watt-hour, 34-cell unit of minimum weight for evaluation as a flight prototype.

2. SUMMARY

During this period primary emphasis was placed on the testing of single cells with various electrode structures in order to improve cycle life capabilities and to obtain better understanding of modes of deterioration of cell performance. The exact primary cause of the performance deterioration with cycling has not as yet been determined, but it appears contaminants in the asbestos may be causing the difficulty. A new test method was evolved utilizing an oxygen concentration cell for evaluation of electrode performance, which offers the promise of reducing the variables in a cell test and enabling the pinpointing of the exact cause of deterioration. Asbestos tests were continued to obtain a better understanding of asbestos structure and variations.

3. TECHNICAL DISCUSSION

3.1 Single Cell Test

Fifty single cell tests were conducted during this period to evaluate the performance of various electrode structures. Test results and construction variables of these tests are summarized in Table 1. Cell No. 120 contained American Cyanamid electrodes that had been previously used in cells 124, 122 and 119, the results of which are described in the Sixth Quarterly Report. These electrodes were washed prior to use and the cell was assembled with a new asbestos mat. The cell was cycled 25 times and exhibited poor performance, discharging at approximately 0.6 of a volt and charging at 1.7 to 1.9 volts.

Cell No. 126 consisted of electrodes fabricated by gold coating and platinizing porous nickel plaques and an asbestos mat that had been acid washed. The acid washed asbestos mat was prepared by shredding two standard six-inch mats in a beaker to which is added 200 CC's of concentrated hydrochloric acid. Initial mixing of the acid with the asbestos results in considerable gassing due to the reaction of the acid with certain constituents of the mat. After one hour, the asbestos fibers are repeatedly mixed with distilled water and then poured into a Buchner funnel in which the free liquid is vacuum filtered from the fibers. This process is repeated until the filtrate water reaches a pH of 7. For the final mat formation procedure, the asbestos fibers are mixed with distilled water to form a slurry and are poured into the Buchner funnel under vacuum to form an even layer of asbestos. The mat is then dried and cut to a 6-inch diameter. Initial performance of cell 126 with the acid washed mat was very poor, discharging at approximately 0.5 to 0.6 of a volt and charging at 1.7 to 1.8 volts. Therefore, the test was discontinued. An examination of the cell components showed a reddish discoloration of the asbestos mat adjacent to the hydrogen electrode. It was concluded that all the acid had not been completely washed from the asbestos mat.

TABLE 1

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick (In.)	Mat Dry Wt (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt (gm)		
125	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt cm^2	.060	27.0	40.0	31.0	Electrodes from Cell 124.	Cycled 25 times. Poor performance.
126	A_u8	7 mg Pt/ cm^2	A_u14	7 mg Pt/ cm^2	-	-	40.0	31.0	Acid washed mat.	Very poor performance.
127	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	-	28	40.0	31.0	Acid-KOH washed mat.	Good initial performance. Δp transducer failed during 3rd cycle.
128	Am.Cy.	"	Am.Cy.	"	Same as Cell No. 125				Repeat of 125 with new O_2 electrode. Same mat,H_2 elect.	Good performance. 16 cycles. Internal reaction over weekend.
129	Am.Cy.	"	Am.Cy.	"	.060	27.5	40.0	31.0	Electrodes from cell 123. New mat.	Cycled 84 times. Fair performance. Gradual degradation developed a short.
130	Am.Cy.	"	Am.Cy.	"	.060	27.2	40.0	31.0	Electrolyzed KOH	29 cycles,initial. Fair performance.
131	Am.Cy.	"	Am. Cy.	"	-	-	-	-	Electrode from 127. Thin acid washed mat.	Internal reaction during flushing.
132	Am.Cy.	"	Am. Cy.	"	.060	27.5	40.0	31.0	Same electrode as 131	Used to check damage to electrode. Poor performance.

TABLE 1 (cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick (In.)	Mat Dry Wt (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt (gm)		
133	Am.Cy.	9 mg Pt/cm^2	Am.Cy.	9 mg Pt/cm^2	.060	26.5	40.0	31.0	Same electrode from cell No. 130	Poor performance.
134	Am.Cy.	"	Am.Cy.	"	.060	27.0	40.0	31.0	Electrode from cell 128. Electrolyzed KOH.	Cycled 72 times. Fair performance, gradual degradation.
135	Am.Cy.	"	Am.Cy.	"	.060	28.0	40.0	31.0	Electrolyzed KOH.	75 cycles. Gradual degradation.
136	Am.Cy.	"	Am.Cy.	"	.060	26.8	40.0	31.0	"	10 cycles. Stopped test due to gas leakage.
137	A_u22	7 mg Pt/cm^2	A_u30	7 mg Pt/cm^2	.060	28.0	40.0	31.0	-	Poor performance.
138	A_u25	"	A_u31	"	-	-	-	-	-	25 cycles. Fair performance.
139	A_u19	14 mg Pt/cm^2	A_u18	14 mg Pt/cm^2	.060	26.7	40.0	31.0	-	20 cycles. Fall off in performance at end of discharge.

TABLE 1 (cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick (In.)	Mat Dry Wt (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt (gm)		
140	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	0.060	27.0	40	31.0	Electrodes from Cell 136	Not subjected to test due to gas leakage.
141	A_u14	15 mg Pt/ cm^2	A_u18	15 mg Pt/ cm^2	0.060	27.5	40	29	Electrodes from Cell 139	Still cycling.
142	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	0.060	27.5	40	31.0	Electrodes from Cell 134	Cycled 46 times. Developed internal reaction & short. Gradual degradation.
143	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	0.060	27.0	40	31.0	Electrodes from Cell 135	Shorted out during 12th cycle.
144	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	0.060	27.5	40	31.0	Electrodes from Cell 140	9 cycles. Fair performance.
145	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	Nickel Plaque	0.060	27.3	40	31.0	-	Charged for 1-1/2 hrs.
146	Am.Cy.	Nickel Plaque	Am.Cy.	9 mg Pt/ cm^2	0.060	26.8	40	31.0	-	Charged for 1-1/2 hrs.
147	Mod. Am.Cy.	9 mg Pt/ cm^2+ 7 mg Pt/cm^2	Am.Cy.	9 mg Pt/ cm^2	0.060	27.5	40	31.0	Electrodeposited Pt on top of Am. Cy. electrode	Flat good initial performance. 53 cycles, gradual degradation.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode No.	O_2 Electrode Catalyst	H_2 Electrode No.	H_2 Electrode Catalyst	Mat Thick (In.)	Mat Dry Wt (gm)	Electrolyte % KOH	Electrolyte Wt (gm)	Comments	Results
148	Am.Cy.	9 mg Pt/cm^2	Am.Cy.	Nickel Plaque	0.060	27.4	40	33.0	-	Charged for 2 hours.
149	-	Nickel Plaque	Am.Cy.	9 mg Pt/cm^2	0.060	28.0	40	33.0	-	Charged for 2 hours.
150	Mod. Am.Cy.	9 mg Pt/cm^2+ 15 mg Pt/cm^2	Am.Cy.	9 mg Pt/cm^2	0.060	27.0	40	31.0	Electrodeposited Pt on top of Am. Cy. electrode	Cycled 11 times. Fair performance.
151	Mod. Am.Cy.	9 mg Pt/cm^2 + 7 mg Pt/cm^2	Am.Cy.	9 mg Pt/cm^2	0.060	27.0	40	31.0	Electrodes from Cell 147	-
152	Am.Cy.	9 "	"	"	0.060	27.0	40	31.0	Electrodes from Cell 150	Cycled 7 times. Fair performance.
153	A_u26	14 mg Pt/cm^2	A_u36	14 mg Pt/cm^2	0.060	27.0	40	30.0	-	2 cycles. Poor performance.
154	"	"	"	"	0.060	27.0	40	30.0	Electrodes from Cell 153	2 cycles. Slight improvement.
155	Mod. Am.Cy.	9 mg Pt/cm^2 + 14 mg Pt/cm^2	A_u40	14 mg Pt/cm^2	0.060	26.5	40	31.0	Pt Am. Cy. O_2 electrode. Nickel plaque hydrogen	2 cycles. Poor performance.
156	A_u26	14 mg Pt/cm^2	A_u36	14 mg Pt/cm^2	0.060	27.0	40	31.0	Electrodes from Cell 154	2 cycles. Poor performance.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode No.	O_2 Electrode Catalyst	H_2 Electrode No.	H_2 Electrode Catalyst	Mat Thick (In.)	Mat Dry Wt. (gm)	Electrolyte % KOH	Electrolyte Wt. (gm)	Comments	Results
157	Mod. Am.Cy.	9 mg Pt/cm^2 + 15 mg Pt/cm^2	A_u40	14 mg Pt/cm^2	0.060	27.0	40	31.0	Electrodes from Cell 155	1 cycle. Poor performance.
158	Bishop	7 mg + 15 mg Pt/cm^2	-	15 mg Pt/cm^2	0.060	27.5	40	31.0	Electroplated Bishop O_2 electrode	8 cycles. Good performance
159	Mod. Am.Cy.	9 + 15 mg Pt/cm^2	A_uCy.	9 mg Pt/cm^2	0.060	27.0	40	31.0	O_2 Electrode from Cell 157	17 cycles. Poor performance
160	A_u26	14 mg Pt/cm^2	-	14 mg Pt/cm^2	0.060	27.09	40	31.09	O_2 Electrode from No. 156. Quick change of electrode and mat.	Very poor discharge. 1 cycle.
161	Bishop	7 mg + 15 mg Pt/cm^2	-	15 mg Pt/cm^2	0.060	27.09	40	31.09	Electrodes were run in No. 158	12 cycles. Poor performance
162	A_u46	14 mg Pt/cm^2	A_u45	14 mg Pt/cm^2	.030 (Two of them)	27.09	40	31.0	Continuous concentration cell with reference.	Ran 6 days. Internal reaction during night.
163	A_u48	14 mg Pt/cm^2	A_u49	14 mg Pt/cm^2	.030 (Two)	26.5	40	31.0	Cycling concentration cell	Ran 34 cycles. Voltage went up to 10 on one side and 11 on the other.
164	A_u47	14 mg Pt/cm^2	-	14 mg Pt/cm^2	Same mat From 160	-	-	-	Put new O_2 electrode in cell No. 160	17 cycles. Ran poor.
165	A_u43	14 mg Pt/cm^2	A_u44	14 mg Pt/cm^2	.030 (Two)	27.1	40	31.09	Cycling concentration cell.	Gradual degradation.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick (In.)	Mat Dry Wt. (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt. (gm)		
166	-	EOS elect. with gold. over	-	Old washed gold.	.060	27.5	40	31.09	-	Improved with performance. 130 cycles.
167	A_u42	14 mg Pt/cm^2	A_u43	14 mg Pt/cm^2	.030 (Two)	27.0	40	31.0	Concentration cell.	Ran 6 days, turn off due to high voltage ref.
168	A_u47	14 mg Pt/cm^2	-	14 mg Pt/cm^2	.060	27.0	40	31.0	Electrodes from Cell 164	130 cycles. Ran poor.
169	57	EOS regular	56	EOS.	.060	27.0	40	31.0		105 cycles. Ran fair.
170	64	14 mg Pt/cm^2	65	14 mg Pt/cm^2	.060	27.0	40	31.09		Cont. conc. Still running.
171	Am.Cy.	7 mg Pt/cm^2	Am.	7 mg Pt/cm^2	.060	27.2	40	31.0	Concentration cell.	30 cycles. Voltage went up 1 Volt.
172	Chem Cell	Std.	Chem Cell	-	.060	27.1	40	31.0		Runs poor.
173	A_u52	-	A_u51	-	KTP-Pa	21.5	40	31.0		Still on test.
174	Chem Cell with Hydrophobic		Chem Cell with Hydrophobic		.060	27.2	40	31.09		Did not run.

A new mat was prepared as described above. After completion of the distilled water washing of the mat, it was reshredded and mixed with 40 percent KOH and allowed to react for one hour. After the KOH treatment, the fibers were once again washed with distilled water until a pH of 7 was reached. The mat prepared in this manner was assembled in cell No. 127 in conjunction with American Cyanamid electrodes on the hydrogen and oxygen sides. The cell was cycled three times and showed good performance as shown in Figure 1. At the end of the third cycle, an instrumentation failure (the differential pressure transducer) caused the test to be stopped.

Satisfactory performance of the acid-alkali treated mat (at least initially) indicates that this approach warrants further study.

Cell No. 128 consisted of the same hydrogen electrode and asbestos mat as employed in cell No. 125. A new oxygen electrode of the American Cyanamid type was substituted for the old oxygen electrode. Initial performance of the cell was good and the cell was allowed to cycle continuously. During the sixteenth cycle, which occurred over the weekend, recorded data shows that the cell developed an internal short. Performance of this cell is shown in Figure 2. Recovery of this cell's performance, by replacement of the oxygen electrode after a long successive series of tests, shows that the major form of deterioration in cell performance is a result of deterioration of the oxygen electrode that cannot be recovered by washing.

Cell No. 129 contained American Cyanamid type electrodes that have been utilized in cells 123, 121, and 120 (reported in the Sixth Quarterly report). This cell was cycled 84 times continuously during which it showed a gradual degradation in performance. Initial performance was fair, discharging at 0.7 - 0.75 volts. The results of this cell are inconsistent with the results obtained from cell No. 125, which had electrodes of a similar prior history. The lack of reproducibility between cells 125 and 129 indicates that differences exist in the cell active components. However, the conclusions drawn from

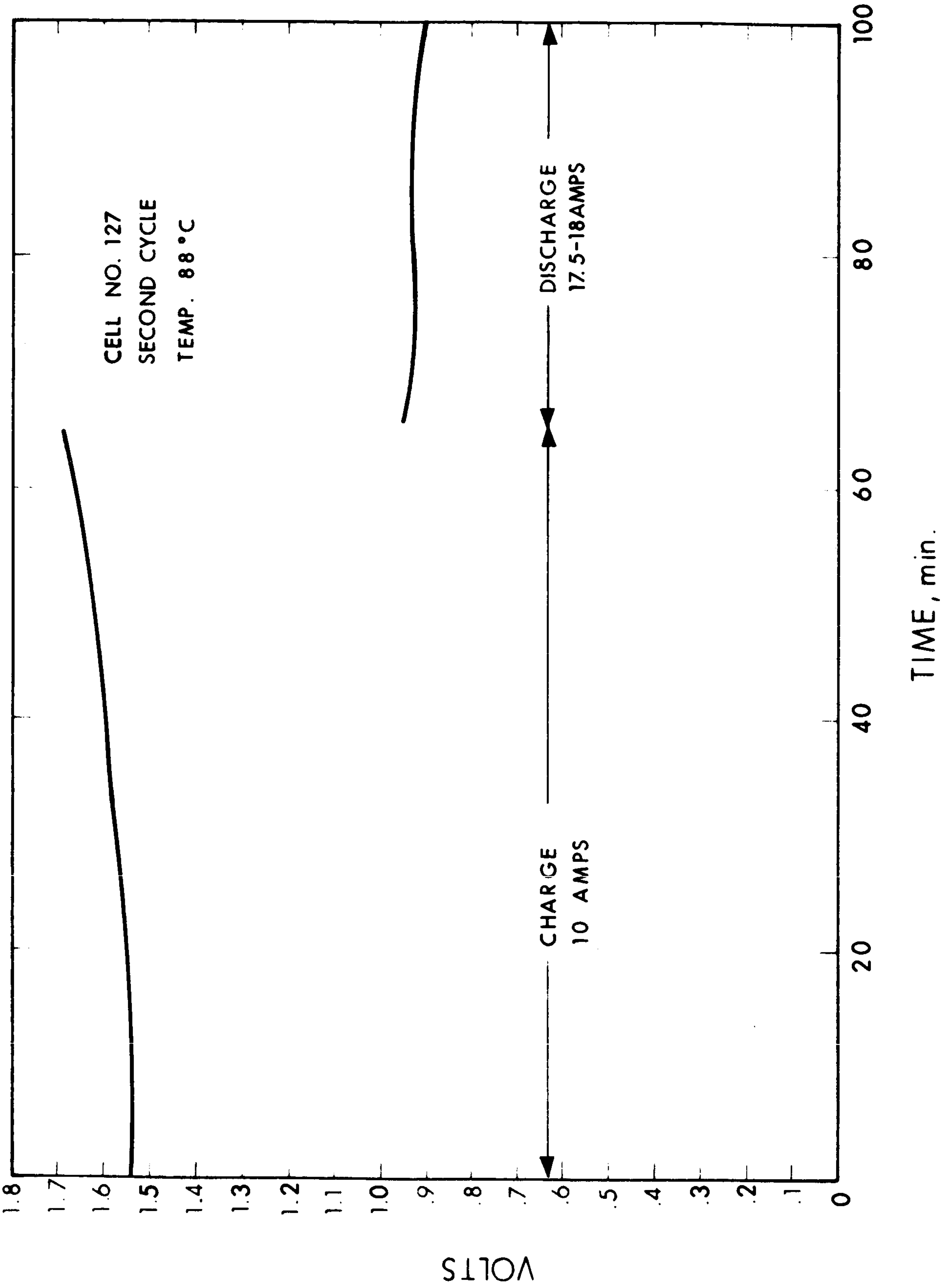

FIG. 1 CELL PERFORMANCE WITH HCl-KOH WASHED ASBESTOS

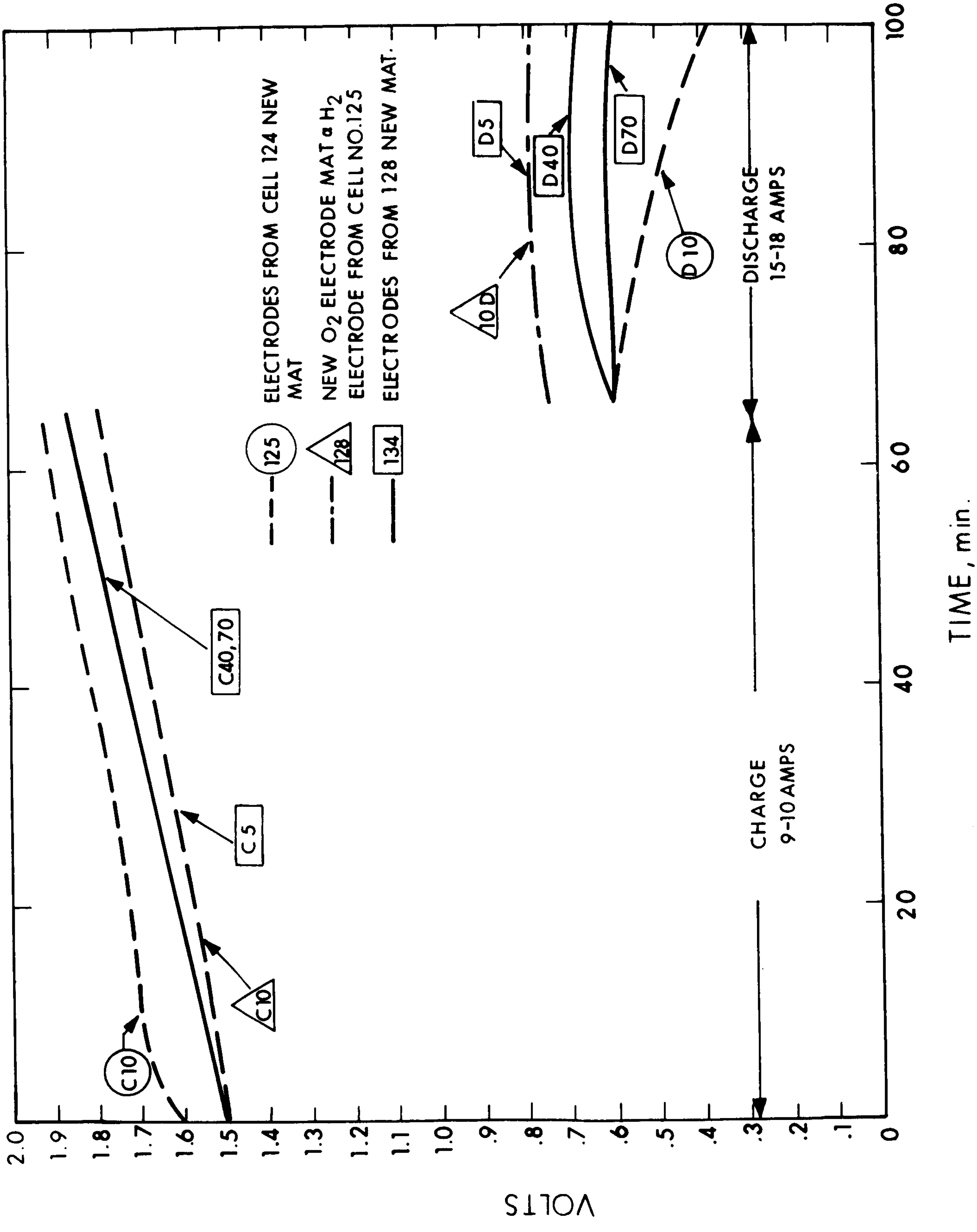

FIG. 2 CYCLING PERFORMANCE OF USED ELECTRODES

cells 125 and 128, i.e., that the oxygen electrode does not recover performance after use, were reconfirmed.

Cell No. 130 contained a new set of American Cyanamid electrodes with an asbestos mat that had been impregnated with pre-electrolyzed KOH. Pre-electrolyzing of the KOH solutions resulted in a deposition of a black deposit on the anode hydrogen evolving electrode. The pre-electrolyzing was conducted on platinum foil electrodes at 100 milliamps per square centimeter until no new black deposit was observed. This type of electrolyte was used in all subsequent cells and will be continued to be used in all future cell testing.

Cell 130 was cycled 29 times, and was discontinued because of mediocre performance. Cell No. 131 contained the Cyanamid electrodes previously used in cell No. 127 plus an acid-base washed mat that was somewhat thin. During initial flushing of the cell, an internal reaction occurred. Examination of the disassembled cell showed the edge of the asbestos mat did not make a satisfactory seal.

Cell No. 132 contained the American Cyanamid type electrodes employed in cell 131 with a standard asbestos mat to see if the reaction had caused any permanent damage to the electrodes. Initial performance was poor and the test was discontinued.

Cell No. 133 was essentially a repeat of cell 130, utilizing the same electrodes with a new asbestos mat and electrolyzed KOH. The performance of this cell was also poor.

Cell 134 contained the same electrodes as utilized in cell 128 with a new asbestos mat. The cell was cycled continuously for 72 times, initially showing fair performance and as cycling continued, a gradual degradation in voltage was noted. Figure 2 also shows the performance of this cell.

Cells 135 and 136 contained new American Cyanamid electrodes with standard asbestos fuel cell grade mats and electrolyzed KOH. They were run as controls. Cell 135 was cycled 75 times showing a gradual degradation in performance as shown in Figure 3. Cell 136 was cycled only 10 times at which time the test was stopped due to a gas leak in

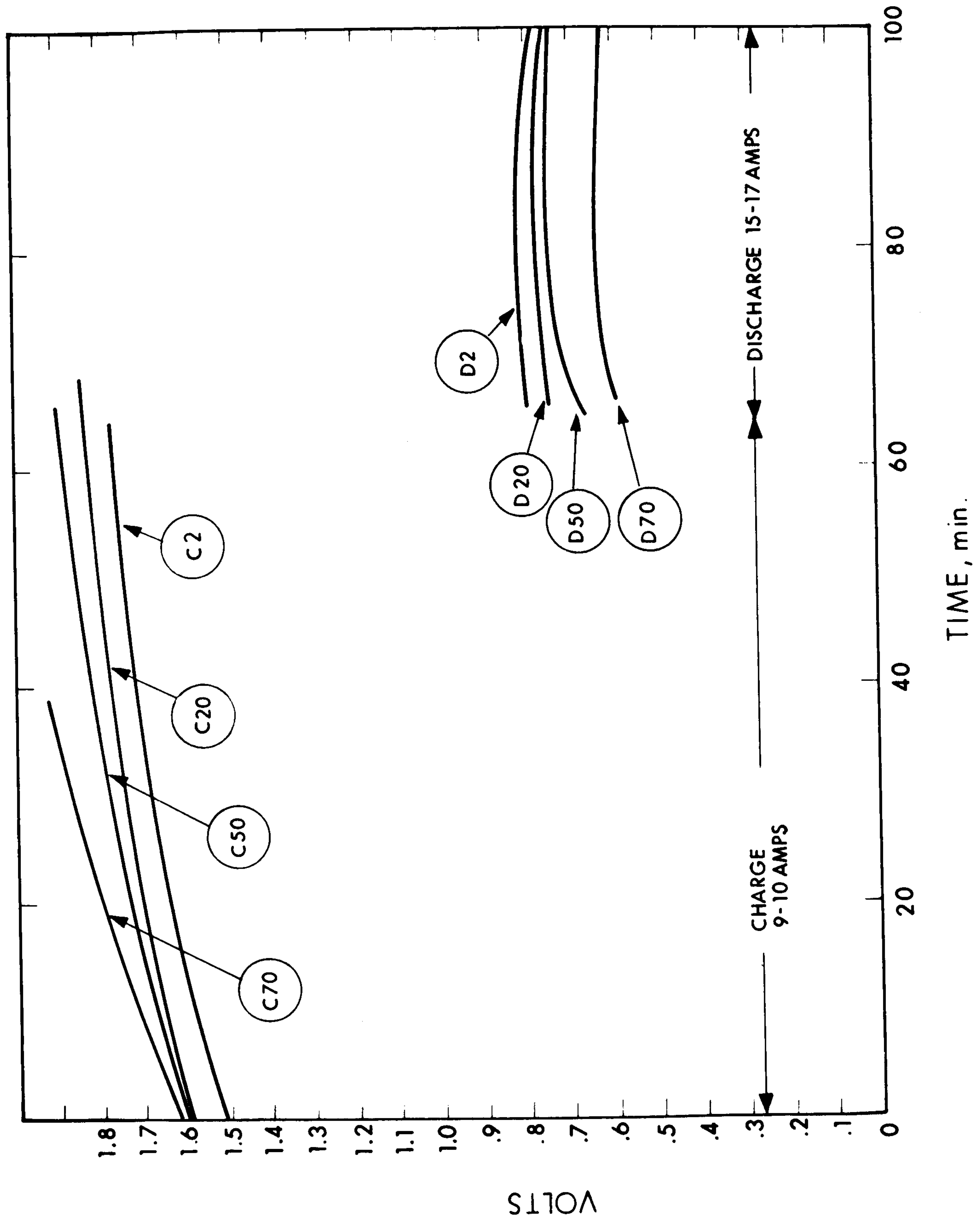

FIG. 3 CYCLING PERFORMANCE OF CELL 135

the hydrogen plumbing of the cell.

Cell Nos. 137 and 138 were also control tests, utilizing porous nickel plaque electrodes that had been gold coated and platinized plus mats containing electrolyzed potassium hydroxide. Cell number 138 was cycled for 25 times discharging at 0.75 to 0.8 volt. After 25 cycles the test was discontinued. Cell 137 exhibited similar performance. The above results showed that pre-electrolyzing of the KOH did not result in improvements in cell performance or life.

In order to improve the voltage levels obtained with gold-coated platinized electrode, a new set of electrodes was prepared utilizing gold-coated porous nickel plaque subsequently platinized, but the platinum loading was doubled to 14 milligrams per square centimeter. These electrodes were subjected to tests in cell number 139. The cell was cycled 20 times. During discharge, initial voltage performance was satisfactory, 0.85 to 0.9, but as the discharge proceeded, voltage fell off rapidly indicating flooding.

Cell No. 140 consisted of oxygen and hydrogen electrodes of the American Cyanamid type AB-6 that had been previously used in Cell 136. During the initial flushing of this cell, in the normal checkout procedure, a gas leak was observed and the cell was never subjected to test due to this gas leakage.

Cell No. 141 consisted of gold-coated and platinized porous nickel plaques containing 14 milligrams of platinum per cm^2. These electrodes have previously been used in Cell No. 139 showing good initial performance but a fall-off towards the end of discharge, indicating flooding. In the assembly of this cell, a smaller quantity of electrolyte than normally used was employed. The cell was subjected to the standard test cycle of 65 minutes charge and 35 minutes discharge and has been cycled continuously for 400 cycles. Performance of this cell is shown in Figure 4. There was a gradual deterioration in voltage with cycle life, but at a considerably slower rate than with previous cells. Performance degradation in all previous cells has resulted in a fall-off in voltage at the beginning and end of discharge. In the case

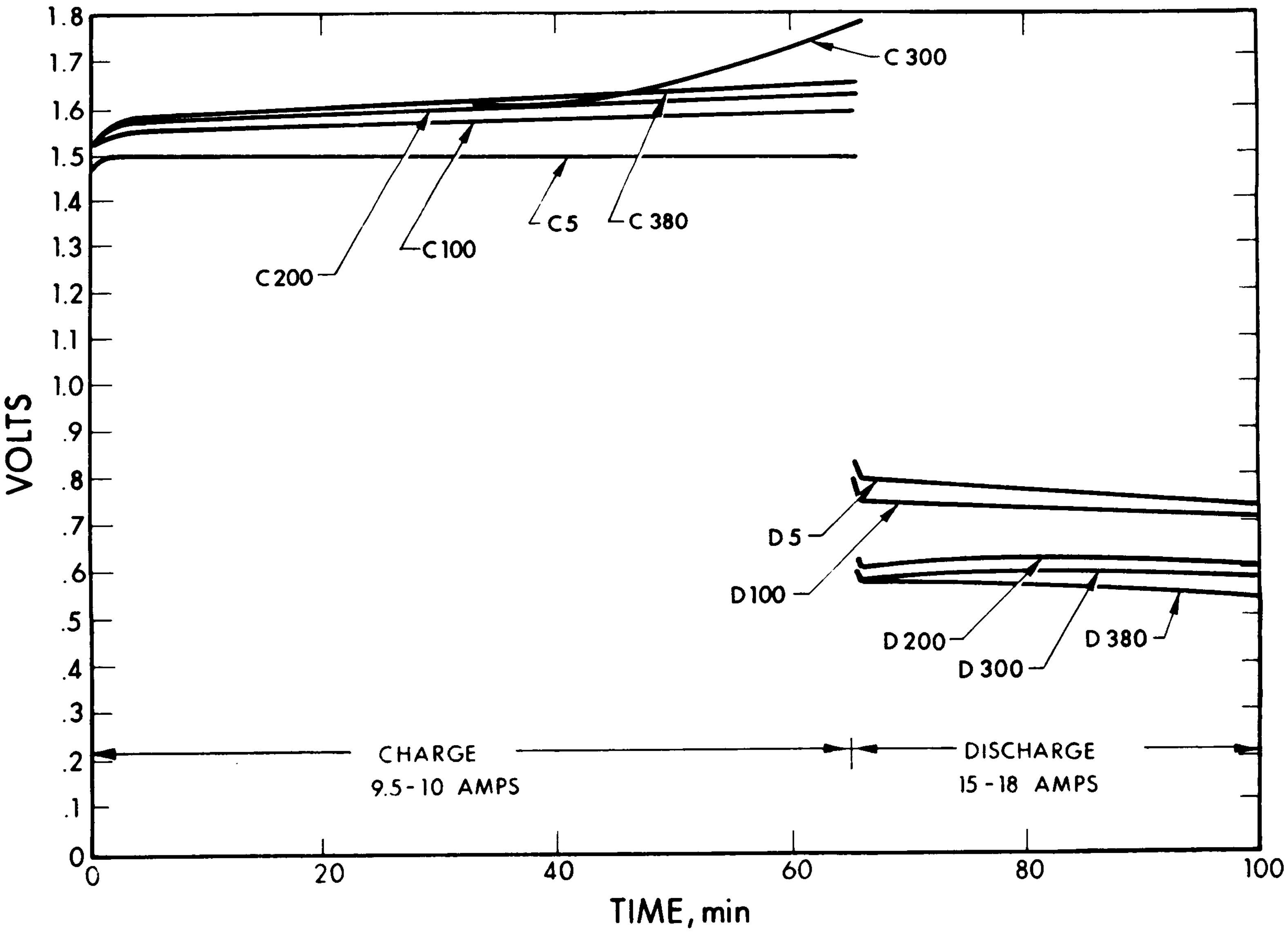

FIG. 4 CYCLING PERFORMANCE OF CELL SERIAL 141

of this cell, degradation showed up as a gradual lowering, but still maintaining a flat discharge voltage.

Cell No. 142 consisted of American Cyanamid type AB-6 electrodes that have been previously used in Cell 134. The electrodes were washed and reassembled with a new asbestos mat. The cell was cycled 46 times at which time an internal reaction and short developed within the cell. During the cycling period gradual degradation in the performance was observed.

Cell No. 143 consisted of American Cyanamid type AB-6 electrodes that had been previously used for Cell 135. These electrodes were also washed prior to use and assembled in the cell with new asbestos mat. This cell showed fair initial performance, but developed an internal short during the 12th cycle and the test was discontinued.

Cell No. 144 consisted of American Cyanamid type AB-6 electrodes that have been previously used in Cell 140. This cell did not exhibit any unusual performance characteristics and was shut down after 9 cycles.

Cells 145 and 146 were assembled in an attempt to determine the source of discoloration and platinum migration observed in previous cells. Cell 145 consisted of an American Cyanamid type electrode on the oxygen side and a porous carbonyl nickel plaque, uncatalyzed, on the hydrogen side. Cell 146 consisted of a nickel plaque on the oxygen side and an American Cyanamid type electrode on the hydrogen side. Both of these cells were charged continuously for 1-1/2 hours. It was difficult to remove the asbestos mats from the electrodes (due to loss of water during electrolysis), and no conclusions were reached from the test.

Cell No. 147 consisted of an American Cyanamid type AB-6 oxygen electrode that was modified at EOS by electrodepositing an additional 7 mg/cm^2 black platinum layer on one surface that was subsequently assembled in the cell adjacent to the asbestos mat. The hydrogen electrode employed was a standard American Cyanamid type AB-6 electrode. This modification of the electrode was an attempt to increase the thickness of the electrode structure and make the electrodes less sensitive to matrix water content. As shown in Figure 5, this electrode structure initially showed good flat performance, but as cycling continued, gradual degradation in the performance was observed. Previous tests with American Cyanamid electrodes showed considerably higher charging voltages, presumably due to a lack of availability of water in the electrode during the charge mode. Since the outer layer of electrodeposited platinum was not wet-proofed, this section of the electrode probably contributed considerably to the lowering of the charge voltage. This type of electrode structure indicates a possible technique for increasing the performance capabilities of American Cyanamid type electrodes, if the gradual deterioration in performance problem can be overcome.

Since the results of cells Nos. 145 and 146 were inconclusive, the repeat of this type test was conducted with cells 148 and 149. Cell 148 consisted of an American Cyanamid oxygen electrode and a nickel plaque hydrogen electrode. Cell 149 consisted of a nickel plaque oxygen electrode and American Cyanamid hydrogen electrode. In this case, the asbestos mats initially were impregnated with an additional two grams of electrolyte above that normally used in order to prevent sticking of the mats to the electrodes. The cells were charged continuously for two hours at 10 amps and then disassembled. There were no obvious discolorations observed on either of the asbestos mats. The only conclusion that can be drawn is that it is necessary to run the cells for a longer period of time to encounter meaningful changes in the asbestos mat.

Cell No. 150 consisted of a modified American Cyanamid type AB-6 electrode of the type used in Cell No. 147 with the exception that instead of 7 milligrams of platinum being electrodeposited on the electrode, 15 milligrams of platinum per cm^2 were electrodeposited. Performance of this cell was mediocre, and cycling was stopped after the eleventh cycle. It was hypothesized that the additional platinum possibly was blocking the lower active material and was not accomplishing any useful purpose.

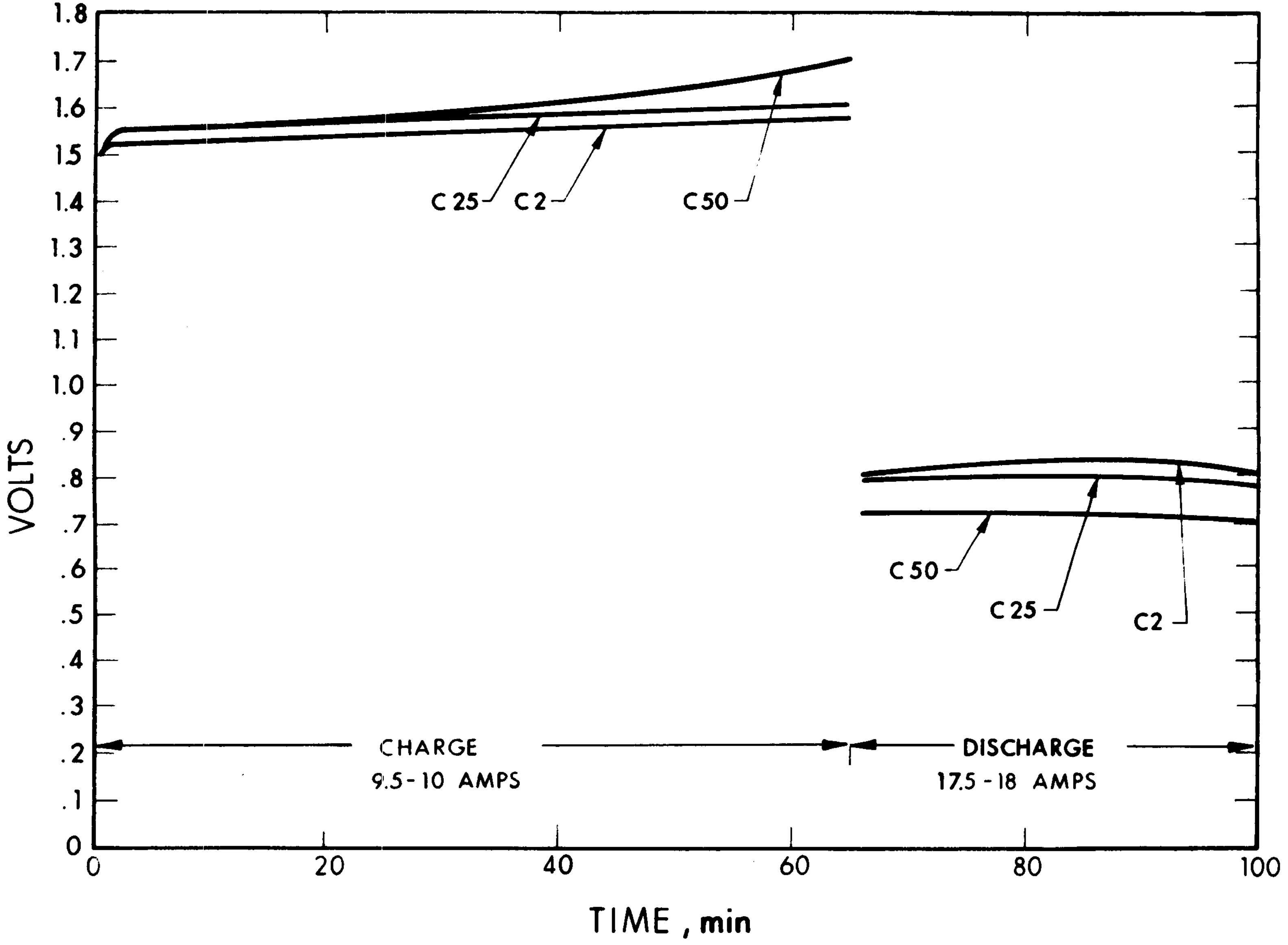

FIG. 5 CYCLING PERFORMANCE OF MODIFIED PLATINIZED AMERICAN CYANAMID ELECTRODE CELL SERIAL 147

Cell No. 151 consisted of the original modified American Cyanamid electrode that was used in cell 147. The electrodes were washed and reassembled with a new asbestos mat. Initial performance of this cell indicated some performance improvement over that obtained from the latter cycle performance of cell 147. The cell was cycled intermittently for an additional 53 cycles with gradual degradation.

Cell 152 consisted of the modified American Cyanamid oxygen and hydrogen electrodes that were previously used in cell 150. The electrodes were washed and then assembled with a new asbestos mat. The cell exhibited fair performance very similar to what was obtained with cell 150. After 7 cycles, the test was discontinued.

Cell No. 153 consisted of a new set of electrodes containing 14 milligrams of platinum per cm^2 electrodeposited on porous nickel plaques that had been gold-coated. The cell was put on the test and subjected to two cycles. Performance of the cell was extremely poor, operating at about 0.5 volt at 10 amps. Therefore, the cell was disassembled, the electrodes were washed, and reassembled with a new asbestos mat, in a new cell designated Cell No. 154. This cell showed only a slight improvement in performance over the results of the previous cell. After two cycles, this test was also discontinued.

Cell No. 155 consisted of an oxygen electrode of the American Cyanamid type that had 15 milligrams of platinum electrodeposited on the surface assembled adjacent to the asbestos mat. The hydrogen electrode was a porous nickel plaque that contained 15 milligrams of platinum electrodeposited on the surface assembled adjacent to the asbestos mat. This nickel plaque contained no gold-coating. The cell was subjected to test, exhibited poor performance and after two cycles, the test was discontinued.

Cell No. 156 consisted of the gold-coated platinized electrodes previously used in Cell No. 154. The electrodes were washed and assembled in the cell with a new asbestos mat. This cell showed poor performance and after two cycles, the test was discontinued.

Cell No. 157 consisted of the modified American Cyanamid electrode and the platinized nickel electrode previously used in Cell 155. These electrodes were washed and assembled with a new asbestos mat to see if the washing process would improve the performance. The cell showed very poor performance and after one cycle, the test was discontinued.

Cell No. 158 consisted of a porous nickel plaque oxygen electrode that was gold-coated at EOS and platinized by Bishop Metals to half the depth of the electrode with 7 milligrams of platinum per cm^2. An additional 15 milligrams of platinum per cm^2 was electrodeposited at EOS on the heavy platinum side which was assembled adjacent to the asbestos mat. The hydrogen electrode employed was a standard gold-coated platinized nickel plaque containing 15 milligrams of platinum per cm^2. The cell showed good initial performance, however, the cell developed a leak and the test was discontinued after 8 cycles.

Cell No. 159 consisted of the modified American Cyanamid electrode employed in Cell No. 157 on the oxygen side, an American Cyanamid electrode on the hydrogen side, plus a new asbestos mat. Performance was poor again, and the test was stopped after 17 cycles.

Cell No. 160 consisted of oxygen electrode AU26 which had been previously used on Cell No. 156, unwashed and a new hydrogen electrode and asbestos mat. The cell was cycled one time and exhibited a very poor discharge so the test was discontinued.

Cell No. 161 consisted of the oxygen and hydrogen electrodes that had been previously used in cell No. 158 with a new asbestos matrix. The electrodes were washed prior to the new assembly. The performance of the cell was flat in the charge and discharge mode, but the discharge voltage was poorer, i.e., approximately 0.6 of a volt. The cell was cycled 12 times, and then the test was discontinued.

Cell No. 162 was the first in a series of concentration cells that was initiated to obtain a better understanding of modes of deterioration. In oxygen concentration cell tests, both halves of the cell gas cavities are filled with oxygen to approximately 100 psig, and the electrodes. However, in this case, the polarity of the current flowing through the cell was reversed every 35 minutes to simulate a cyclic mode of operation. Therefore, for a 35 minute period one electrode was consuming oxygen and the counter electrode generating oxygen, and then the reverse polarity caused the electrodes to reverse function. Cycling was continued in this manner for a total of 34 cycles, or a total of approximately 40 hours. During this period, the voltage rose gradually to a value of approximately 1 volt at which time the test was discontinued. Initially, the cell exhibited a flat voltage during a cycle. As cycling continued, the voltage performance exhibited a slope as shown in Figure 7.

Cell No. 164 consisted of the same hydrogen electrode and asbestos mat that had been used previously in Cell 160 with a new oxygen electrode. The cell was cycled 17 times. It exhibited poor performance, possibly due to the exposure of the asbestos mat to CO_2 in the air during reassembly.

Cell No. 165 consisted of a cycling concentration cell similar in construction to Cell 163 utilizing a new set of electrodes and asbestos mat. The cell was cycled continuously for 146 times on the 35 minute cycle at 18 amps. During this period of cycling, the voltage rose gradually from initial level of approximately 0.6 to 0.95 volts. Similar to cell 163, the initial voltage performance was flat and as cycling proceeded, the voltage developed the gradual slope, as shown in Figure 8.

Cell No. 166 consisted of a new type oxygen electrode that was platinized by manually recycling chloroplatinic acid through a porous nickel plaque until 20 milligrams of platinum per cm^2 was picked up by the electrode. The O_2 electrode was then coated with electroless gold by recycling the electroless solution through the plaque. This was an attempt to coat over those areas of nickel that had not been coated by the platinum. The cell was assembled with a previously used gold-coated platinized hydrogen electrode in a standard 60 mil asbestos mat. The cell exhibited unusual performance initially operating at 0.5 to 0.6 volts and as cycling proceeded, discharge voltage performance increased gradually to a level of approximately 0.75 volts. During additional

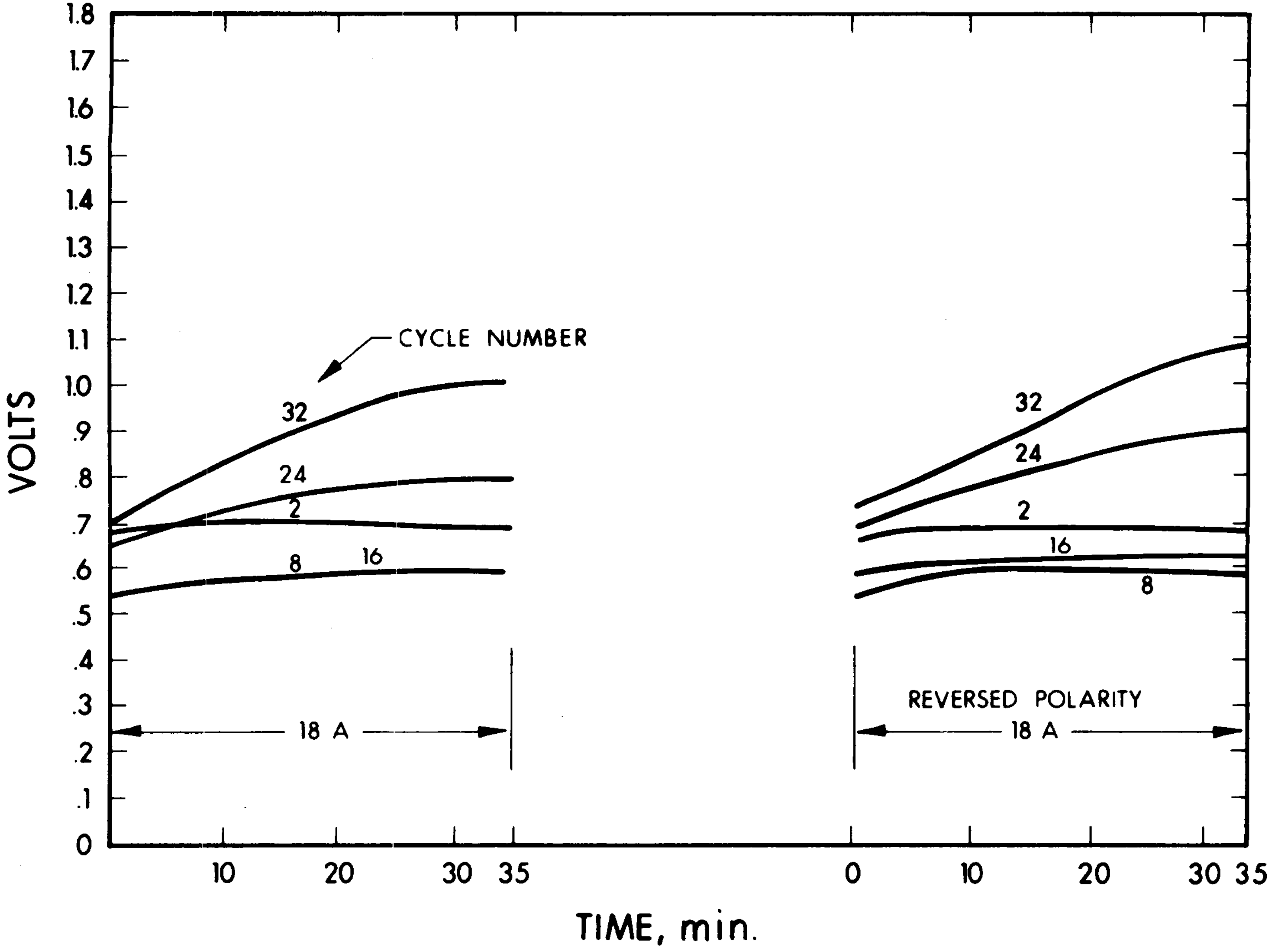

FIG. 7 CYCLING O_2 CONCENTRATION CELL 163

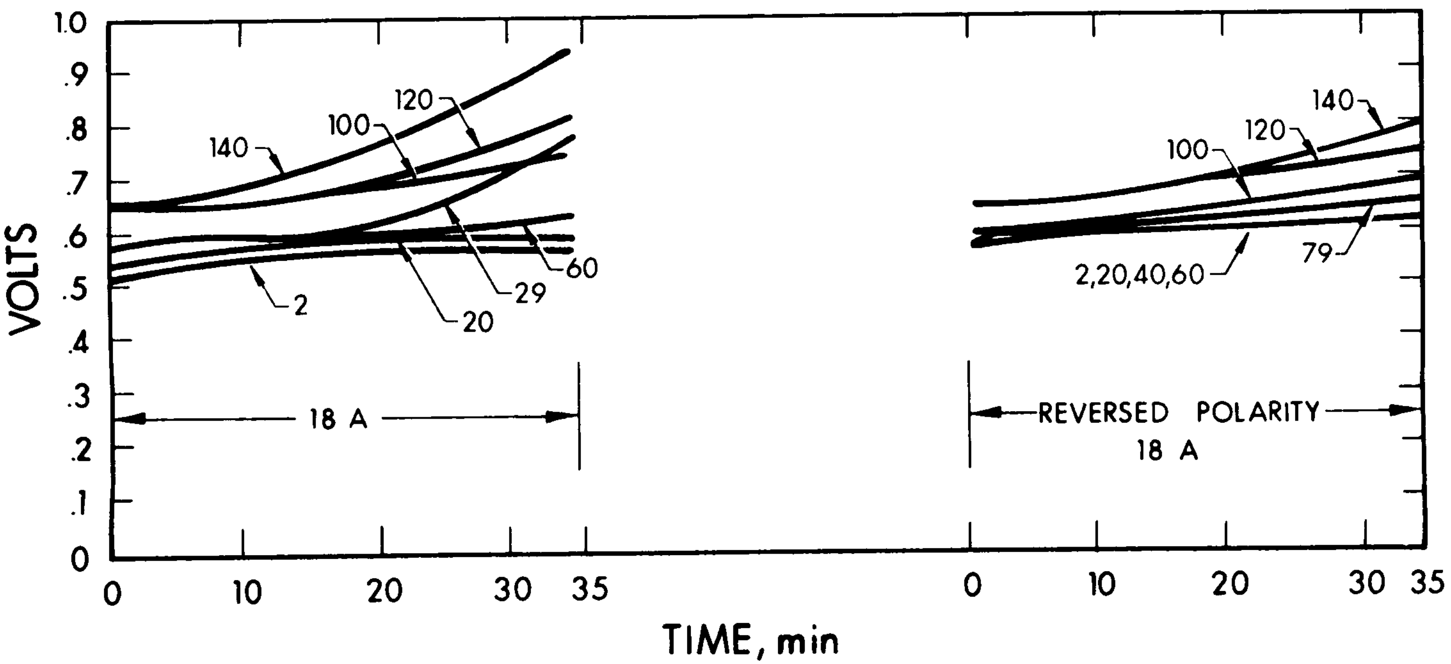

FIG. 8 CYCLING O_2 CONCENTRATION CELL 165

cycling, the discharge voltage degraded gradually to approximately 0.6 volts. Throughout this test, the unit was cycled a total of 130 times. Apparently, the gold layer initially had coated over the platinum and as testing continued (for some unknown reason), more catalyst area became available and performance improved.

Cell No. 167 contained gold coated, platinized electrodes. It was subjected to test in a continuous concentration cell similar to Cell No. 162. The cell was put on test at a constant 18 amps and exhibited a gradual rise with voltage in time. At the end of 4-1/2 days, recorded data showed a sharp increase in voltage. Examination of the test cell revealed that electrolyte was being squeezed out of the cell through the hole in the spacer between the cell halves that was used to install the reference electrode. This test was therefore discontinued.

Cell No. 168 consisted of the same electrodes as used in Cell No. 164, unwashed, with a new asbestos mat. The cell was cycled 130 times continuously and showed fair to poor performance discharging at 0.65 to 0.75 volts.

Cell No. 169 consisted of hydrogen and oxygen electrodes fabricated by the standard EOS technique of recycling a hot solution of chloroplatinic acid through a porous nickel plaque. The cell was cycled continuously for 105 times and showed a very slight degradation in performance during this period. Figure 9 shows the performance of this cell.

Cell No. 170 was a new continuous concentration cell utilizing gold-coated, platinized EOS electrodes. The cell was put on test at 18 amps and has shown a gradual increase in voltage with time. Figure 10 shows the voltage performance of the cell and at this point the cell is still running.

Cell No. 171 contained American Cyanamid type AB6 electrodes and was subjected to a cycling concentration type cell test. During a period of 30 cycles, the voltage rose gradually and reached 1 volt at the end of the 30th cycle. The test was therefore discontinued. The voltage of this cell exhibited an upward slope rather than a flat characteristic during cycling.

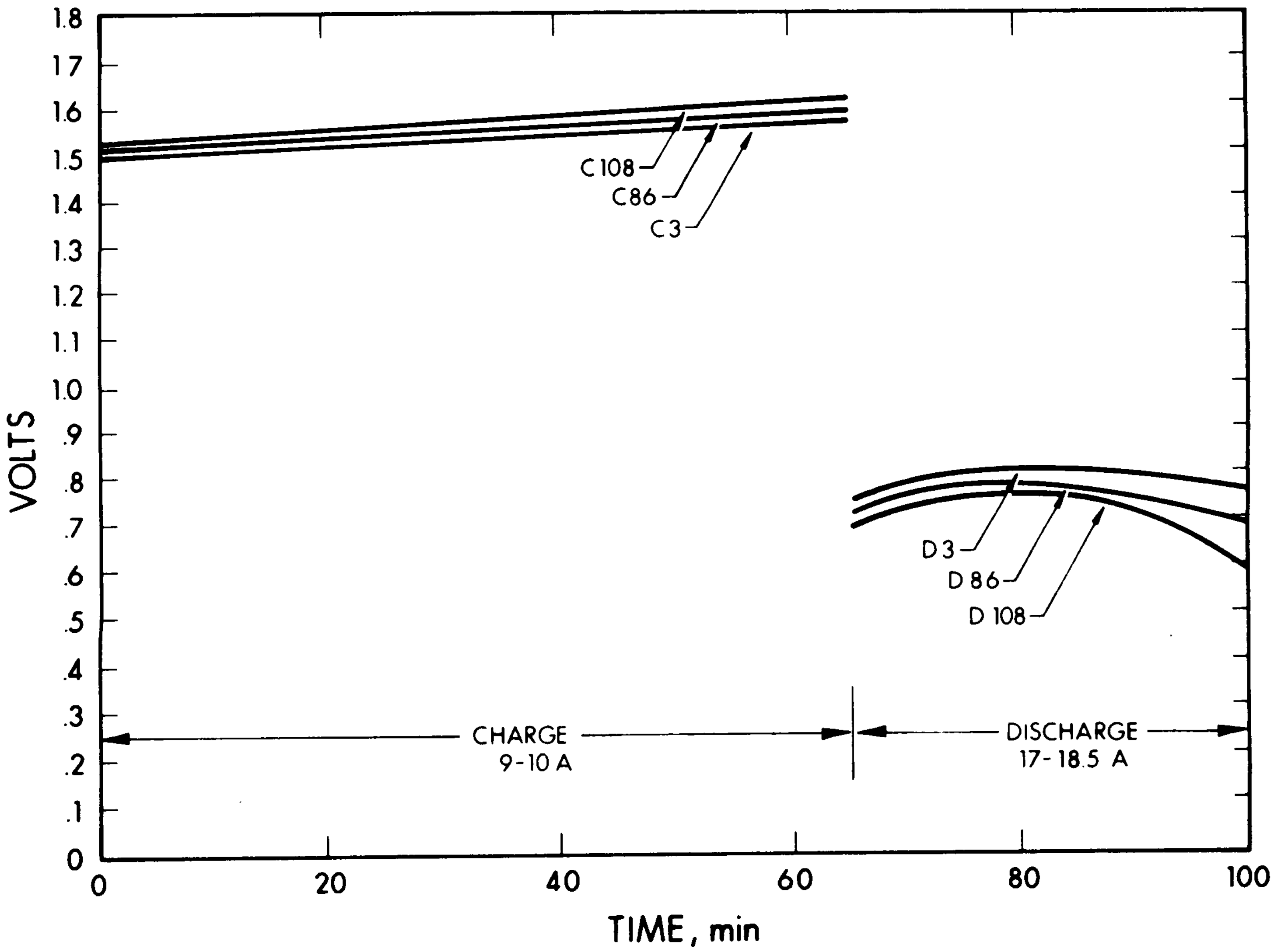

FIG. 9 CYCLING PERFORMANCE OF CELL 169

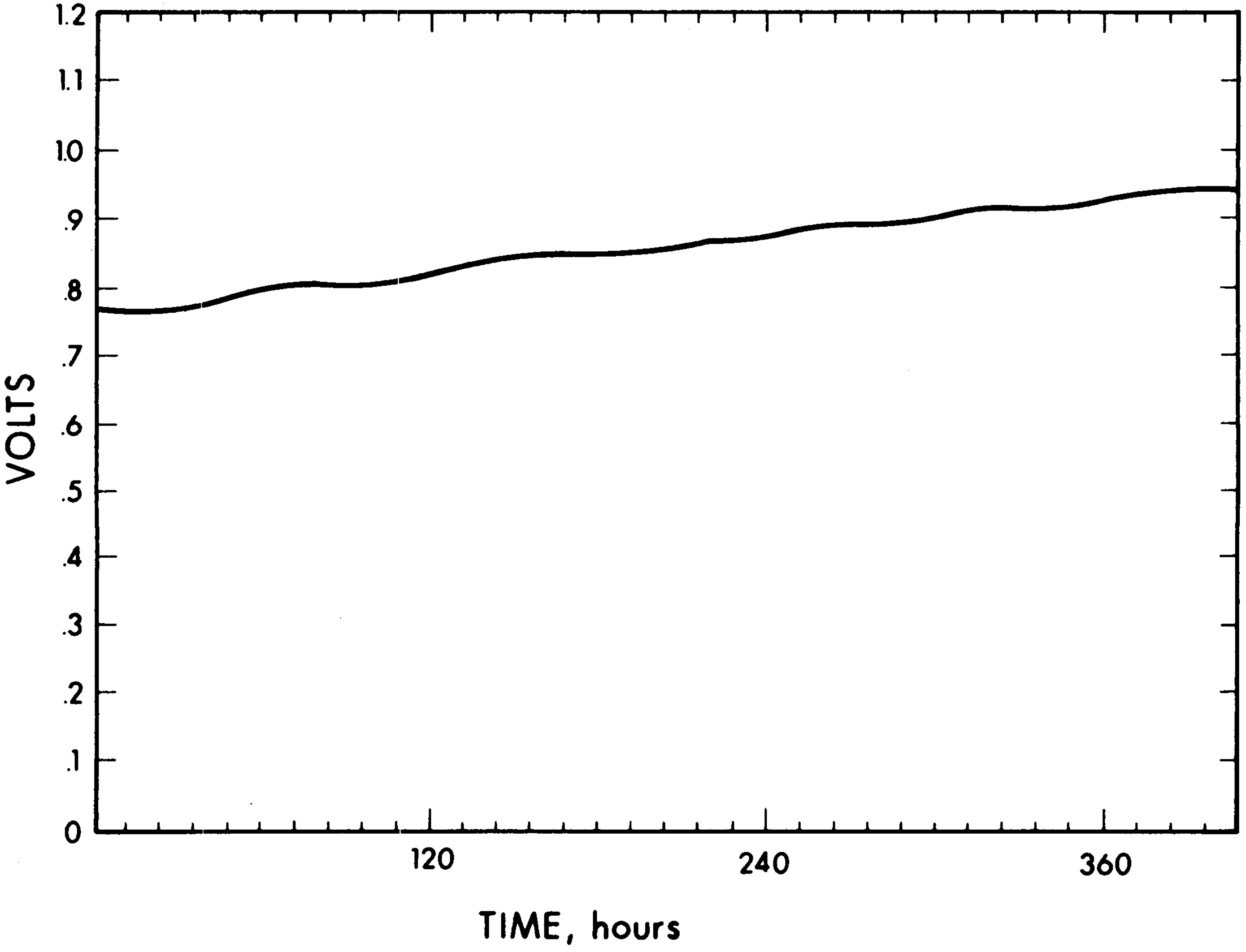

FIG. 10 CONTINUOUS O_2 CONCENTRATION CELL 170

Cell No. 172 consisted of a hydrogen and oxygen electrode that had been purchased from the Chem Cell Corp. for evaluation. These electrodes consist of a gold plated fine mesh nickel screen on which a mixture of black platinum and teflon is applied to form the reacting surface. The cell was put on the standard test cycle, but performance was such that cycling could not be carried out. These type electrodes appear very sensitive to the variations in water content that occur during the charge and discharge process of the secondary fuel cell. During one stage of the discharge the voltage performance was good i.e., approximately 0.85 volts at 17.5 amps, but the performance was poor at the beginning and end of discharge and charge, indicating flooding and drying of the electrodes. It appears that this type of electrode structure is not satisfactory for use in the RHO battery since it is apparently too thin and cannot tolerate the changes in moisture that are encountered with cycling.

Cell No. 173 consisted of gold-coated and platinized electrodes that were subjected to test in a cycling concentration cell. This cell utilized potassium titanate as the matrix material to determine if this material could tolerate the cell environment. This cell is still on the test and has been subjected to 150 hours of testing at which point there has been a very gradual degradation of performance. Figure 11 shows cell mid-point voltage vs. test time.

Cell No. 174 consisted of a variation of a Chem Cell electrode containing an added porous teflon hydrophobic layer located adjacent to the asbestos mat in the cell assembly. The water proofing layer is supposed to prevent drowning of the electrode as water is formed in the cell. However, it was found that the cell could not be subjected to test due to an extremely high internal impedance. This apparently was due to the non-wetting nature of the hydrophobic layer between the electrodes and the asbestos mat. It is possible that the electrolyte to asbestos weight ratio and the asbestos compression ratio used in setting up this cell, which was originally optimized for EOS type electrodes, is not well suited for the Chem Cell type of structure.

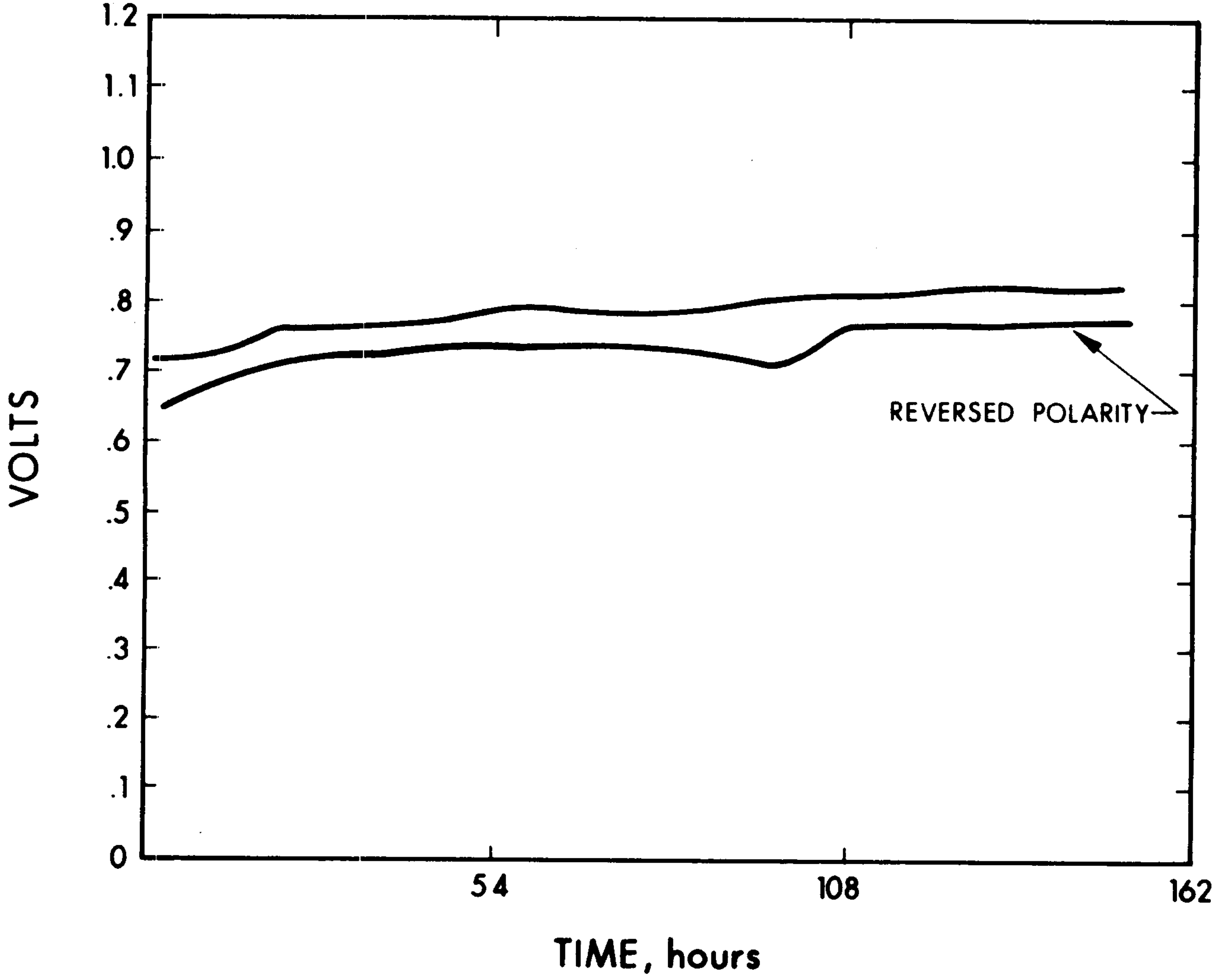

FIG. 11 CYCLING O_2 CONCENTRATION CELL 173
(MIDPOINT VOLTAGE)

Table 2 shows a summary of the concentration cell tests that have been conducted thus far. In all cases there has been a gradual degradation in performance with time, and the final KOH analysis of the asbestos mats have revealed drops in excess of 10 percent KOH. There is considerable discrepancy in the duration of tests to a common point of degradation. The discrepancy in rate of degradation encountered with the concentration cells and other single cell tests points to a lack of consistency in the cell components, or test method. The active cell components consist of electrodes, electrolyte and asbestos matrix. High purity (pre-electrolyzed) electrolyte has been used in recent cell tests and this would certainly not be the cause of the lack of reproducibility. Both EOS and American Cyanamid type fuel cell electrodes have shown a lack of reproducibility in our performance tests. However, American Cyanamid has reported good reproducibility when using their electrodes in primary fuel cells. Further, it is believed that the process techniques and materials used in the fabrication of EOS type electrodes are reproducible. Therefore, the material that has potentially the greatest variability is the asbestos matrix. Fuel cell grade asbestos is fabricated from a variable mined ore which is pulped to remove large impurities, washed to remove water soluble impurities and then processed into mats. Thin mats, i.e., 0.020" and under are made in a "paper" machine while mats thicker than 0.020" are made on a "board" machine. Apparently more asbestos per unit thickness is applied to the board material as compared to the paper material. Since in the RHO battery, an 0.060" board is used as the matrix rather than the 0.020" paper used in primary cells, it appears that we have 3-4 times the possible contamination present. Therefore, it is certainly possible that the more rapid rate of degradation of the RHO battery compared to primary cells of the American Cyanamid type is due to a higher quantity of asbestos impurity being present. Further, the irreproducibility could be attributed to the same factor.

TABLE 2

SUMMARY OF CONCENTRATION CELL RESULTS

	Continuous			
Cell No.	Electrolyte	Matrix Type	Time to .9-1 V.	Final KOH
162	Gold coated and Pt.	Asbestos	140 hours	Reaction
167	" "	"	130 hours	29.8%
170	" "	"	410 hours	Still on test at .92 volts
	Cycling			
163	" "	"	40 hours	-
165	" "	"	152 hours	26.8%
171	Am. Cy.	"	35 hours	28.8%
173	Gold Coated and Pt.	Kt.	150+	Still on test

3.2 Multicell Testing

In accordance with the program work statement, any single cell electrode type having a cycle life capability of > 100 hours at a satisfactory performance level is to be evaluated in a 6-cell test assembly. The gold-coated, electro-platinized nickel substrate electrodes exceed this criterion. Therefore, a 6-cell, 75-watt nominal battery was assembled. The 6-cell unit, designated serial No. 108, was similar in construction to the multicell unit serial No. 107, described in the 4th Quarterly Report dated 18 July 1965, with the following exceptions:

1. The electrodes on both the hydrogen and oxygen sides were gold-plated nickel plaques platinized to 14 mg/cm^2.
2. The backup gas distribution screens behind the electrodes and the bipolar plates were gold-plated to prevent possible oxidation of the nickel surfaces.
3. The hydrogen and oxygen end plates employed were of the type used in the 34 multicell design that allows for the incorporation of the end stack electrodes directly on the end plate.
4. A bellows with a modified flange to accommodate the 34 cell end plate.
5. A new teflon bellows stop to compensate for stack length changes caused by (3) and (4).

The cell was operated for 10 cycles at which time a short developed that fully discharged the unit to 0 volts. The short was traced to a failure of the insulation on the main feed-thru terminal through the stack. This terminal consisted of a 1/4 inch copper rod that is sealed and insulated from the tank by means of a teflon ring. Apparently this ring of teflon gradually flowed allowing the copper rod shank to short out. Difficulties with this fitting have been encountered in the past, and it was decided to replace this with a different feed-thru that employs a 1/8 inch copper rod and allows for a much thicker teflon insulation ring.

The feed-thru terminal was replaced and the cell was once again subjected to test. The cell was operated on an intermittent basis due to gradual differential pressure buildups that indicated possible gas leakage. The voltage performance of the unit is presented in Figure 12. As can be seen, there is a gradual degradation in performance as the cycling continued.

Testing of the unit was stopped after 150 cycles at which point the unit was disassembled and examined. In the examination of the internal assembly, it was found that a build up of corrosive products had developed on the end of one of the stack bolts, which was moist with liquid. Apparently, liquid was squeezed out of the stack and collected across the insulating washer between the bellows flange and the stack bolt. The stack is designed such that the stack bolts are electrically in contact with the opposite end plate and the full stack potential exists between the bellows flange and the bolts which is insulated by a fiber glass washer. Apparently, moisture bridged this washer and set up local corrosion and electrolysis in this area. The source of the moisture was not determined, but it was decided in future units to insulate the stack bolts on both ends of the stack to prevent this problem. All other internal components appeared clean and showed no visual signs of corrosion. Gold plating of the bipolar plates and internal hardware apparently quite effectively prevented any oxidation or corrosion. As in the past, the asbestos matrices had a gray to black discoloration adjacent to the hydrogen electrode. Samples of asbestos from the 1st, 2nd and 5th cell were analyzed for KOH concentration and were found to be 26.9%, 28.9% and 29.5% respectively.

To evaluate the new insulated bolts and to obtain additional test data, a second six-cell unit utilizing the same type of electrodes, serial No. 109, was assembled and subjected to test. The only difference between multicells 108 and 109 was that a thin teflon sleeve was used on the stack bolts where they fed through the hydrogen end plate to isolate the stack bolts electrically. The unit was subjected to the standard test cycle, and from the start exhibited poor

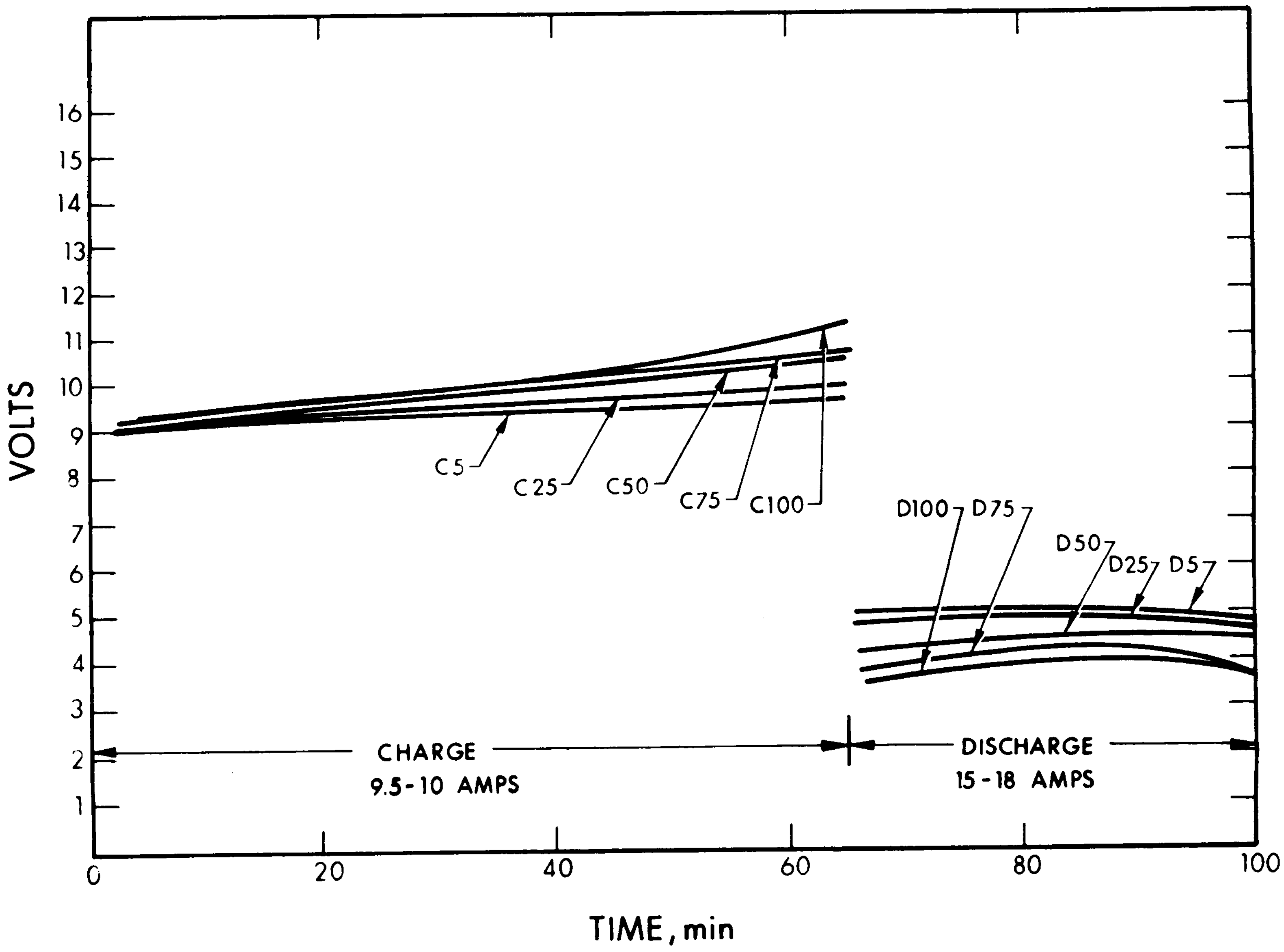

FIG. 12 CYCLING PERFORMANCE OF 6-CELL REGENERATIVE FUEL CELL SERIAL 108

performance as shown in Figure 13. The unit was cycled 15 times and the test was stopped to evaluate the cause of the poor performance. This evaluation is in process and the results will be reported in the next quarterly report.

3.3 Asbestos Studies

Wet chemical analysis of standard fuel cell grade asbestos has been completed and was reported in the Sixth Quarterly Report. In order to determine the effect of KOH impregnation of the mat on the analytical procedure, asbestos samples impregnated with 40 percent potassium hydroxide were analyzed as per procedures recommended by Johns-Manville. Results of the previous test and the new test are shown in Table 3 for comparison. The major constituents weight percentages are in fair agreement from the two samples. The loss upon ignition is considerably different, apparently due to the potassium hydroxide. Weights percentages in this table are based on the dry weight of asbestos i.e., without potassium hydroxide.

Additional analytical tests have been initiated on samples removed from cells that have been subjected to electrical tests, results of which will be reported in the future.

Analytical tests were conducted on samples of asbestos taken from cells that had been cycled to determine quantities of platinum in the mat. A new gravimetric procedure was used to make these determinations. A one square inch sample, taken from the mat used in cell No. 103, yielded 26.3 milligrams of platinum. The results of electrical testing of cell No. 103 were reported in the Sixth Quarterly Report. (The electrodes used in cell 103 consisted of a gold-coated platinized nickel plaque as the oxygen electrode and a platinized nickel plaque as the hydrogen electrode.)

Similar tests were conducted on a mat sample from cell No. 106, which employed American Cyanamid type electrodes. The mat in this case yielded 13.6 milligrams of platinum from a one square inch sample. (Electrical test results of cell 106 were also reported in the Sixth Quarterly Report.) These tests verify the previously reported

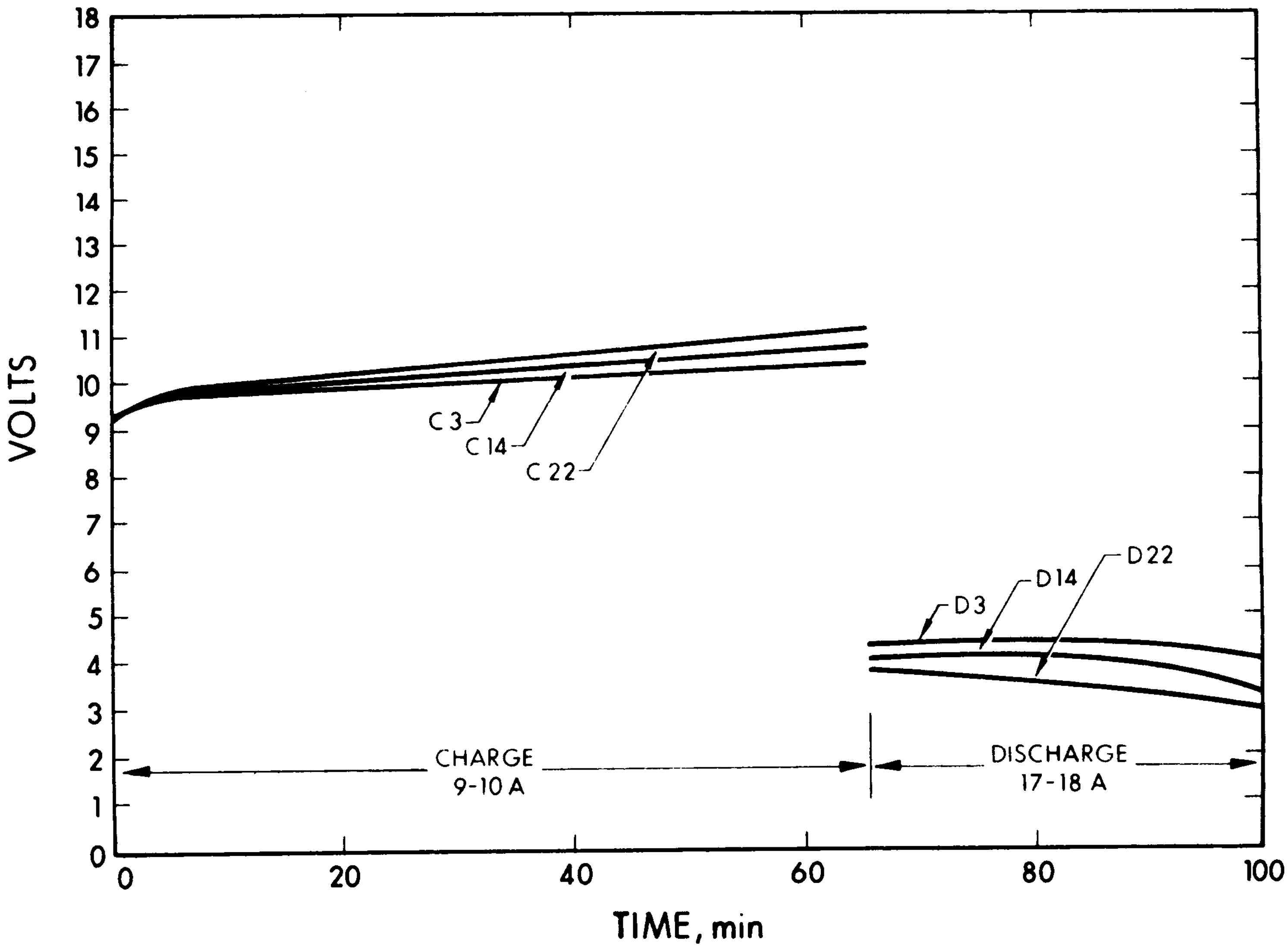

FIG. 13 CYCLING PERFORMANCE OF 6-CELL REGENERATIVE FUEL CELL SERIAL 109

TABLE 3

ASBESTOS ANALYSIS RESULTS

	Standard Asbestos (%)	Asbestos Impregnated with KOH (%)
Loss upon ignition	17.39	38.8
S_i	18.25	16.7
M_yO_x	2.4	1.53
Ca	2.45	2.0
Mg	22.9	21.3

results in that quantities of platinum are migrating into the asbestos mat during cycling. The source of the platinum has been determined as the H_2 electrode, but the mechanism of migration has not been determined as yet.

To establish better control of the physical characteristics of the asbestos matrix, a new control procedure was developed to determine porosity. Figure 14 shows a sketch of the test apparatus, which consists of a cell in which the asbestos sample is clamped, compressed N_2 gas and a flow meter. The rate of flow of gas through the matrix is dependent on pressure and the porosity of the matrix. An arbitrary differential pressure of 24 mm of mercury was selected for test of the asbestos matrices, and the flow rate was recorded for 49 different samples. Figure 15 shows a distribution of flow rate as a function of the number of samples. As can be seen, approximately half of the mats fall in the range of 230-240 ml/min. Figure 16 shows a weight distribution of the same group of matrices. Based on this test data, it has been decided to utilize asbestos mats in the range of 230 to 240 ml/min. flow as a criteria of selection for material to be used in cells. There was no apparent correlation between the mat weight and flow rate. However, all of the mats in the 230-240 ml/min. range fell within ± 5% of the average weight of $\approx$ 26.8 grams. This procedure appears to be a suitable first cut at selecting matrices and will be utilized in future testing.

3.4 Analytical Tests

An analytical test of a gold-coated carbonyl nickel plaque was conducted to determine the quantity of gold that is actually deposited in the plaque. The electroless process employs 400 cc per electrode of a solution that contains one ounce of gold per gallon. This results in the theoretical deposition of 3.29 grams of gold per plaque. Next, the plaque is electroplated with gold at a half an ampere per hour, resulting in a theoretical yield of 3.66 grams of gold. Therefore, the total theoretical yield is 6.95 grams of gold per electrode. In the analytical test, a standard gold-coated plaque was dissolved in acid, yielding 6.64 grams of gold. This represents slightly better than 95%

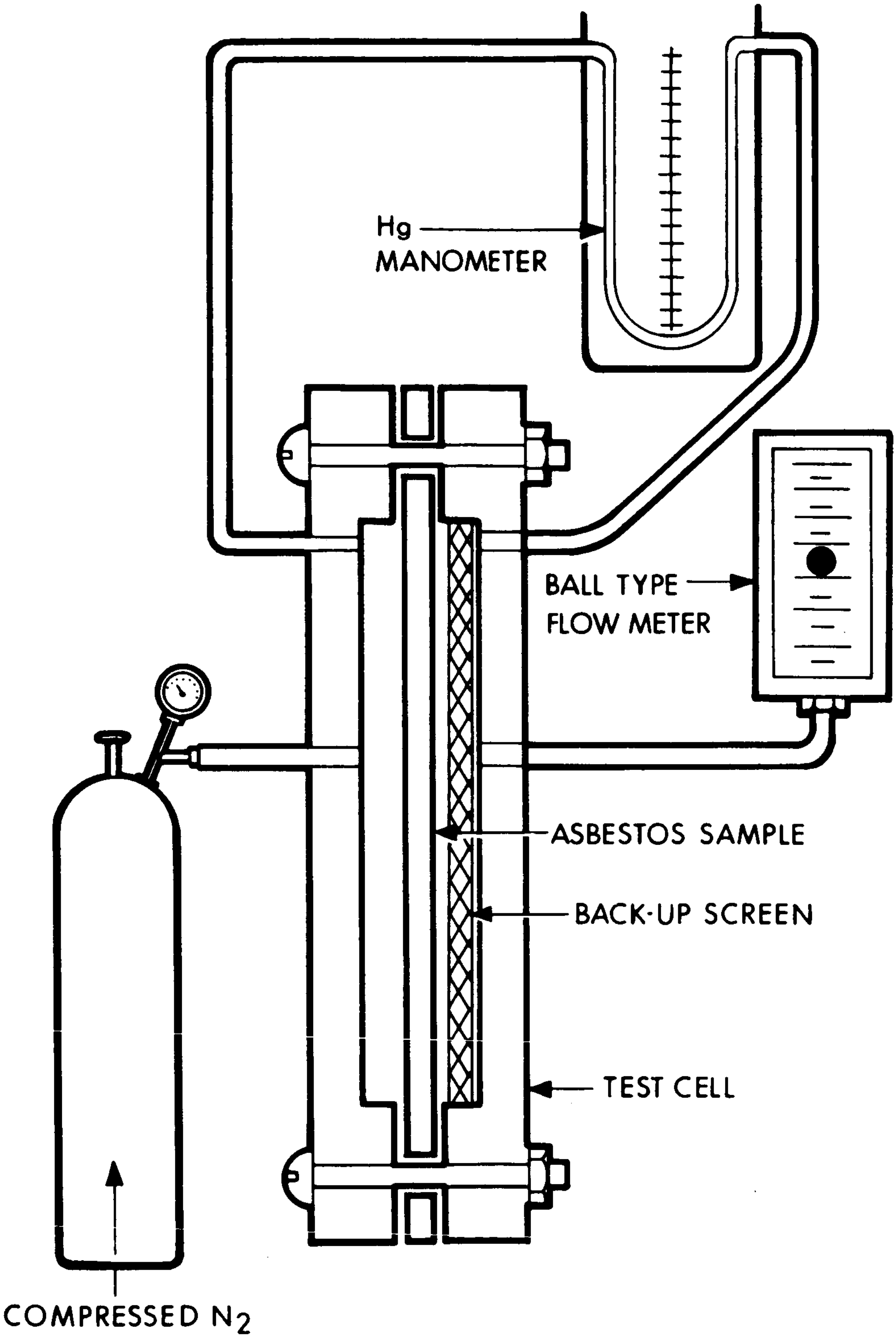

FIG. 14 FLOW TEST APPARATUS FOR ASBESTOS

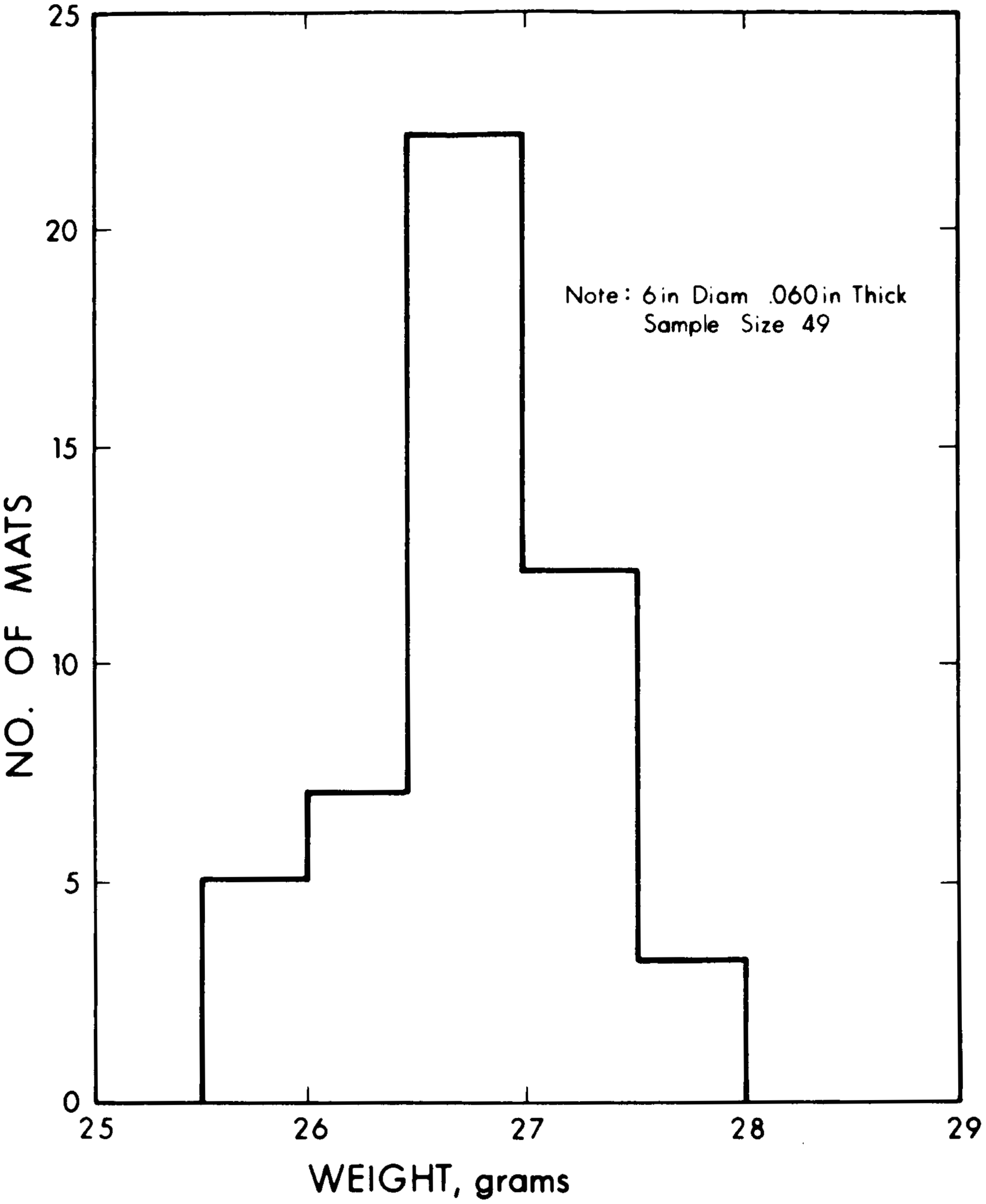

FIG. 15 PLOT OF WEIGHT DISTRIBUTION FOR ASBESTOS SAMPLES

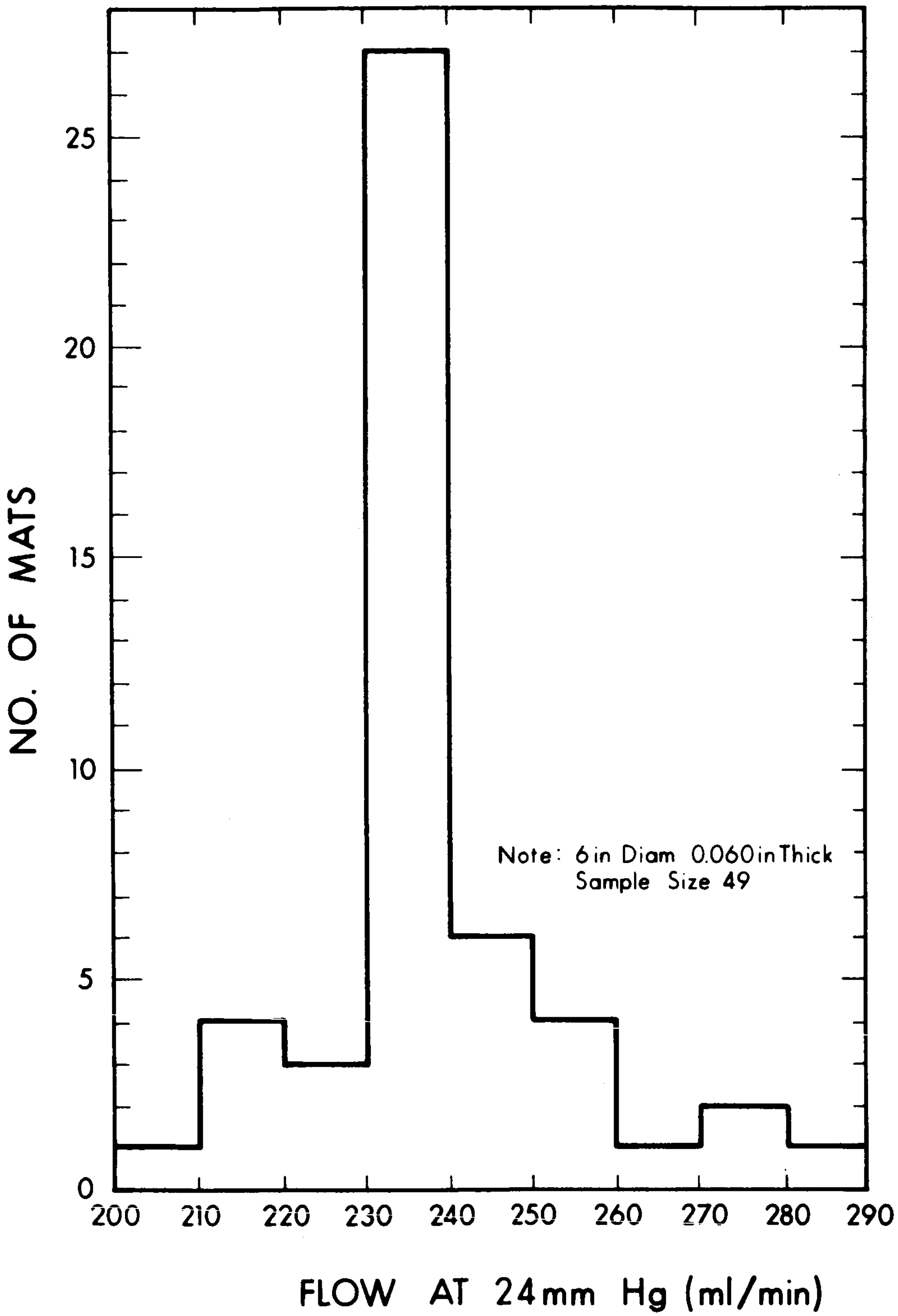

FIG. 16 PLOT OF FLOW DISTRIBUTION FOR ASBESTOS SAMPLES

of the theoretical gold employed. The test was conducted as a check of our processing, to assure that we were obtaining the gold coverage intended.

3.5 Instrumentation

During this report period, a new test console, capable of cycling five single cells and two six cells, was completed. The console is capable of continuously monitoring cell voltage, cell current, differential and total pressures and temperature for the five single and two six cell units. A wiring diagram for the test console is shown in Figure 17.

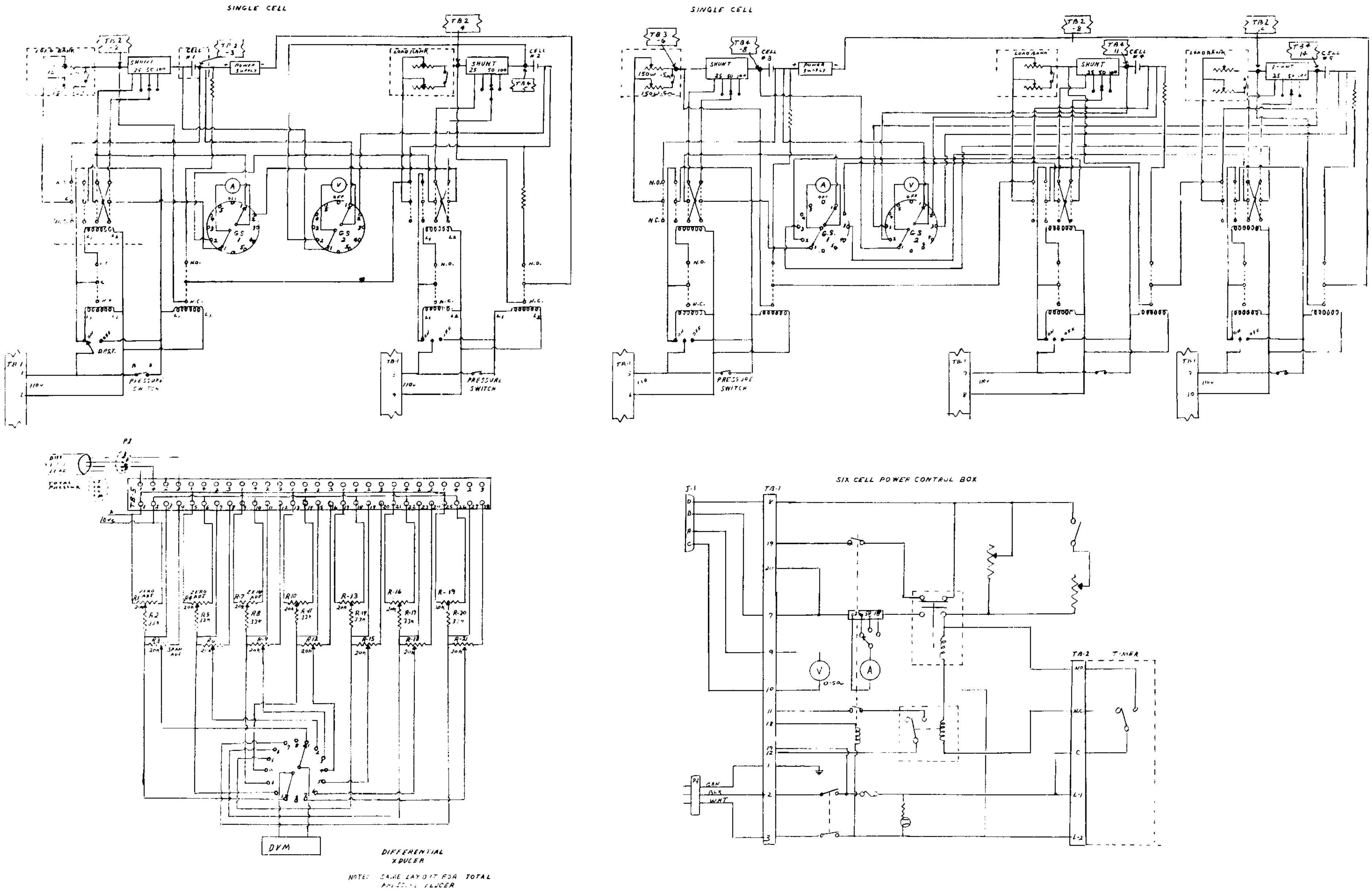

FIG. 17 WIRING DIAGRAM OF FUEL CELL TEST CONSOLE

4. CONCLUSIONS

The primary mode of deterioration of cell performance with cycling has not as yet been determined. It appears that secondary modes, consisting of (a) gradual reaction of the potassium hydroxide electrolyte with the asbestos matrix; (b) gradual oxidation of the porous nickel electrode substructure; and (c) migration of platinum from the anode, also exists. Replacement of the asbestos matrix in a degraded cell does not result in performance recovery showing that electrolyte consumption is not the primary mode. Gradual degradation in performance of American Cyanamid type electrodes which contain essentially no available nickel indicates that nickel oxidation is also not a primary mode of failure. Likewise, the gradual degradation of performance in concentration cells, with no apparent visual platinum migration, indicates that this too is not the primary mode. All indications by difference and circumstance indicate that the asbestos matrix contains constituents that cause the degradation. Further, these constituents either become active in the electrolysis mode or are more concentrated in the RHO battery, since primary fuel cells are reported to have been run successfully for long durations using similar type asbestos materials.

A considerable number of tests have been made with Cyanamid and EOS electrodes. Table 4 compares these based upon an overall observation of performance. It is our belief that the ideal regenerative electrode would combine certain aspects of the EOS and Cyanamid electrodes. Desirable electrode features are as follows:

(1) No nickel content

(2) Relatively thick (20 mil) porous substrate catalyzed uniformly in depth

(3) Catalyst loading of 10 - 20 mg/cm^2

(4) Catalyst mechanically bonded to substrate.

The desirability of wet-proofing is not well defined as yet, but indications are that it is not required.

TABLE 4

ELECTRODE PERFORMANCE COMPARISON

Performance Test	Std. EOS 20 mg/cm^2	Am. Cya. AB4, AB6, 9 mg/cm^2	Gold-Plated EOS, 7-14 mg/cm^2
Charge Performance	Very good	Fair to Poor	Very good
Discharge Performance	Good	Excellent at peak. Poor to Fair at beginning and end of cycle.	Fair
Cycle life	Fair	Poor	Fair
Oxidation resistance of substrate	Poor	Excellent	Initially fair, gradually changing to poor.

5. PLANS FOR THE NEXT PERIOD

On March 1, 1966, a technical meeting was held with Messrs. H. Schwartz, D. Soltis and P. Pomerantz of NASA, and Messrs. M. Klein and E. Findl of EOS in attendance. The discussions included a review of past test results and conclusions to be drawn from some plus tests and test methods to uncover the primary mode of cycle life failure. In addition, a priority status was given to each test. The new tests are as follows.

First Priority Tests

1. Assemble single cells and operate same as an O_2 concentration cell at $\approx$ 150 PSIG in a continuous mode at 100 ma/cm^2. Parameters to be evaluated are:

   (a) effect of charge and discharge on cell performance

   (b) Pt migration into matrix.

   This test is to be conducted to eliminate high voltage i.e., $>$ 1.5 volts, effects on the matrix and to see if long term performance can be achieved if cycling is eliminated.

2. Assemble single cells and operate same as an O_2 concentration cell at $\approx$ 150 PSIG in a cyclic mode at 100 ma/cm^2. Parameters to be evaluated are:

   (a) effect of cyclic operation

   (b) Pt migration into matrix.

   This test complements Test 1 to determine if it is low voltage cycling that causes degradation.

3. Set up asbestos quality control procedure. Investigated, (a) gas porosity of the dry mat, and (b) weight.

4. Pretreat electrolyte to eliminate any possible contaminants. Two procedures are to be employed, namely, triple distillation of the water solvent and pre-electrolysis of the KOH electrolyte.

Second Priority Tests

5. Study possible changes in Pt catalyst characteristics by (a) measurement of electrode surface area before and after cycling; and (b) measure crystalographic properties of catalyst before and after cycling.

6. Evaluate matrices other than fuel cell grade asbestos. Initial suggested materials included potassium titanate paper, acid leached fuel cell grade asbestos and non-reactive porous plastics.

7. Evaluate the chemical purity of the fuel cell grade matrix by electrolyzing an asbestos pulp in pre-purified electrolyte after a 24-hour soak at $\approx 80^\circ C$. The electrolyzed asbestos pulp is to be reconstituted and formed into a mat for testing in a single cell.

8. Evaluate the performance of Chem Cell Corp. electrodes in single cells.

Third Priority Tests

9. Evaluate cell performance using optimum electrodes, optimum mat and high purity pre-electrolyzed electrolyte.

10. Set up a single cell using two multicell end plates plus a 6-cell bellows in the six-cell tankage to eliminate ΔP effects.

Certain of these tests have been completed and reported herein. Others are currently being conducted. Representatives of American Cyanamid have agreed to study samples of their AB-6 electrodes that have degraded in our usage to determine the mode of failure. Preliminary results from American Cyanamid indicate degradation is due to a physical coating on the platinum black. Item 9 above has been dropped due to the high cost of the gold screen electrode and item 11 has been revised to be run in a single cell rather than a 6-cell test assembly.

During the next period, efforts will continue to define the primary mode of degradation. Tests will be continued as defined in the revised test plan.

6. FINANCIAL STATEMENT

Man hours and dollar expenditure for the period 4 March 1966 through April 1966 were as follows:

Direct Labor Hours	865
Direct Labor Dollars	$3,623.02
Purchases and Commitments	$2,281.59
Total Dollar Expenditure	$12,416.23

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