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ANNUAL REPORT FOR 1981 ON RESEARCH, DEVELOPMENT, AND DEMONSTRATION OF NICKEL-IRON BATTERIES FOR ELECTRIC-VEHICLE PROPULSION

Contract No. 31-109-38-4292

by

Eagle-Picher Industries, Inc.

MASTER

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Annual Report for 1981 on

RESEARCH, DEVELOPMENT, AND DEMONSTRATION OF NICKEL-IRON BATTERIES FOR ELECTRIC-VEHICLE PROPULSION

Prepared for

The Office for Electrochemical Project Management
Argonne National Laboratory
Under Contract No. 31-109-38-4292

by

Eagle-Picher Industries
Joplin, Missouri

March 1982

ANNUAL TECHNICAL STATUS REPORT
Report No. 4
Fiscal Year 1981

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APPROVAL PAGE

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1.0 INTRODUCTION AND OVERVIEW

The objective of the Eagle-Picher Nickel-Iron battery program is to develop a Nickel-Iron battery system suitable for use in the propulsion of electric and electric/hybrid vehicles. The Near-Term Electric Vehicle Battery Development goals as set forth by the defined Statement of Work are as follows:

PROGRAM GOALS

Battery Capacity	-	25-30 KWH
Specific Energy (C/3 Rate)	-	60 WH/Kg
Specific Power - Peak	-	100 W/Kg
- Sustained	-	20 W/Kg
Duty Cycle - Discharge	-	2-4 Hours
- Charge	-	4-8 Hours
Energy Efficiency	-	$>60\%$
Cost	-	$70/KWH (1977)
Cycle Life (80% DOD)	-	2,000 Cycles

The program as conducted in FY-1981 continued to show marked progress in reaching the above referenced goals. The FY-1981 program concentrated upon the fabrication, characterization and testing of the required electrodes together with the assembly and testing of full-scale cells and 6 volt (270 AH) modules. The FY-1981 program was structured to advance the technical aspects of the Nickel-Iron program while simultaneously reducing its potential future cost in both the materials and process areas. Initial full-size electrodes reached 2,300 cycles, full-scale, 270 AH cells exceeded 800 cycles, and five-cell, 6 volt modules reached 725 cycles during the reporting period. All tests are presently on-going. Based on the fade rate experienced to date, lifetime of the initial electrodes is expected to be 2,500 cycles.

During the period efforts focused on the development of suitable 2.4 mm single-pass plaque. At the end of the reporting period, satisfactory 2.0 mm plaque had been achieved and demonstrated. A 6 volt module has been fabricated and placed under test at the National Battery Test Laboratory. This module has accumulated fifty (50) cycles operating in the range of 45 WH/Kg like the other modules on test.

Development efforts were initiated in the areas of single-point watering systems and flame arrestor systems from a total full-scale battery standpoint. Currently, a single-point watering system has been demonstrated successfully at the 6 volt module level. Work was in progress toward the development of a fault-free flame arrestor system at the end of FY-1981.

Temperature tests were completed during the period ranging from +60^{o}C to -15^{o}C. The only effect seen was a maximum of 10% capacity loss at the -15^{o}C temperature.

Overall, marked progress was demonstrated both in the area of technical achievement and potential cost reduction of the system. A solid base has been firmly established from which to evolve the required battery system.

2.0 INVESTIGATIONS

Several investigations were conducted during the report period pursuant to further refinement of the Nickel-Iron battery technology and improvement in the cost effectiveness of battery manufacturing operations. Some equipment was refurbished and modified (Section 2.2) to implement technological advances stemming from these investigations. New equipment and fixturing were also developed and/or acquired as necessary to foster continued technological progress. Positive nickel electrode research studies (Section 2.3) were conducted contributing to the overall development effort. Similarly, dry sinter development (Section 2.4), cell experimentation (Section 2.5), battery development (Section 2.6), and performance testing of cells and modules (Section 2.7) continued in FY-1981. Some improvement in production capability (Section 2.1) was realized as a result of technological advance during the reporting period.

2.1 Production Capacity Development

The experiments, tests and other battery development effort discussed herein are presented for report purposes as an integral part of the Nickel-Iron battery development program underway at the Eagle-Picher plant in Joplin. Specifically, this prototype program coordinates the development efforts of three facilities under the direction of management at Joplin. The Swedish National Development Company (SU) has contracted to select a suitable separator, fabricate iron negative electrodes and conduct cell and battery characterization experimentation. Eagle-Picher - Colorado Springs supplies nickel sinter plaques which are impregnated with nickel hydroxide at the Joplin facility completing fabrication of positive nickel electrodes to be incorporated (also at the Joplin plant) into cells and batteries. Eagle-Picher now has an increased overall manufacturing capacity stemming from technological progress made during FY-1981. The existing facilities have a demonstrated capacity for sintering of over four thousand (4,000) nickel plaques per month; significantly up from the monthly production rate of some two thousand (2,000) plaques reported last year. Monthly capability for iron electrode fabrication remains at four thousand (4,000) negative plates. Nickel plate impregnation and testing facilities are now able to sustain the manufacture of twenty-four (24) eighty-cell batteries a year; up from the twelve (12) battery capacity reported last year. These facilities can be readily modified and augmented to meet the demands of any forthcoming pre-pilot program.

2.2 Equipment Modification

Extensive refurbishing and repair of the sintering furnace (Figure 1) was accomplished during the report period. Major repair of the reducing gas generator, the sintering furnace muffle and the heating elements was completed. Standard maintenance and repair of the thick plaque pulling tower (Figure 2) the drying cabinets (Figure 3) and the electronic controls of the sintering furnace was also accomplished. The performance of this electrode manufacturing equipment during post-repair operations matched or exceeded its pre-repair capability. Heat profile characteristics, exhibited by the sintering furnace were equivalent to pre-repair parameters. Tests further indicated tighter temperature control, including a significant reduction of response time required to effect intrafurnace temperature change response to temperature controls. Improvement was also observed in

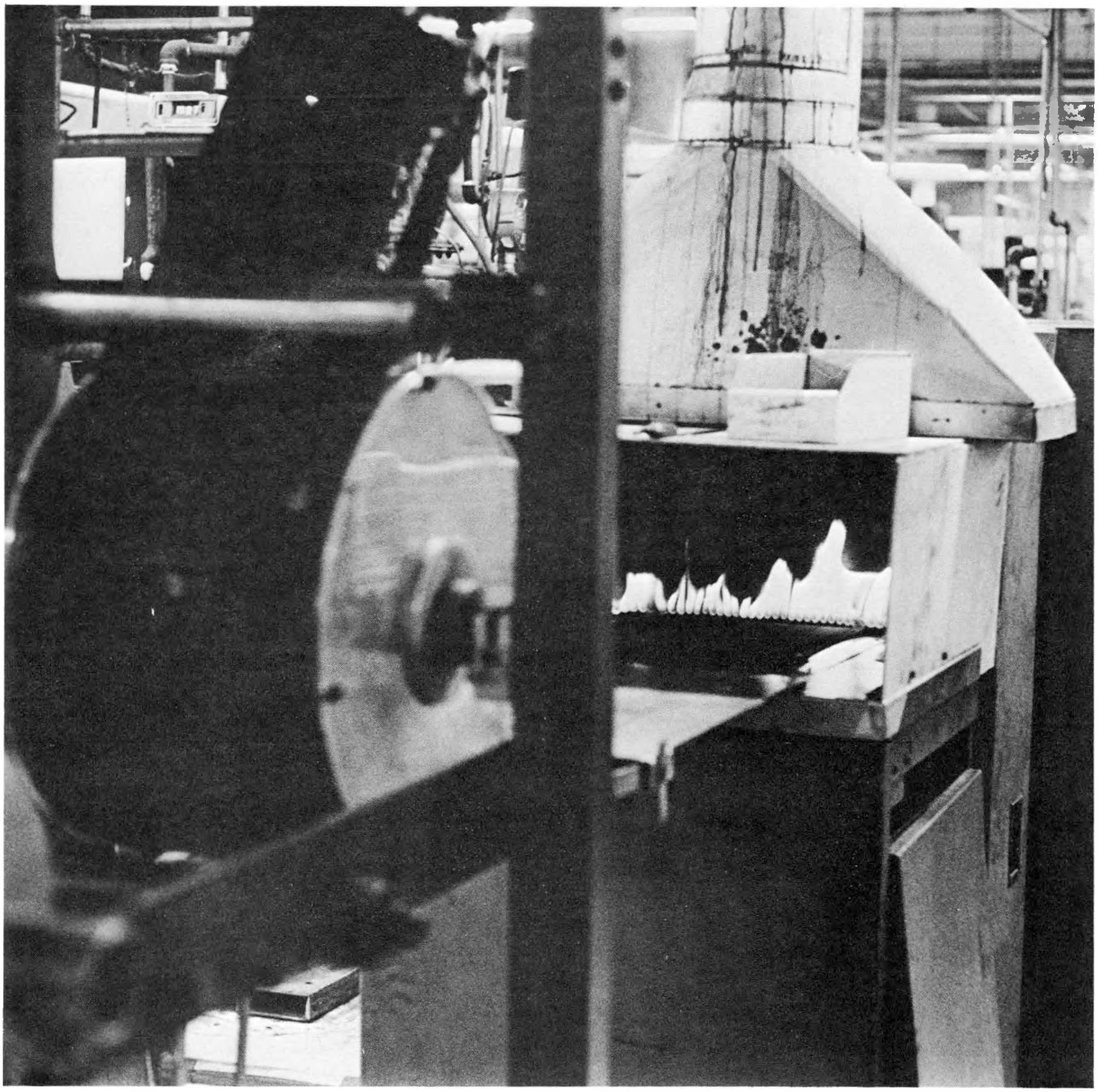


FIGURE 1
Slurry Sintering Furnace

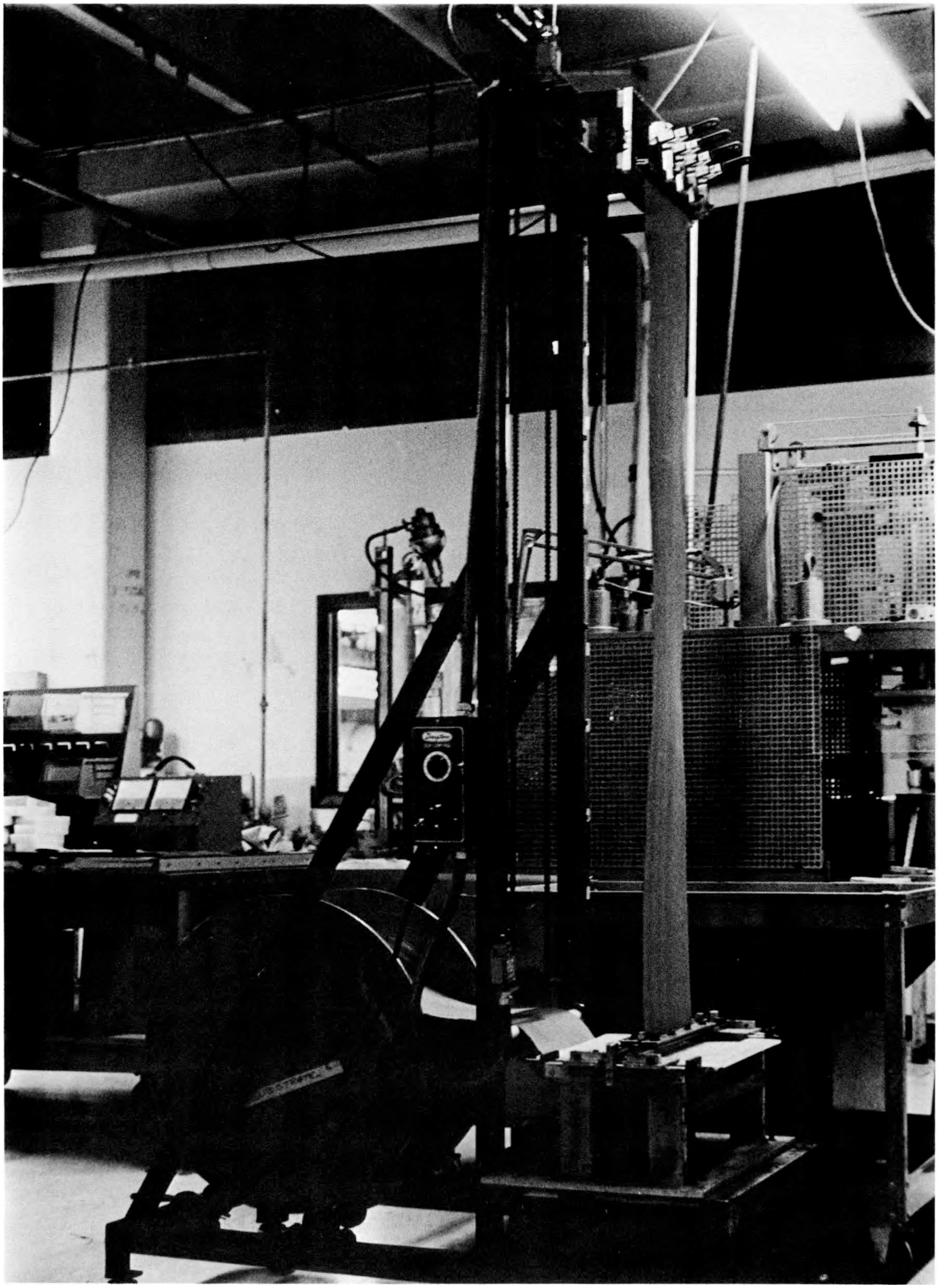


FIGURE 2
Experimental Pulling Tower

FIGURE 3
Drying Cabinet

the reducing atmosphere flow. Repair of the furnace muffle reduced atmosphere controls restored intrafurnace atmosphere conditions to normal. There was no discernable differences in sintered plaque produced before and after renovation of the sintering furnace.

In preparation for resumption of electrode production, clamping fixtures and additional drying equipment were assembled. Additional heating banks were added to the continuous production drying tower. The geometric configuration of this equipment was altered to produce higher temperatures. A proportional band zone temperature control system was developed to be utilized in maintaining control of plaque drying conditions. A new coining die (Figure 4) was installed and test equipment was adjusted to trim plaque in preparation for production. A die was fabricated to cut to length and punch holes in the nickel 200 coil for use as tabs in forthcoming runs of experimental nickel-iron electrodes. A new tab welding configuration was developed seeking to achieve the largest cross-sectional weld area commensurate with the power rating of an in-house 50 KW spot welder. Finally, the new slurry box (Figure 5) was fitted with a geometrical centering device to facilitate more rapid changeover of substrate type. This new slurry box configuration was successfully employed to fabricate several lots of experimental plaque demonstrating its effectiveness in the correction of centering problems.

2.3 Positive Nickel Electrode Research

Work proceeded to advance the technology for the manufacture of thick (2.0 mm) single-pass plaques. Refinements were made in slurry formulation technique (Paragraph 2.3.1). Plaque pulling and slurry drying experimentation was conducted (Paragraph 2.3.2). The substrate development effort continued (Paragraph 2.3.3). Progress was made in the minimization of scrap loss associated with the fabrication of nickel plaque (Paragraph 2.3.4). A preliminary study of reducing atmosphere within the sintering furnace was completed (Paragraph 2.3.5). Impregnation of experimental plaque continued (Paragraph 2.3.6). Finally, electrodes impregnated in a solution containing didymium were cycle tested to determine the impact of didymium upon capacity (Paragraph 2.3.7).

2.3.1 Slurry Preparation Improvement

Roll blending with barrels (Figure 6) adopted during the latter part of the previous fiscal year in place of the "V" blending technique continued to provide an adequate amount of slurry to support the current demand for sintered nickel plaque. Doubling of the present slurry capacity of one hundred (100) gallons per day, by the addition of identical modular units to the roll blending configuration, remains a viable option. The roll blending equipment, featuring direct drive and variable speed drive systems, enhanced control, optimized the blending speed and allowed flexibility of slurry production during FY 1981. The roll blending procedure and equipment were modified to accommodate the manufacture of single pass slurry plaque.

2.3.2 Plaque Pulling and Slurry Drying Investigation

Plaque pulling experimentation utilizing the experimental

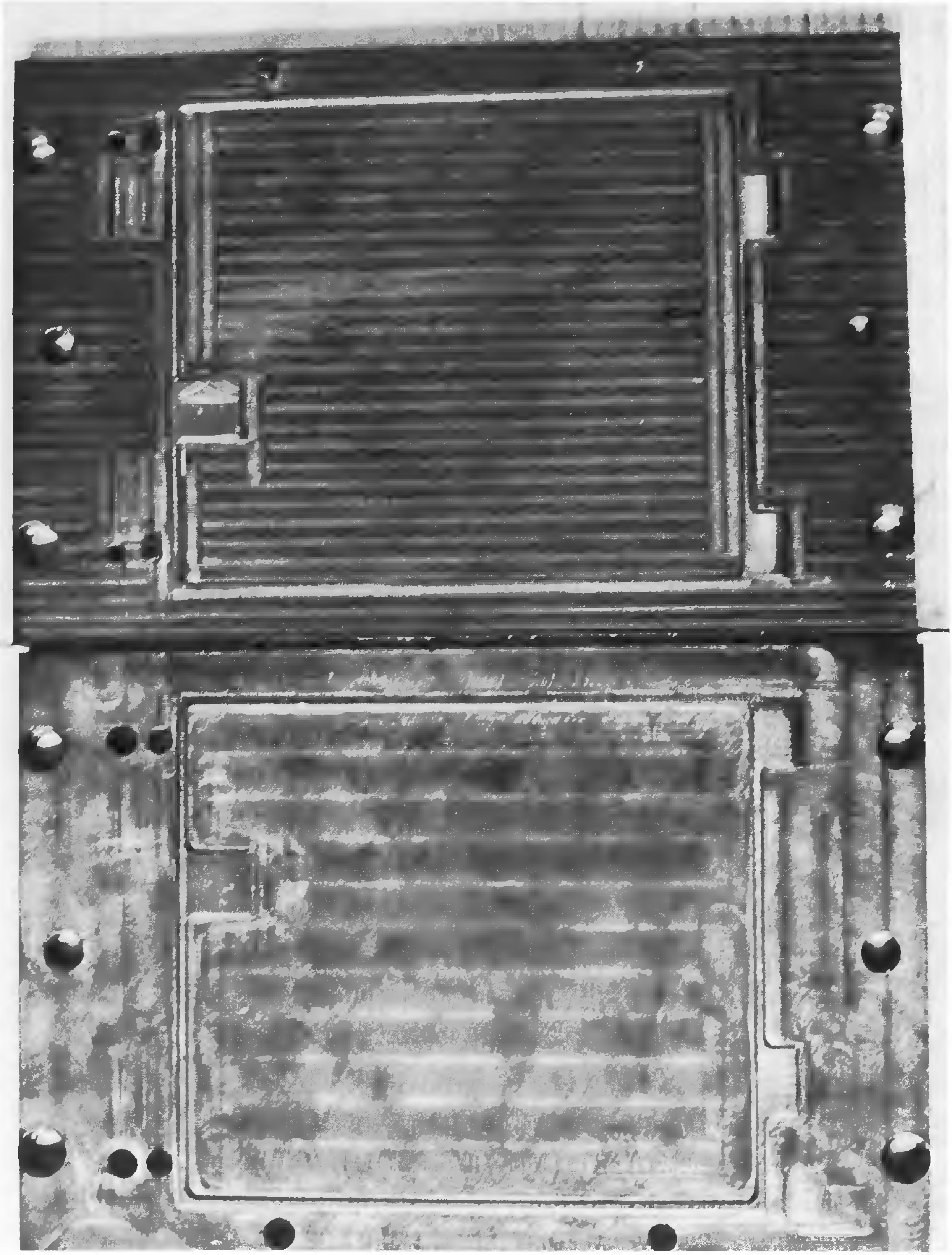


FIGURE 4
Coin Dies

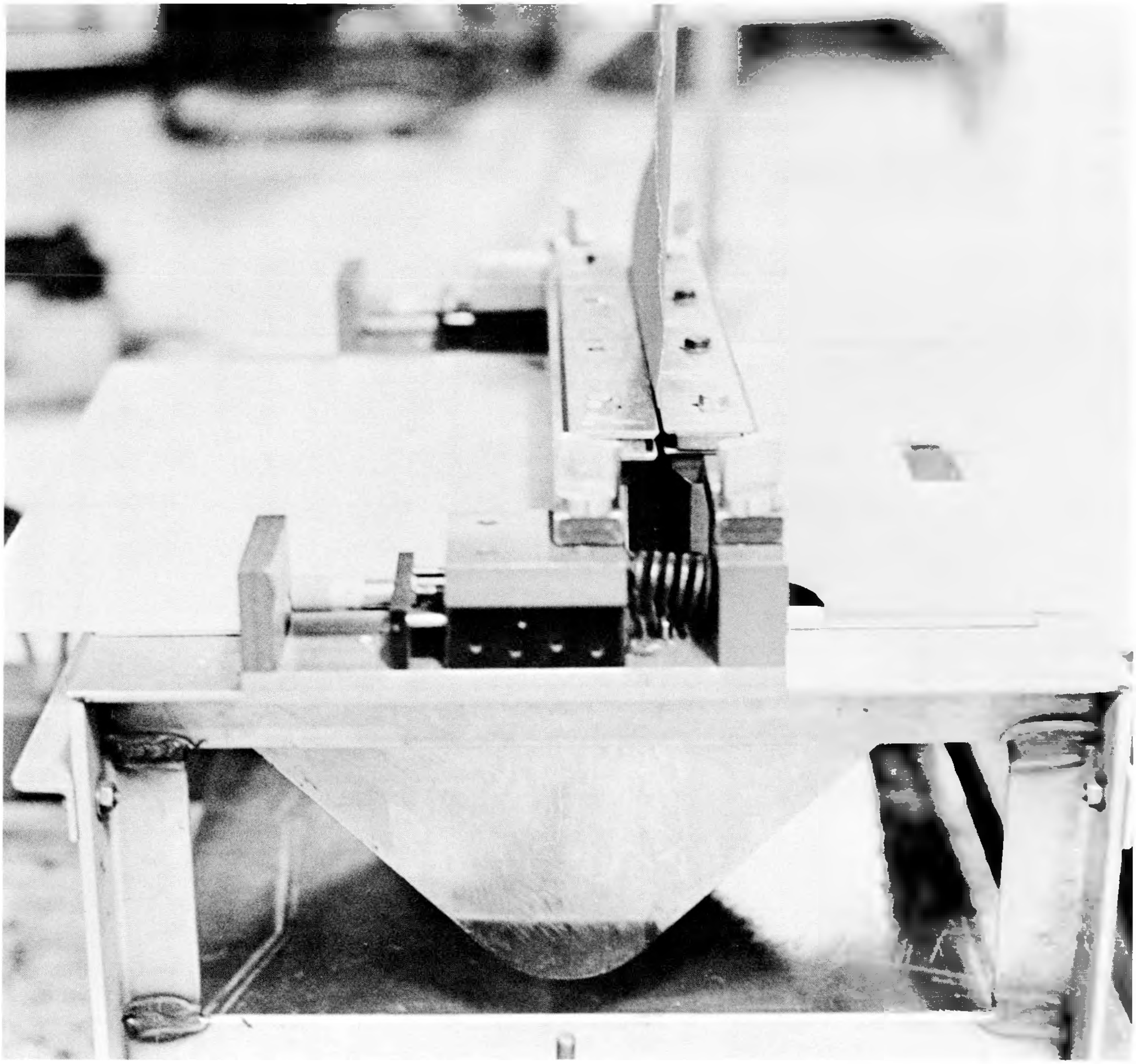


FIGURE 5
New Slurry Box with Doctor Blades

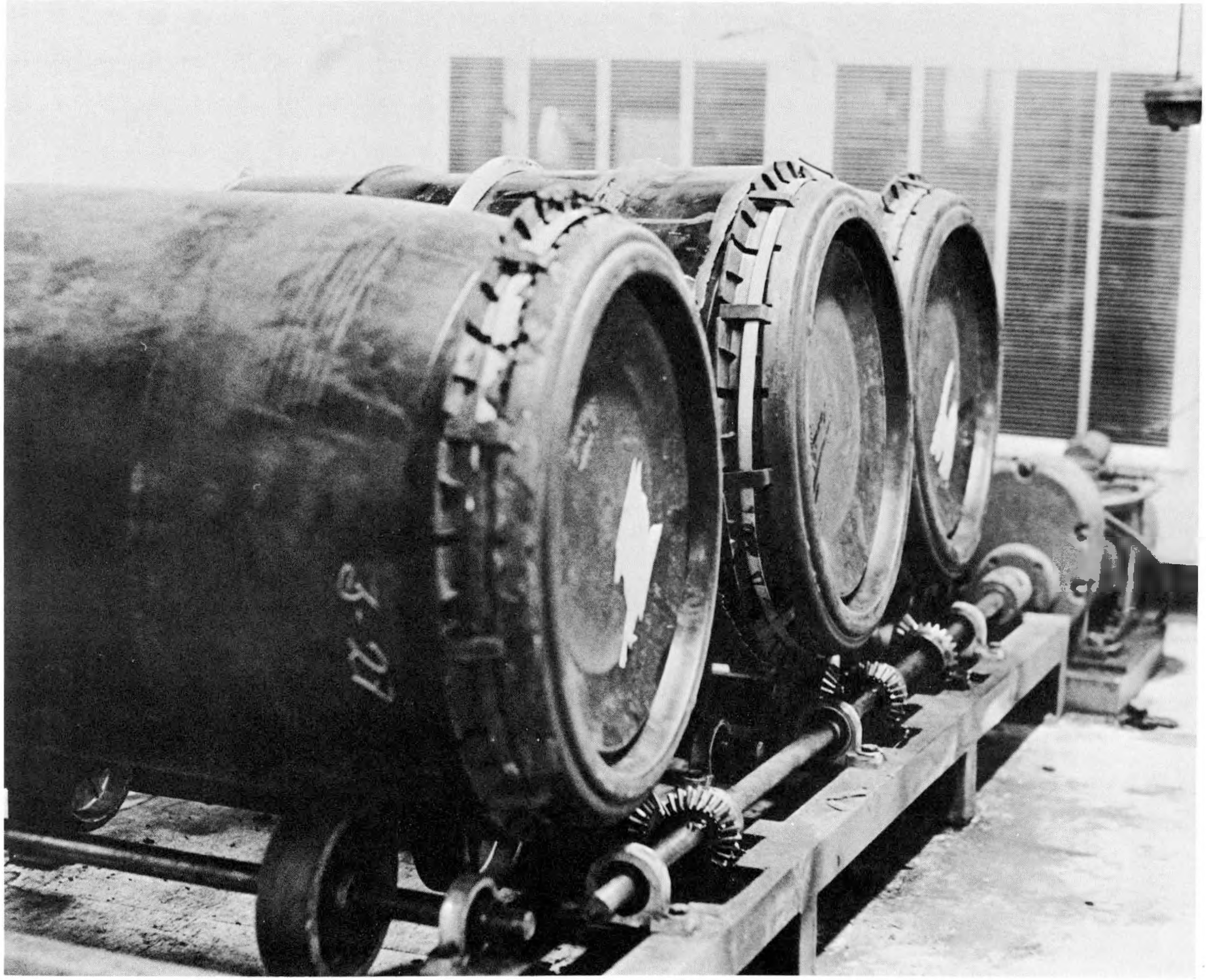


FIGURE 6
Roll Blending Apparatus

pulling tower (Figure 2) was conducted as a part of the continuing development effort to refine further the procedure for the fabrication of single-pass plaque. Pulling speed, "doctor" blade spacing and centering adjustment were the principal variables included in this inquiry. The results of this study were still being evaluated at the end of the reporting period. Effort was also directed toward more rapid slurry drying. The slurry which is used with the double pass process requires controlled drying over a 24 to 48 hour period. The water base experimental slurry for single-pass plaque can be dried in 6 hours with forced warm air ventilation. The plaque maintains the required smooth surface appearance without mud cracking. Sintering these strips is routine with porosity being increased slightly and the bend strength maintained in the 1,500 psi range.

2.3.3 Substrate Inquiry

Current-carrying, substrate development concentrated upon the optimization of variables. These factors included the weight, strength, conductivity and cost of the substrate. Other important properties considered were sinter-substrate adhesion, quality of the final plates and ratios of "inert" nickel to theoretical capacity. Preliminary assessment of the three main types of available metal substrates was made. Woven wires remained a viable substrate option provided the costs of cleaning and calendering could be kept within bounds. The handling ease, thinner configurations, lighter weight and cost parity of perforated sheet combine to make it an acceptable alternative substrate material. A configuration of expanded metal substrate (3 nickel 10-4/0) was shown to offer the greatest degree of flexibility among expanded metal options along with high material quality and reasonable price. The goal of this effort remained the development of the best electrode possible for each substrate type and ultimate selection of a single substrate based on overall performance and cost considerations.

2.3.4 Scrap Minimization Project

Effort continued to reduce the fifty percent (50%) scrap loss commonly experienced in the manufacture of sintered nickel plaque. Plaque scrap recycling procedure was instituted during the previous report period with a projected eighty percent (80%) reduction of scrap loss. Reconstituted slurry, produced in the recycling of unsintered plaque scrap, was employed to fabricate experimental thin plaque during FY-1981. These plates were then impregnated and subjected to life cycle testing to document the merits of the utilization of reconstituted slurry in the manufacture of positive nickel electrodes. The tests showed no significant difference in electrode quality and performance between these standard electrodes. Thick electrodes were also fabricated and were undergoing extensive mechanical testing at the end of the reporting period.

2.3.5 Reducing Atmosphere Study

Preliminary investigation of total reducing atmosphere turnover rates was initiated seeking to develop more data on the mechanisms of plaque strength augmentation and sinter densification. Inquiry was also made into the types and levels of oxide buildup within the sintering furnace and on the belt in the sintering operation. This initial investigation indicated the need for a more thorough study of the thermodynamics of the oxidation reduction processes. It was also apparent that such a study will require more accurate gas analysis equipment and better facilities for analyzing various nickel, chromium and iron oxides.

2.3.6 Experimental Plaque Impregnation

Impregnation of experimental plates continued throughout FY-1981 utilizing the new experimental impregnation tank (Figure 8) having a per run capacity of eight-plates vs. the previous four plate capability. This reduced the processing time lag from about three (3) days to two (2) days. Refinement of plaque fabrication technology and the upgrading of equipment described herein were complemented by improvements made during the year in impregnation and formation technology as set forth in Figure 7. Experimental plaques, fabricated in connection with the overall nickel electrode development effort were impregnated with nickel hydroxide achieving the results displayed in Figure 9.

Figure 7

	FY-1979	FY-1980	Present Status
Impregnation Time	Standard	Standard	Standard
Impregnation Tank Capacity	30 Plates	40 Plates	75 Plates
AH Deposit Before Rejuvenation	3,468 AH	6,069 AH	6,242 AH
Formation Time	54 Hours	46 Hours	46 Hours

2.3.7 Didymium Additive Study

Technical literature suggested a capacity increase of approximately 100% could result from the use of didymium in the impregnation solution. Three plates were impregnated in a nickel nitrate solution containing 5 mole % rare earths nitrates (didymium) seeking to achieve a significant increase in capacity. Two of these plates were then cycled against nickel counters and the other plate was cycled against iron electrodes. None of these plates registered the approximate 100% capacity gain reported in technical literature. Instead the exhibited capacities of these experimental plates were similar to capacities recorded for plates impregnated in a nickel nitrate solution containing 5 mole % cobalt nitrate.

2.4 Dry Sinter Development

A limited dry sinter electrode development program was also continued during the report period. This effort employed the dry sinter forming jig and its associated apparatus (Figure 10) to gain a better understanding of the variables involved in this dry powder technology. Pore former additives were studied in an effort to modify pore size and porosity values in the resulting sintered nickel plaque. Dry sinter plates continued to achieve on the average greater loadings when impregnated with the nickel hydroxide, than plates fabricated by the slurry application procedure.

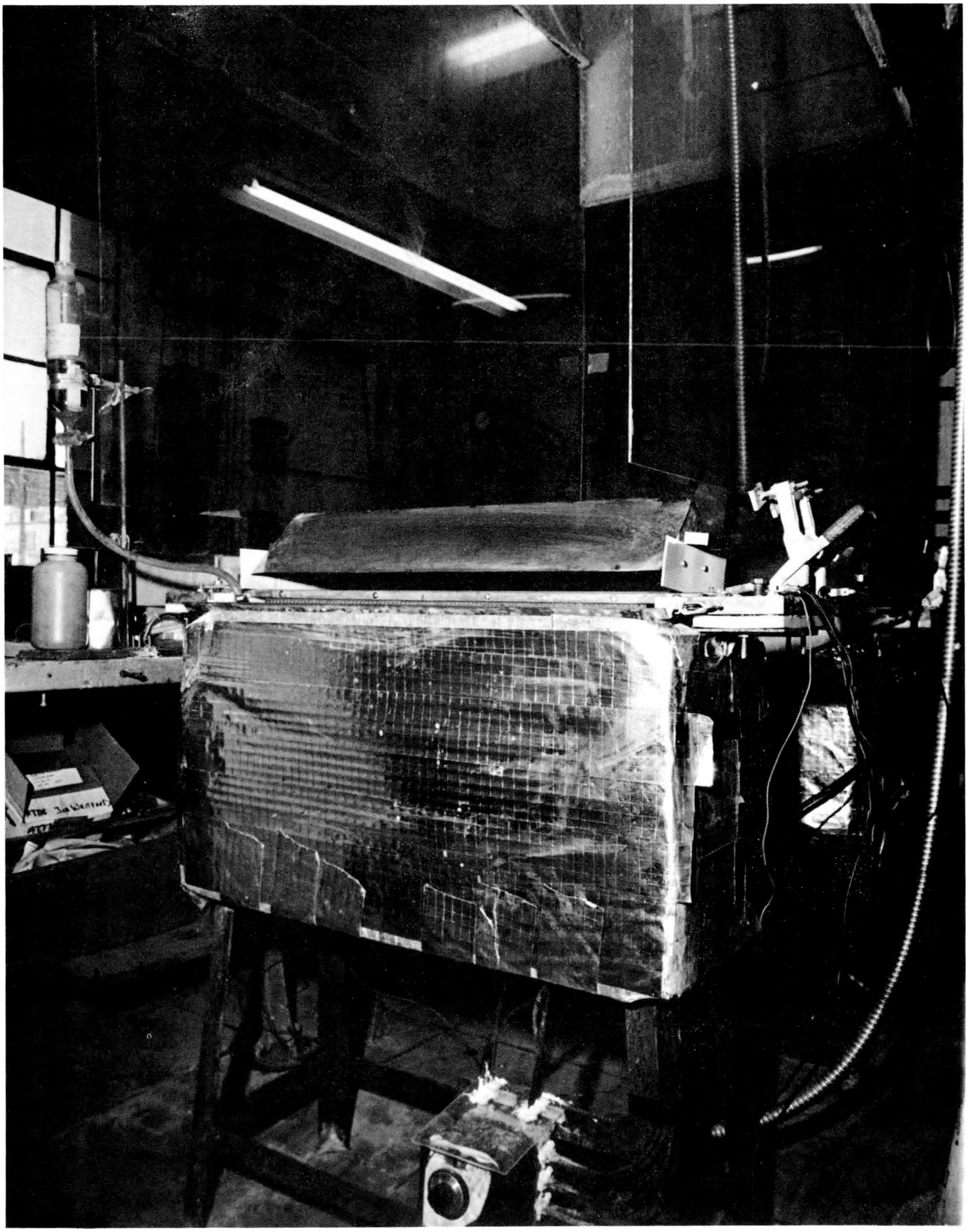


FIGURE 8
New Experimental Impregnation Tank

Figure 9

Experimental Plaque Impregnation Results
Single Pass Slurry Plates 165 mm X 190 mm H

Plate		Description			Loading		Results: Average Theoretical Capacity		
Ident. No.	Quan.	Grid	Porosity %	Thickness Ave. mm.	g, ave	g/cc	AH/gm	AH/cc	AH/plate
X-200	8	Screen	79.3	2.27	106.7	1.50	0.127	.434	30.8
X-207A	8	Ex	79.3	1.75	92.7	1.69	0.136	.488	26.8
X-207C, D	4	Screen	78.4	2.00	111.0	1.77	0.137	.512	32.1
X-208*	4	Screen	80.6	2.33	115.6	1.58	0.135	.457	33.4
X-209A	8	Screen	81.5	2.11	111.3	1.68	0.143	.486	32.2
X-209B	4	Ex (Flat)	81.7	2.52	128.9	1.63	0.141	.471	37.3
X-210A	4	Screen	81.7	2.17	115.7	1.70	0.144	.491	33.4
X-210B	4	Ex	81.2	2.5	104.2	1.33	0.127	.384	30.1
X-219	20	Screen	81.0	2.51	129.0	1.64	0.139	.474	37.3
X-221*	8	Screen	79.7	2.69	142.5	1.69	0.138	.488	41.2
X-91005A	10	Ex	80.8	1.88	96.7	1.64	0.138	.474	27.9
X-91005B**	8	Ex	80.9	1.96	103.2	1.68	0.140	.486	29.8
X-91005C	10	Ex	80.9	1.86	95.0	1.63	0.138	.471	27.5
X-91005D	12	Foil	81.9	1.96	105.2	1.74	0.145	.503	30.4
X-91005E	12	Foil	81.6	1.86	99.5	1.71	0.144	.494	28.8
X-91007	80	Ex	79.9	2.08	97.8	1.50	0.132	.434	28.3

* These plates grew during formation, indicating soft sinter.

"Screen" - Denotes 0.007" x 20 x 20 woven nickel wire 0.034 gm/cm^2.

% Porosity is apparent porosity with grid excluded.

"Ex" Grid is expanded nickel 0.003" base 0.036 gm/cm^2.

"Foil" is 0.0028" thick perforated nickel foil.

Note that average 2.0 mm plaque loads to 1.52 gm/cm^3 or .429 AH/cm^3 and 0.115 AH/gm.

** Two of these plates blistered.

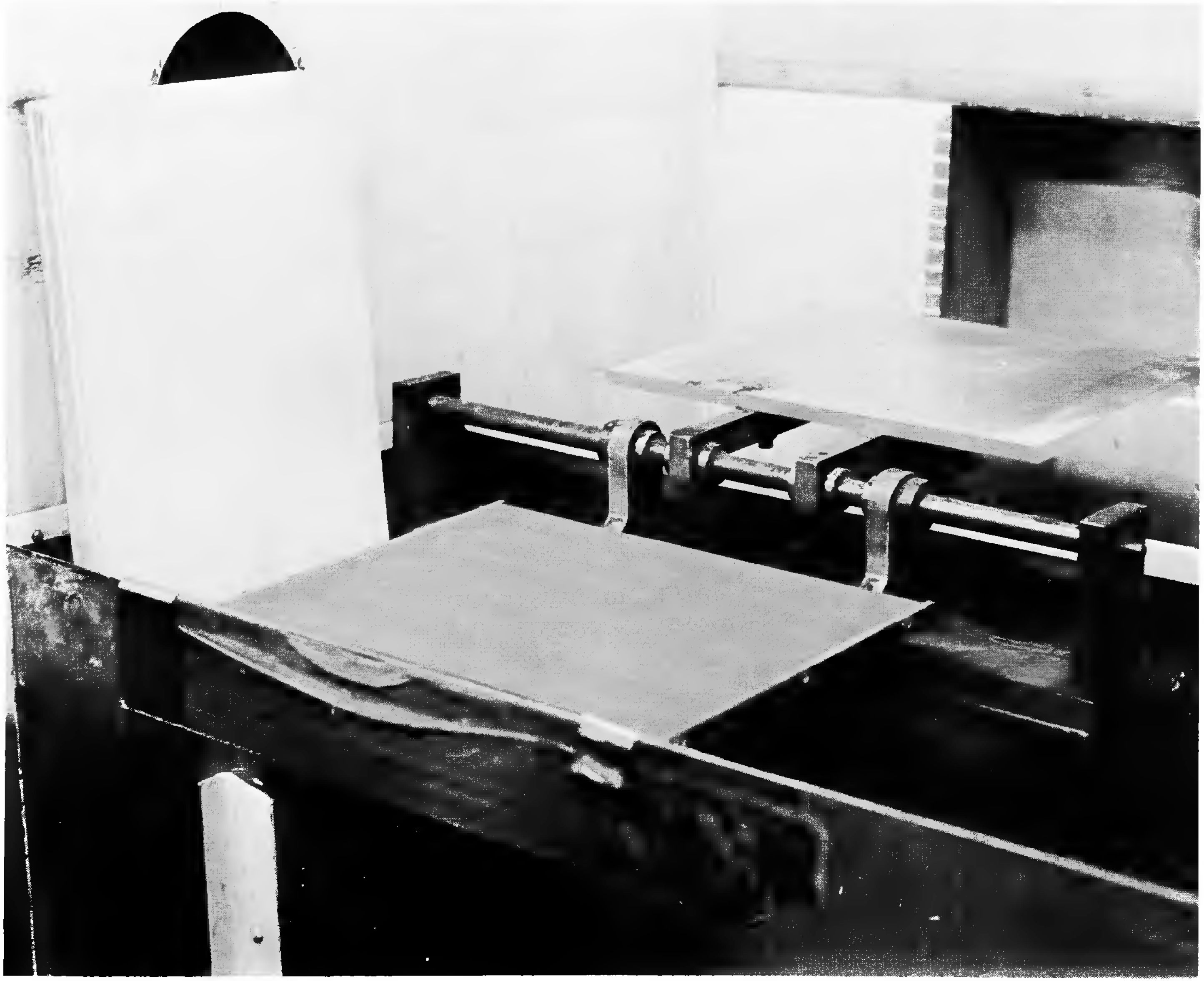


FIGURE 10
Dry Sinter Forming Jig

More study was needed at the end of the reporting period to account for the observed differential loading capabilities of dry sinter plaque and plaque fabricated using slurry-sinter technology. Dry sinter fabrication remained a labor intensive process involving painstaking centering of substrate (grid) between two meticulously formed nickel powder layers, with thickness up to 1.2 mm, to produce an electrode 2.4 mm in thickness. Continued development effort was planned for the next reporting period but it was apparent that significant advanced in this technology would require extensive design work.

2.5 Cell Experimentation

Several experiments involving cells were conducted in FY-1981. Short-term cell experiments, discussed below, included the pressure testing (Paragraph 2.5.1) of cell case and cover (Figure 11), an electrolyte maintenance inquiry (Paragraph 2.5.2), and thermal tests involving Cells 034, 528-30, and 1397-99 (Paragraphs 2.5.3 and 2.5.4). Long term experimental cell testing progressed to study the effects of sulfide electrolyte additive (Paragraph 2.5.5) and the performance of Jungfer separators (Paragraph 2.5.6). Some experimental cells were fabricated to examine aspects of cell construction. Cell CX 219, having a theoretical capacity of 413.1 AH, was assembled using 2.4 mm double-pass positive electrodes. Cells SX-81A and SX-81B were constructed exhibiting initial respective capacities of 270 AH and 267 AH. Cell X91005A was fabricated using three (3) X91005A experimental plates and four (4) iron electrodes, and demonstrated an average capacity of 75.6 when charged to 120% of its theoretical capacity, 86.0 AH. The increased understanding of Nickel Iron technology, derived from the investigations outlined above, was used to advance the state-of-the-art in construction of Nickel-Iron Cells.

2.5.1 Cell Case Pressure Test

Cell case stress testing was initiated to determine whether electrolyte leaks, observed in several cells around the P.S.-18 glue joints between cell cases and covers, were attributable to excessive pressure. Prior to application of P.S.-18 glue, subassemblies of two complete cell cases were cleaned with detergent cleaner while the parts for two other cell containers were cleaned with methanol. After cell covers were securely sealed with P.S.-18, the entire cell case assemblies were subjected to pressurization-pressure release cycling. The cell case assemblies were thus pressure cycled in incremental steps up to 25 psi. After completion of four hundred seventy-five (475) pressure cycles at 15 psi, stress marks had developed on the cell wall immediately below the P.S.-18 glue joints. All test cell case assemblies ruptured at these visible stress points within eight (8) cycles at the 25 psi level of pressure cycling. Since no cracks or leaking developed at the P.S.-18 glue joint of the pressure cycled cell cases, it was concluded that the electrolyte leaks were due to inadequate cleaning of parts before the application of P.S.-18. The procedure was modified to include thorough cleaning of cell case subassemblies with detergent prior to the gluing operations.

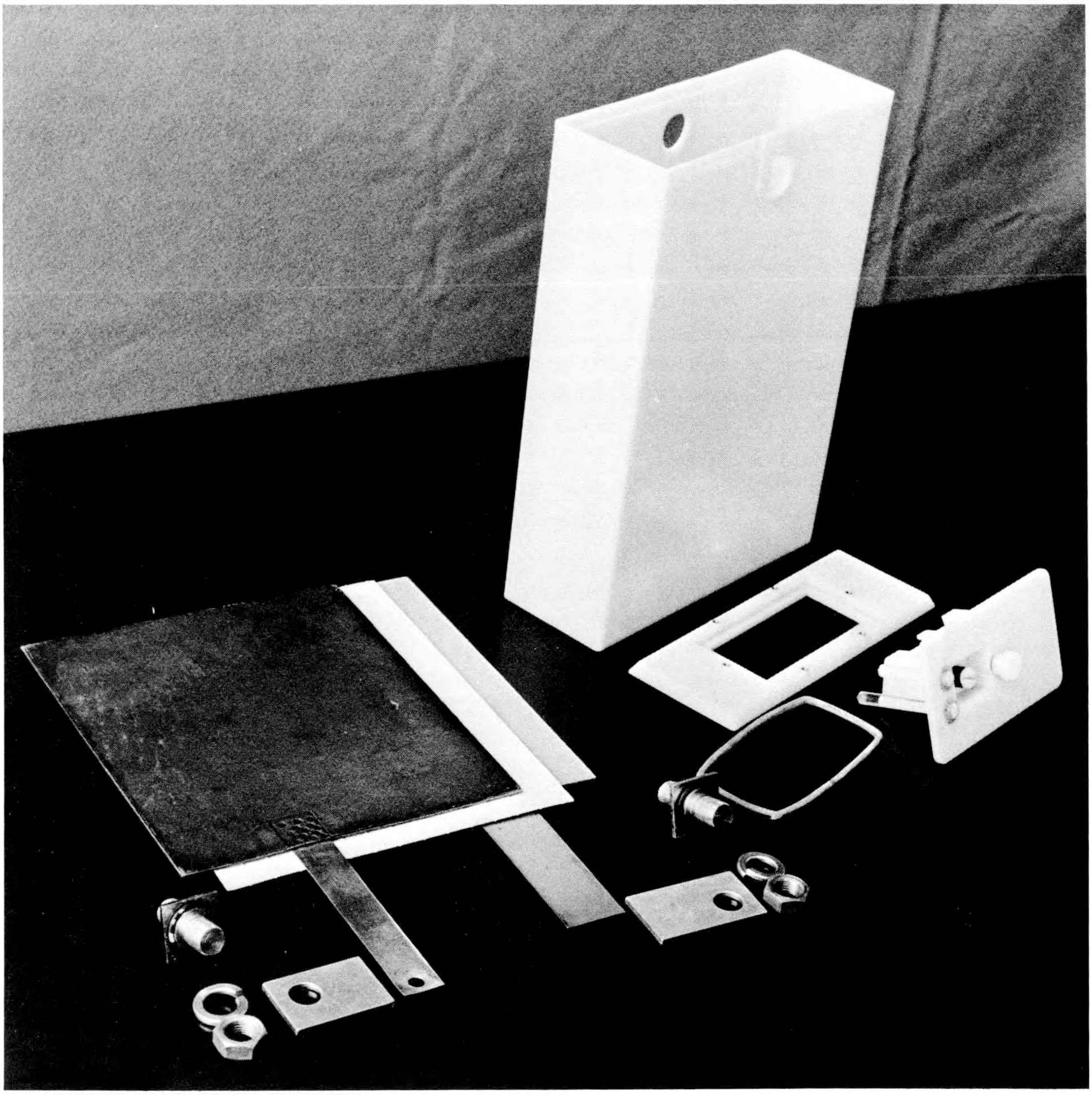


FIGURE 11
Cell Case Hardware

2.5.2 Electrolyte Maintenance Study

A study was completed of the effects of electrolyte replacement by water, a probable common error in maintenance, upon the performance of nickel-iron cells. Cell 007, with a theoretical capacity of 348.3 AH, was routinely overfilled with deionized water until the measured specific gravity of the "electrolyte" approximated that of water. While the electrolyte dilution was in progress, the average capacity of Cell 007 declined from 308 AH to 262 AH. The diluted "electrolyte" was then replaced with fresh electrolyte: 1.24 specific gravity LiOH/KOH. After the electrolyte change, the capacity of Cell 007 rose to a stable 313 AH, 54 WH/Kg at the four hour rate. Cell 007 reached the six hundred fifty-nine (659) cycle mark at the end of this study. A regimen of charging at 76.6 amps for 5 hours and discharging at 80.3 amps to (+) 1.0 volts was maintained in this period.

2.5.3 Thermal Testing: Cell 528-30

A study to assess the influence of positive plate cobalt additive upon the performance of nickel-iron cells at various temperatures from -20°C to +60°C was completed. Cell 528-30 (constructed of three double-pass nickel positive electrodes plus four iron negative electrodes and having a theoretical capacity of 83.8 AH) was selected for this test procedure. The nickel electrodes incorporated into this cell were impregnated in a nickel nitrate bath that did not contain cobalt, the standard positive electrode additive. Cell 528-30 accumulated five hundred fifty (550) cycles prior to undergoing thermal testing. An average utilization of eighty-seven percent (87%) was recorded for Cell 528-30 at the beginning of this investigation. Each test temperature was maintained within $\pm$ 3°C throughout charge and discharge. The ambient test temperatures and testing order were tabulated for inclusion in Figure 12. The charge and discharge rates for each cycle were 18.4 amps for five hours and 27.9 amps to (+) 1.0 volts, respectively.

The effect of these ambient temperatures upon the performance of Cell 528-30 is manifested in the data tabulated in Figure 12 and profiled in Figure 13. Interestingly, Cell 528-30 performed better at 30°C and 40°C than it did at 20°C; possibly a result of the absence of cobalt. A comparison was made of test data for Cell 528-30 with data obtained from temperature testing of Cell 034 with cobalt additive in its twelve (12) positive plates. Figure 14 compares Cell 528-30 test data with Cell 034 test data in terms of amp-hour charge per plate for the listed temperature changes. Results of the comparison suggested the cobalt additive to be effective in improving cold temperature performance. But the possibility remained that the superior performance of Cell 034 at lower temperatures stemmed largely from heat generated and retained in this larger cell with its greater mass during the end of charge and throughout discharge.

2.5.4 Thermal Testing: Cell 1397-99

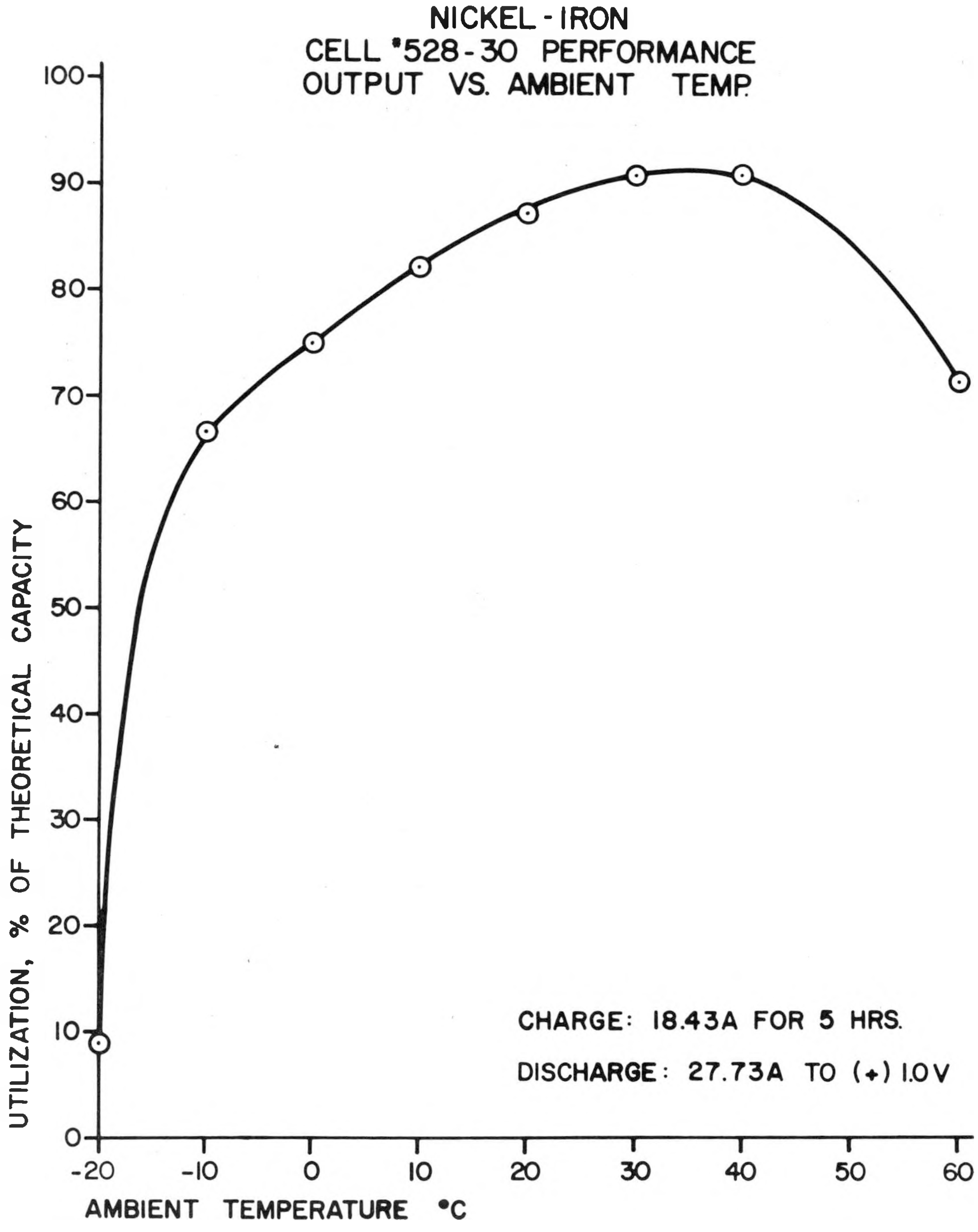
The thermal testing of Cell 1397-99 was undertaken to determine the effects of continued cycling at 60°C. Cell 1397-99 (containing three standard nickel positive electrodes with cobalt additive plus four iron negative electrodes and having a theoretical capacity of 81.3 AH)

Figure 12

TEMPERATURE TESTING OF CELL 528-30
(Theoretical Capacity: 83.8 AH)

Ambient Temp., $^{o}C \pm 3^{o}C$	Number of Cycles	Avg. Coulombic Efficiency	Avg. Utilization, % of Theo. Capacity
20	3	79.3	87.2
-10	3	60.4	66.5
20	2	78.8	86.7
0	2	68.1	74.9
20	3	77.8	85.5
-20	3	8.1	8.7
20	2	83.3	91.7
10	2	74.5	82.0
20	1	78.5	86.3
30	3	82.4	90.6
20	1	78.3	86.1
40	3	82.5	90.7
20	2	79.9	87.9
60	3	64.7	71.2
20	5	78.1	85.9

Figure 13


NICKEL - IRON
CELL #528-30 PERFORMANCE
OUTPUT VS. AMBIENT TEMP.
UTILIZATION, % OF THEORETICAL CAPACITY
100
90
80
70
60
50
40
30
20
10
0
-20
-10
0
10
20
30
40
50
60
AMBIENT TEMPERATURE °C
CHARGE: 18.43A FOR 5 HRS.
DISCHARGE: 27.73A TO (+) 1.0V

was cycled in lithiated electrolyte. This cell had accumulated three hundred seventy (370) cycles before the initiation of this thermal testing. The charge and discharge regime was 17.89 amps for five hours and 27.11 amps to (+) 1.0 volts, respectively. First, Cell 1397-99 was cycled four (4) times in an ambient temperature of $23^oC \pm 3^oC$ establishing a baseline of utilization of eighty-five percent (85%). Ambient temperature was then increased to $60^oC \pm 3^oC$ for twenty-two (22) cycles. During this cycling, cell utilization declined about fifty percent (50%) as set forth in Figure 15. Ambient cell temperature was returned to 23^oC and the previous level of cell utilization was regained after completion of fourteen (14) cycles. It was clear that continual cycling at 60^oC for a limited number of cycles does not permanently damage a nickel-iron cell of this size. Larger cells, however, could be more adversely affected by cycling at this temperature, since they may well retain heat generated at the end of charge and during discharge for a longer time. Another study is required to determine the effects of continual cycling at 60^oC upon the performance of full-scale cells or modules.

Figure 14

COMPARISON OF NICKEL IRON CELLS

Cell #528-30 (3 Positive Plates No Cobalt Addition)

And

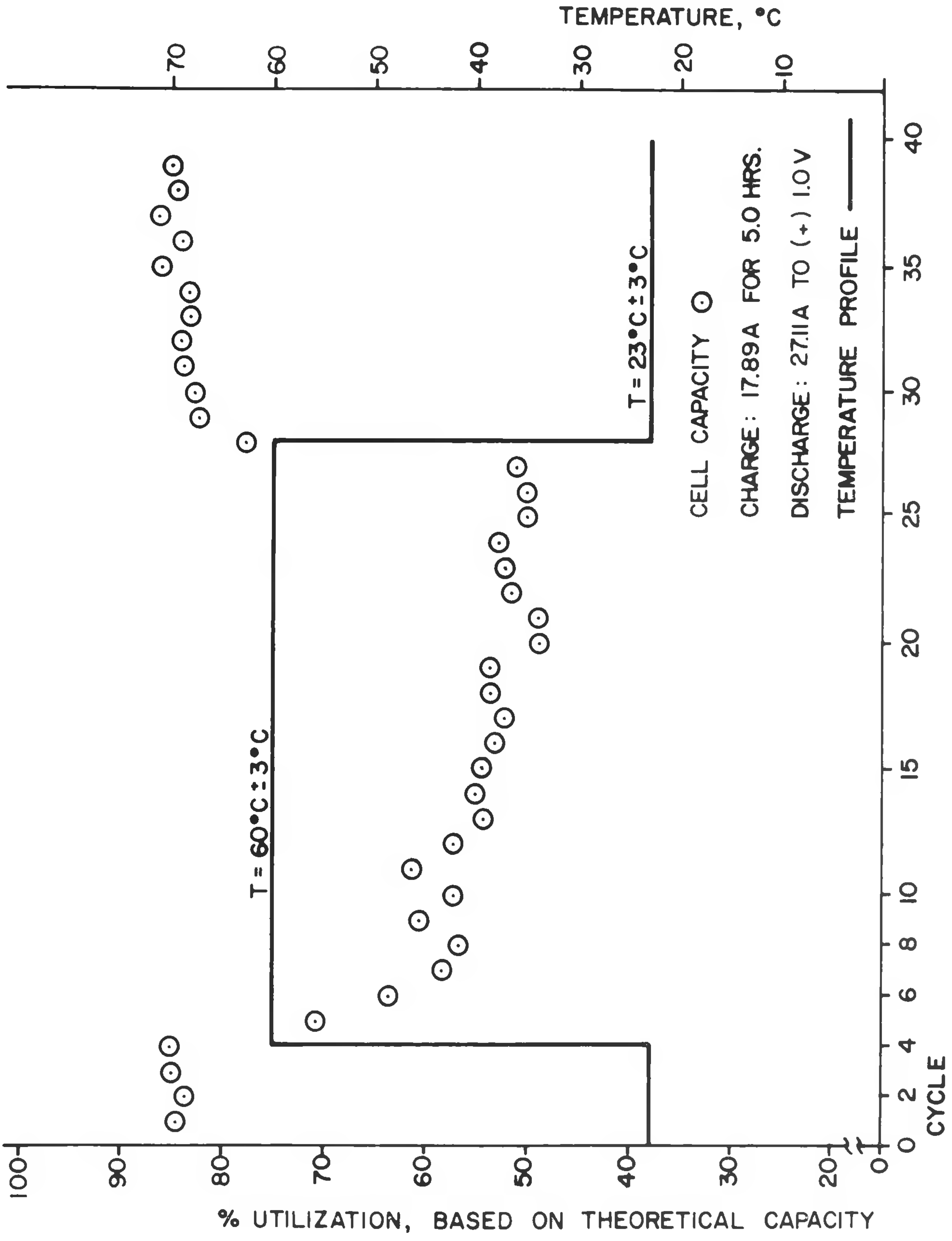
Cell #034 (12 Positive Plates With Cobalt 5%)

Temperature Change, ^oC		Cell 034 AH/plate	Cell 528-30 AH/plate
-20	-10	1.0	16.1
-10	0	1.8	2.3
0	10	0.3	2.0
10	20	0.5	1.4
20	40	-0.2	1.0

2.5.5 Sulfide Additive Inquiry

A study to continue investigation of the positive effects of sulfide electrolyte additive upon cell performance was conducted involving a test group of six (6) iron half cells. These cells were each assembled with an iron electrode sandwiched in between two (2) counter electrodes. They were then subjected to successive charge-discharge cycles at the C/3 rate exceeding one thousand four hundred (1,400) cycles (Figure 16) during FY-1981. Sulfide was initially added to the electrolyte of individual cells for a cell-by-cell observation of its impact upon capacity. Next,

FIGURE 15


TEMPERATURE, °C
% UTILIZATION, BASED ON THEORETICAL CAPACITY
CYCLE
T = 60°C ± 3°C
T = 23°C ± 3°C
CELL CAPACITY
CHARGE: 17.89A FOR 5.0 HRS.
DISCHARGE: 27.11A TO (+) 1.0V
TEMPERATURE PROFILE

LIFE CYCLE TEST

6 Iron Electrode Half Cells - E80:1

Average Specific Capacity

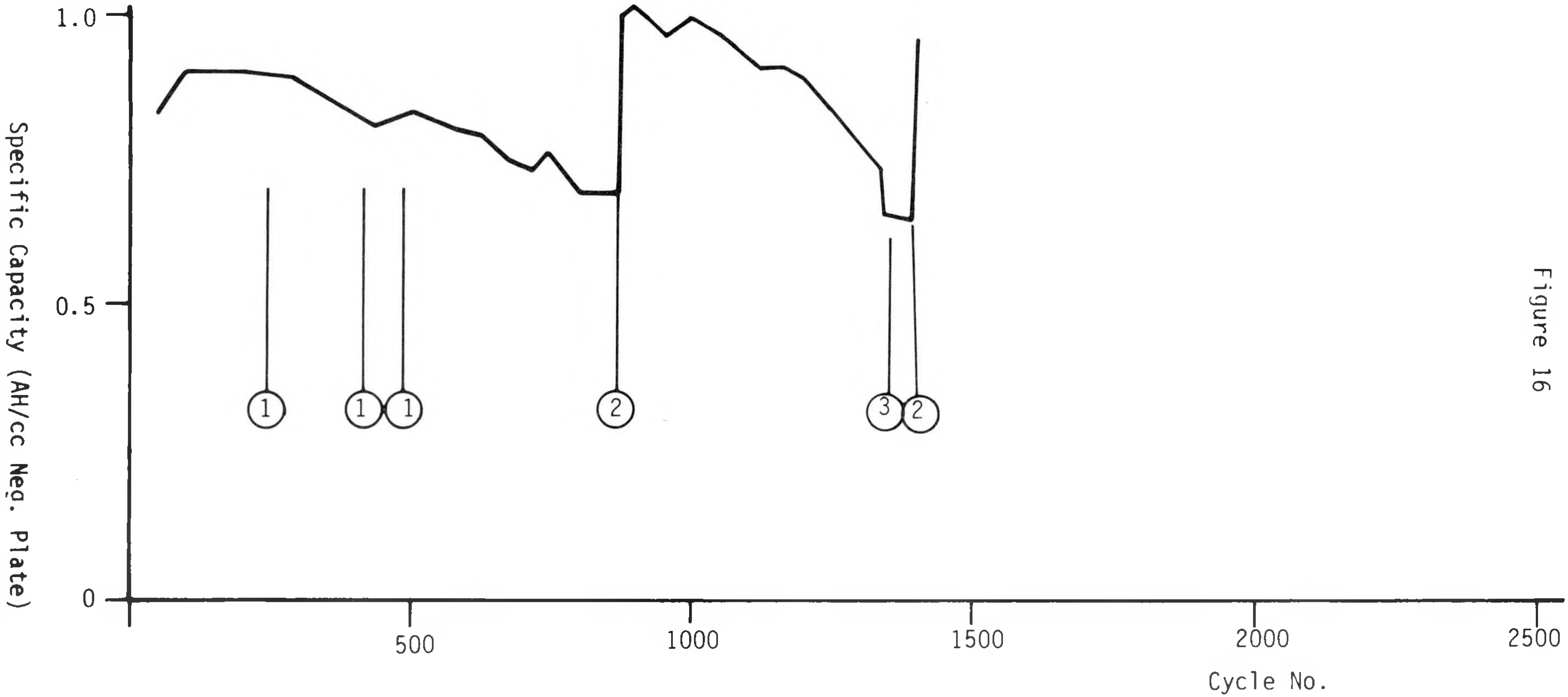


Figure 16

REMARKS:
1 Sulphide added to one cell
2 New Electrolyte with Sulphide
3 One Cell Disconnected

the electrolyte of all six (6) cells was replaced with electrolyte containing the sulfide additive. Average capacity promptly improved from 0.7 to 1.01 AH/cc after addition of this fresh electrolyte. One cell was removed from test at the end of the report period for examination to remedy its low capacity. Further testing to increase understanding of the observed beneficial effect of sulfide additive upon cell performance was planned.

2.5.6 Mechanical Separator Study

The mechanical separator investigation continued throughout FY-1981 as life cycle tests of cells with Jungfer separators. The sintered PVC sheet separators (featuring fine 0.2 mm ribs against the nickel plate, coarse 0.4 mm ribs against the iron electrode, and a 0.25 mm web-sheet thickness) continued to perform well. Two sets of test cells were included in this study. Electrolyte was changed periodically to prevent accumulation of excess carbonate. The first set of six (6) cells underwent successive charge-discharge cycling (Figure 17), at respective C/5 and C/3 rates, exceeding one thousand nine hundred (1,900) cycles. A second set of six (6) test cells (Figure 18), charged at the C/5 rate and discharged at the C/3 rate, was cycled in different ambient temperatures recording over one thousand nine hundred and fifty (1,950) cycles. Three (3) cells were tested at room temperature and three (3) cells were tested while sitting in a $40^{\circ}C$ water bath to accelerate aging. Two cells of this latter group began to exhibit declining capacity after passing one thousand eight hundred (1,800) cycles and were removed from test for examination. One of these cells resumed test cycling after a 15% improvement in capacity was effected by the addition of sulfide to its electrolyte. Post operative examination of the other cell was still in progress at the end of the reporting period.

2.6 Battery Development

Module and full-scale battery development continued during the reporting period. The development of battery ancillary systems continued; the watering system development (Paragraph 2.6.1) continued; and design of the flame arrestor system (Paragraph 2.6.2) progressed. Two (2) five-cell modules completed conditioning cycles early in the year and were shipped to the Laboratory for life test. Module SP1-5, a 6 volt unit rated at 270 AH, was assembled incorporating single-pass positive electrodes utilizing expanded metal substrate. After completion of conditioning cycles, it also was delivered to the Laboratory. Preparations were underway at the end of the reporting period to construct another deliverable, five-cell module utilizing SP-101 experimental plates having expanded metal grid with a porosity of 79.5% and strength ranging from 1,500 to 1,700 psi. Finally, an eighty-cell, 96 volt battery (Figure 19) was assembled and had finished conditioning cycles by the end of FY-1981.

2.6.1 Watering System Investigation

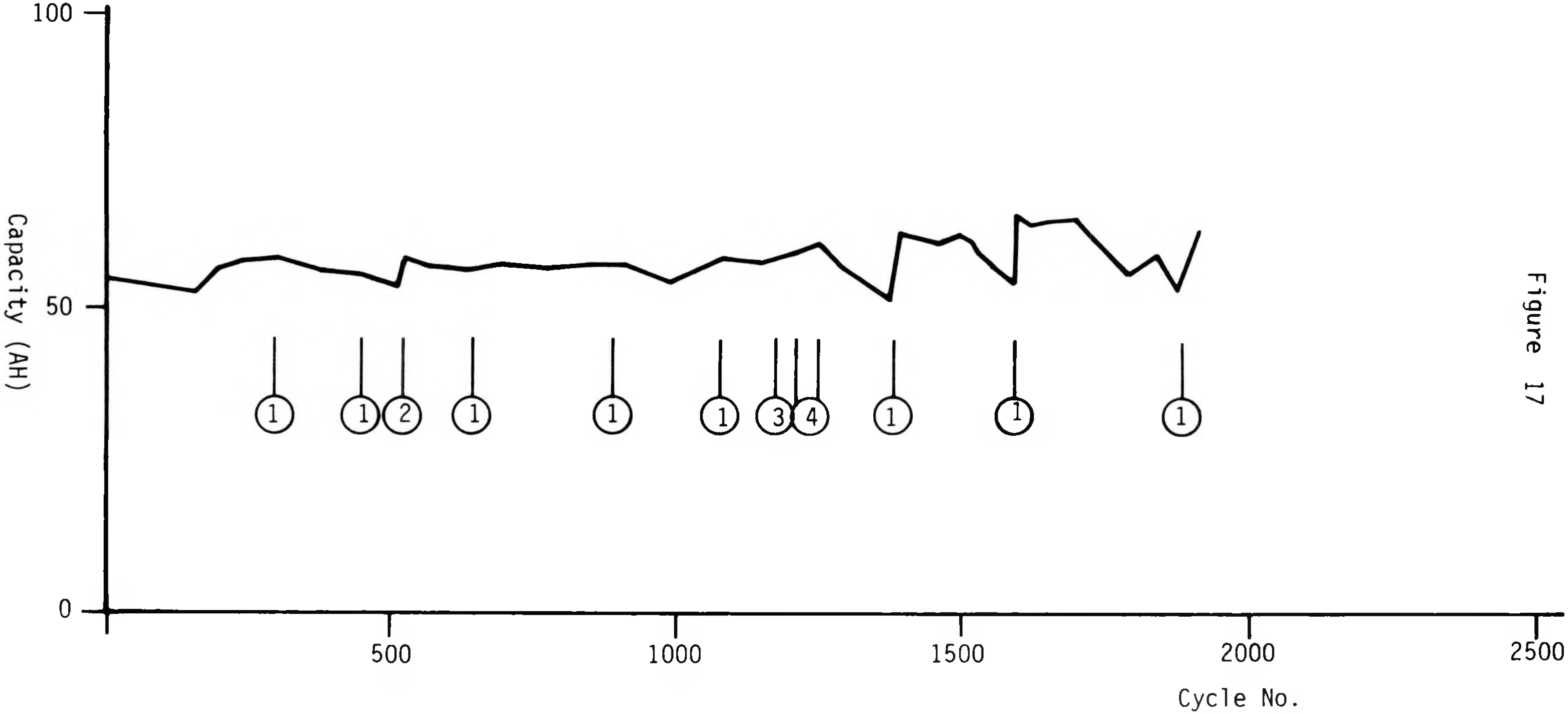
The watering system study reported in Section 4.0 of the report for FY-1980 was completed during this reporting period. The attachment of the watering system apparatus rendered the cell case covers of Module 003 unsuitable for normal cycling. It was decided to transfer the plate/separator cell stacks of Module 003 into new molded cell cases instead of replacing the cell cover assemblies of the older handcrafted

Figure 17

SEPARATOR LIFE CYCLE TEST

6 Cells, Colorado Process Electrodes

Average Capacity, $\frac{\text{total AH}}{6}$, Plot

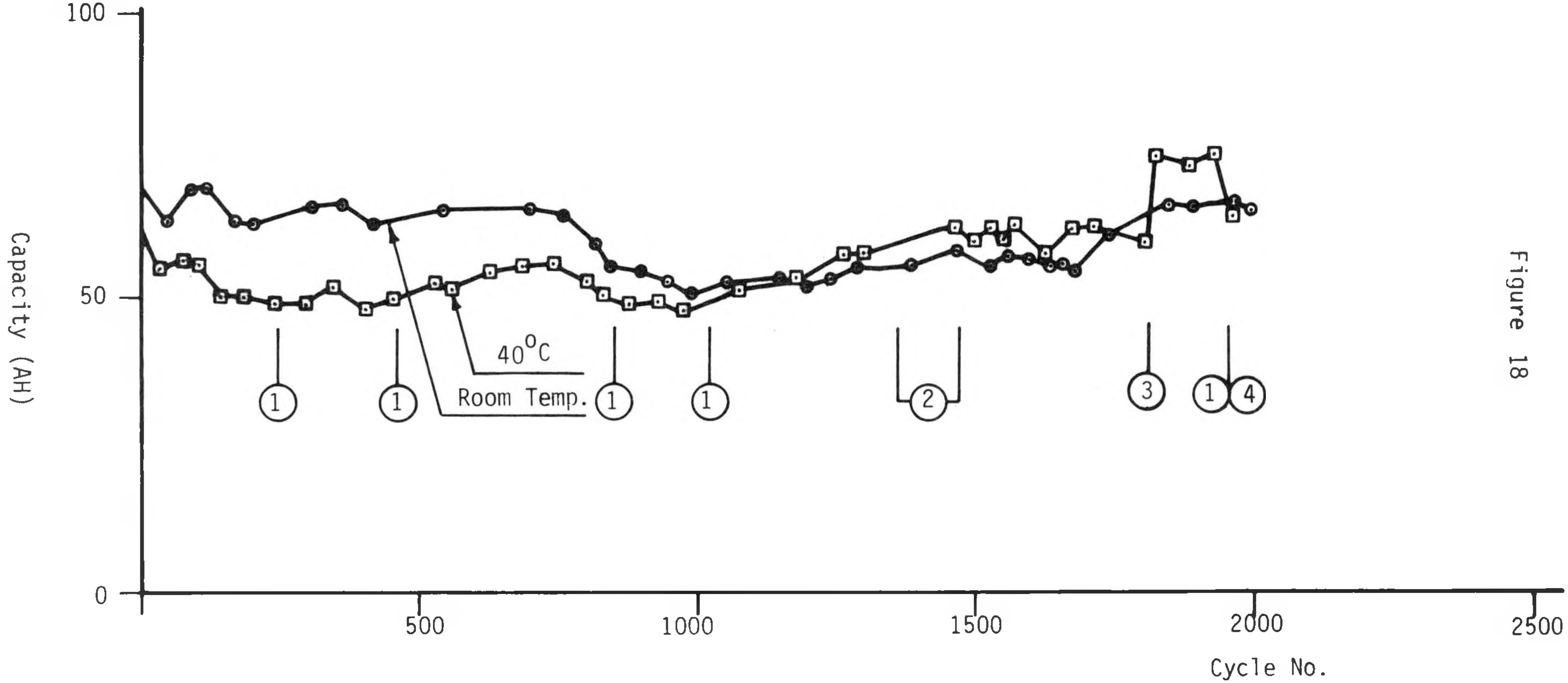


REMARKS:
1 New Electrolyte
2 61 H Charge at C/5
3 New Separators In One Cell
4 Return Factor 170%

SEPARATOR LIFE CYCLE TEST

6 Cells, Joplin Process Electrodes

3 Cell Average Capacity Plots



REMARKS:
1 New Electrolyte
2 Return Factor 170%
3 Two Cells (40°C) Disconnected
4 One Cell (40°C) Disconnected

Figure 18

Figure 19
96 Volt Battery

plexiglass containers such as those employed by Module 005, pictured in Figure 20. Module 003 was later equipped with the present updated watering system and flame arrestor equipment as shown in Figure 21.

2.6.2 Flame Arrestor Development

All flame arrestors previously considered failed to prevent flame propagation back into the system. Then a number of flame arrestor systems employing modifications of the Davy Lamp principle were examined. The most critical set of conditions were determined to occur when, with a pressurized system of stoichiometric amounts of hydrogen and oxygen, pressure of the system approaches atmospheric pressure. Initially, this type of flame arrestor performed its function successfully when used in conjunction with an air dilution system, through which exiting gas was mixed with a directed flow of air. In further testing, however, fan augmented Davy Lamp systems failed, in a significant number of trials, to prevent flame propagation back into the system. The flame arrestor system currently in use on single and dual modules (Figure 21) consists of a water/gas chamber, a one-way valve and an Oldham, flame retardant battery cap. The water/gas chamber is used to add electrolyte or distilled water. It also serves as a gas-collect reservoir. The one-way valve maintains a pressure of from 1/2 to 1 psi in the modules. This prevents outside air intrusion into the cells, which could form carbonates. The Oldham cap consists of small polypropylene pellets and a sintered PVC disk. Repeated tests show the cap functions effectively to exinguish flame from a single ignition preventing flame propagation back into the system. Further flame arrestor development work was scheduled for the next reporting period.

2.7 Life Cycle Testing

Extensive testing of cells and modules took place during the year to determine the performance capabilities of varied cell configurations. Among the numerous cells undergoing performance tests, Cell 007 (Figure 22), Cell 273 (Figure 23), and Cell 1400 (Figure 24) registered respective output capacities of 315 AH, 310 AH and about 25 AH. Cell 007 approached eight hundred (800) cycles achieving 55 WH/Kg specific energy toward the end of the report period while Cells 273 and 1400 exceeded two hundred fifty (250) cycles and seven hundred fifty (750) cycles respectively. A performance test group of eight (8) 85 AH cells finished one thousand five hundred and thirty (1,530) cycles with an average capacity as profiled in Figure 25. Another test group of twelve (12) 300 AH cells passed five hundred eight (580) cycles with average capacities of its six-cell subgroups as set forth in Figure 26. Two and five-cell modules, composed of cells incorporating twelve nickel plates and thirteen iron electrodes continued life cycling during FY-1981. Module 003, with a theoretical capacity of 329.0 AH, completed eight hundred (800) cycles (Figure 27) maintaining a stable output capacity of 265 AH. Finally, Module 005, with a theoretical capacity of 337.7 AH finished six hundred (600) cycles (Figure 28) registering a capacity of 240 AH. All modules demonstrated specific energy about 45 WH/Kg.

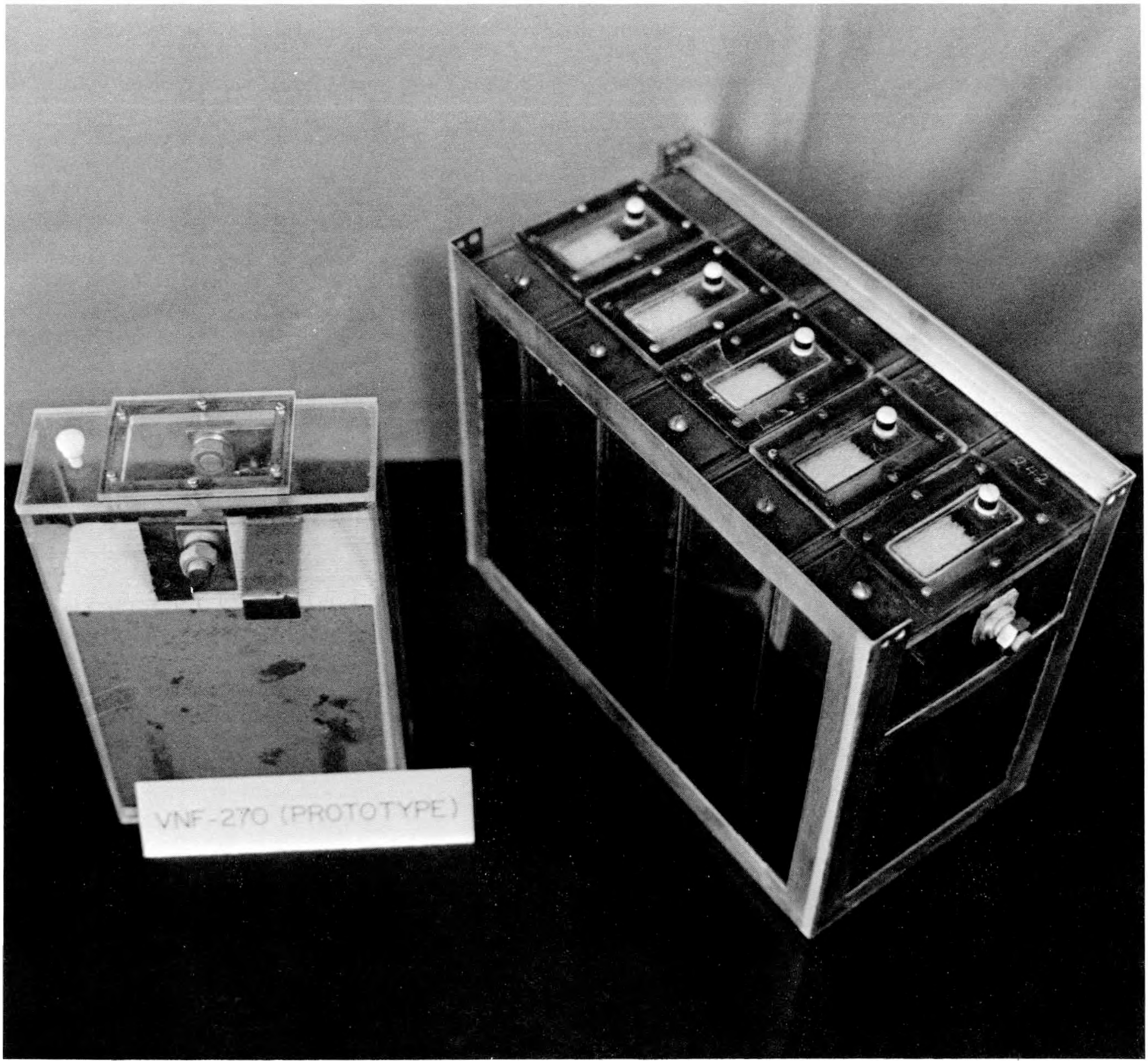


FIGURE 20
Module 005

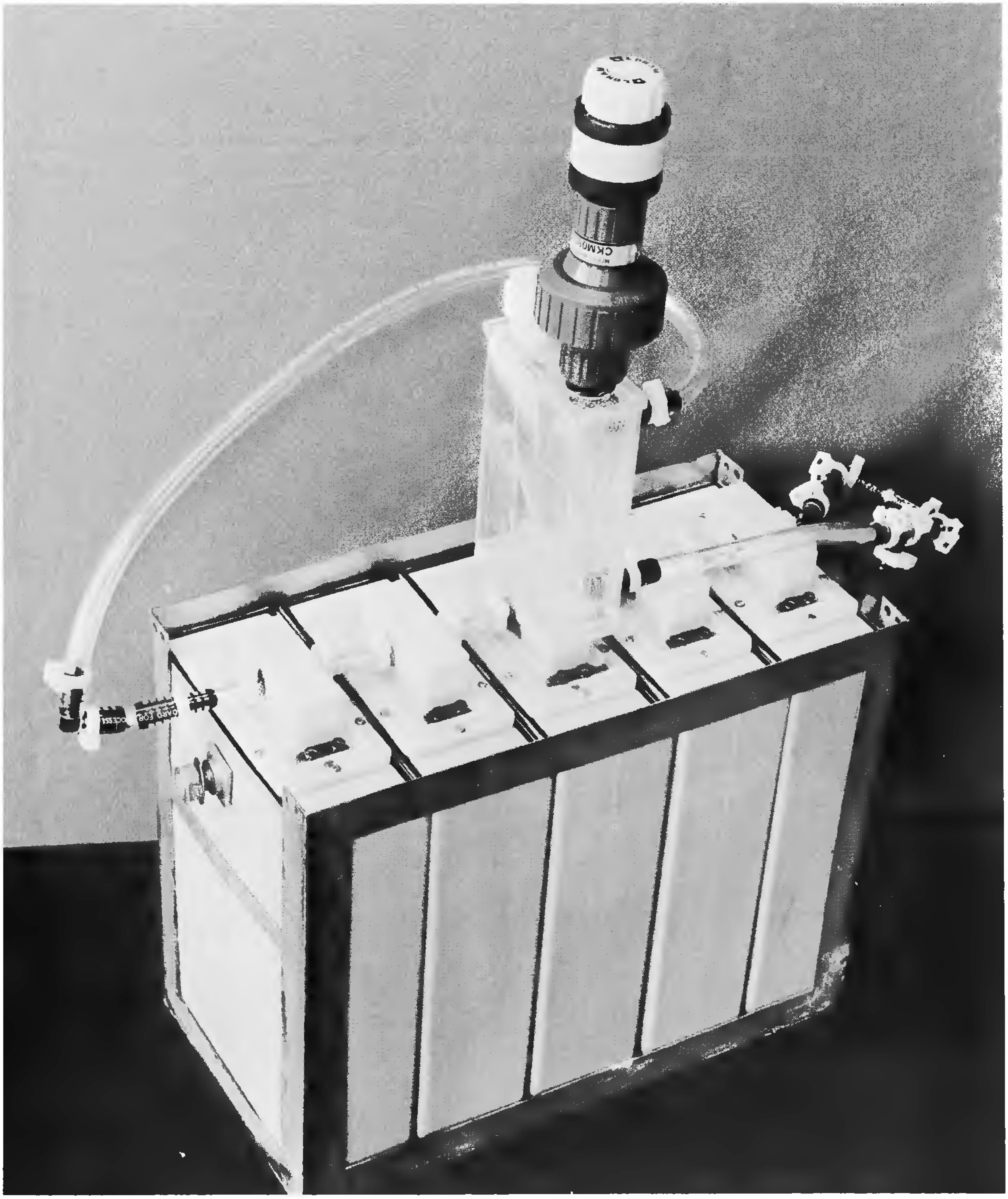
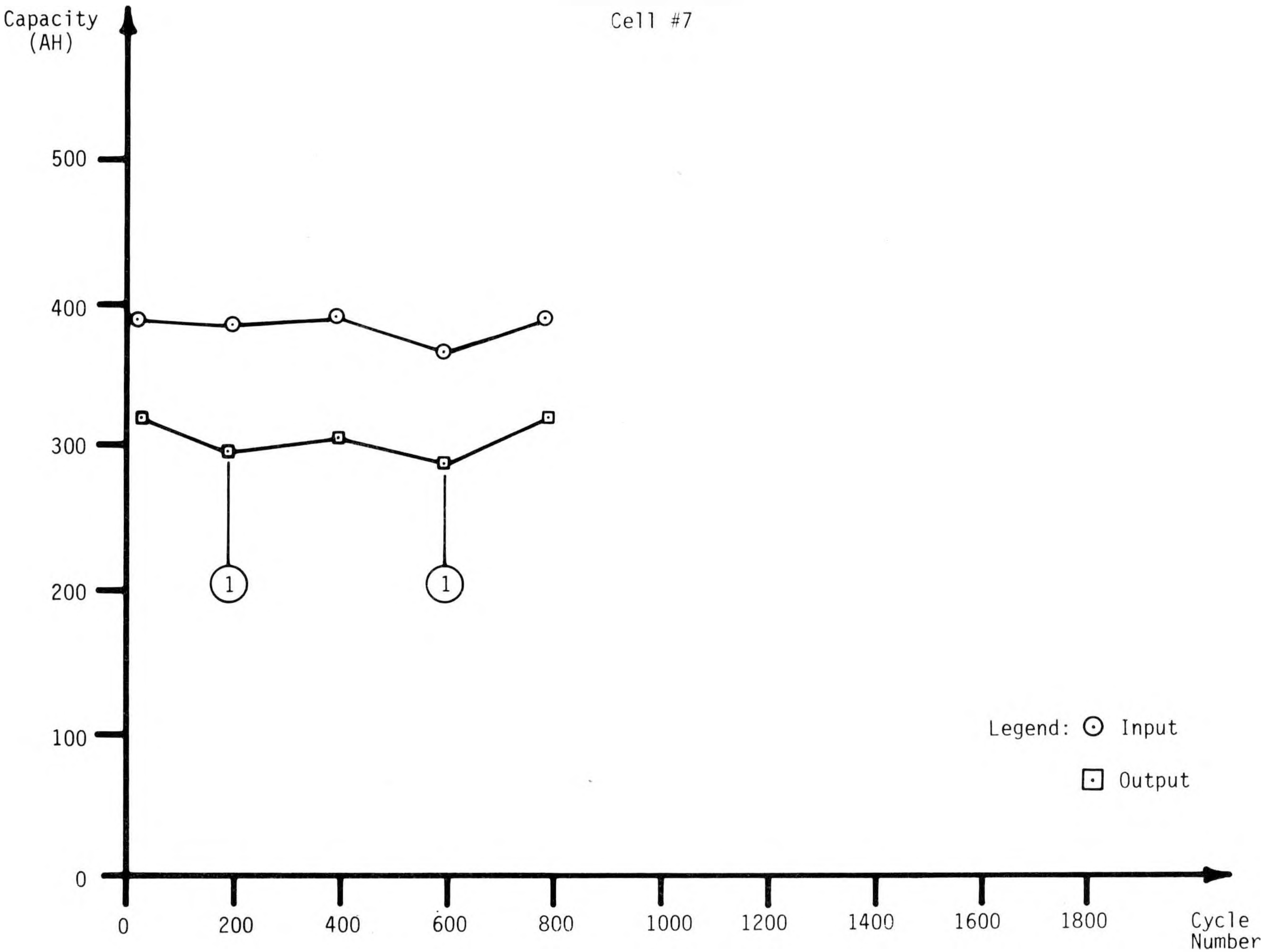


FIGURE 21
Module 003

LIFE CYCLE TEST

Cell #7

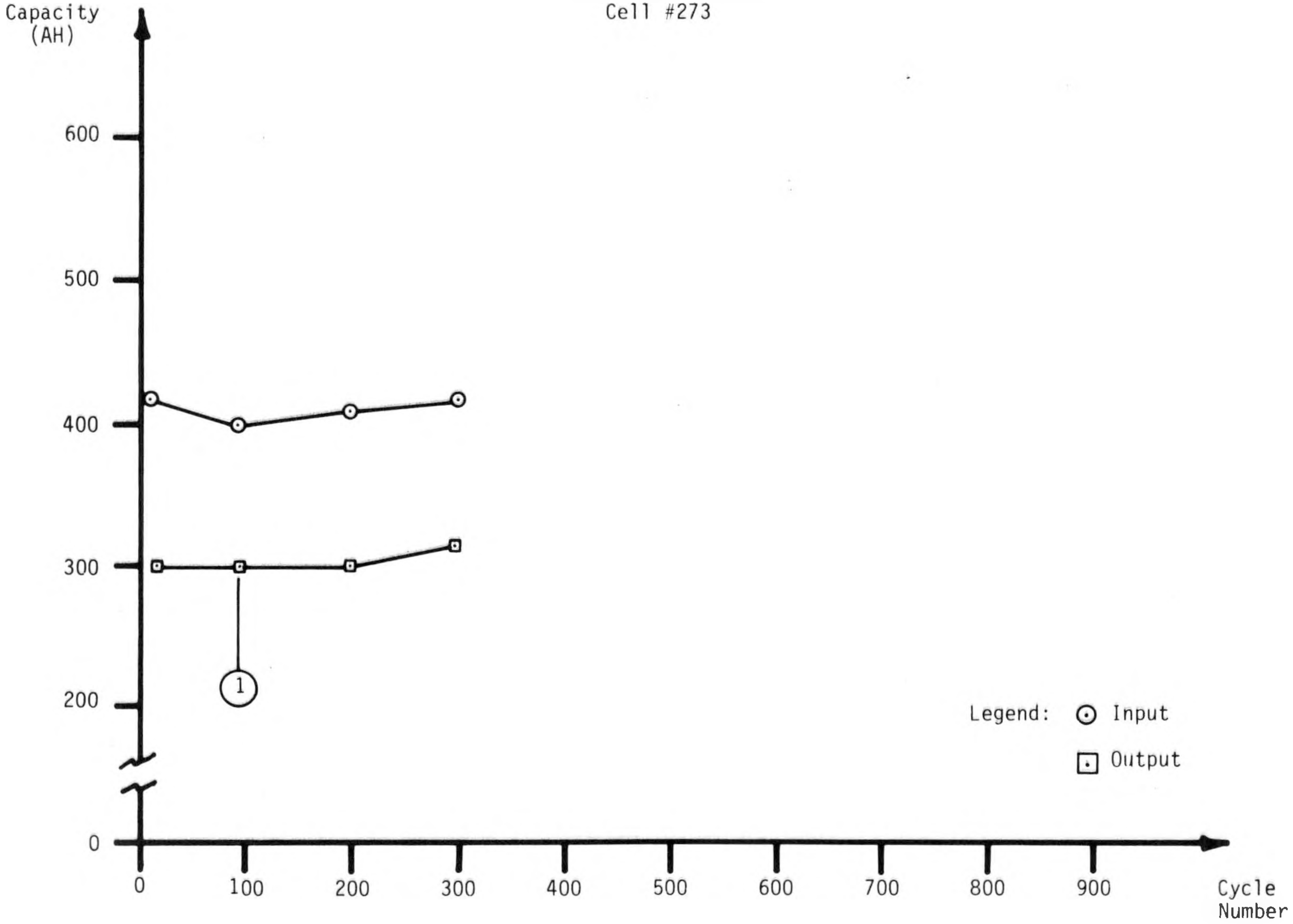


REMARKS: 1 NEW ELECTROLYTE

FIGURE 22

LIFE CYCLE TEST

Cell #273



REMARKS: 1 NEW ELECTROLYTE

FIGURE 23

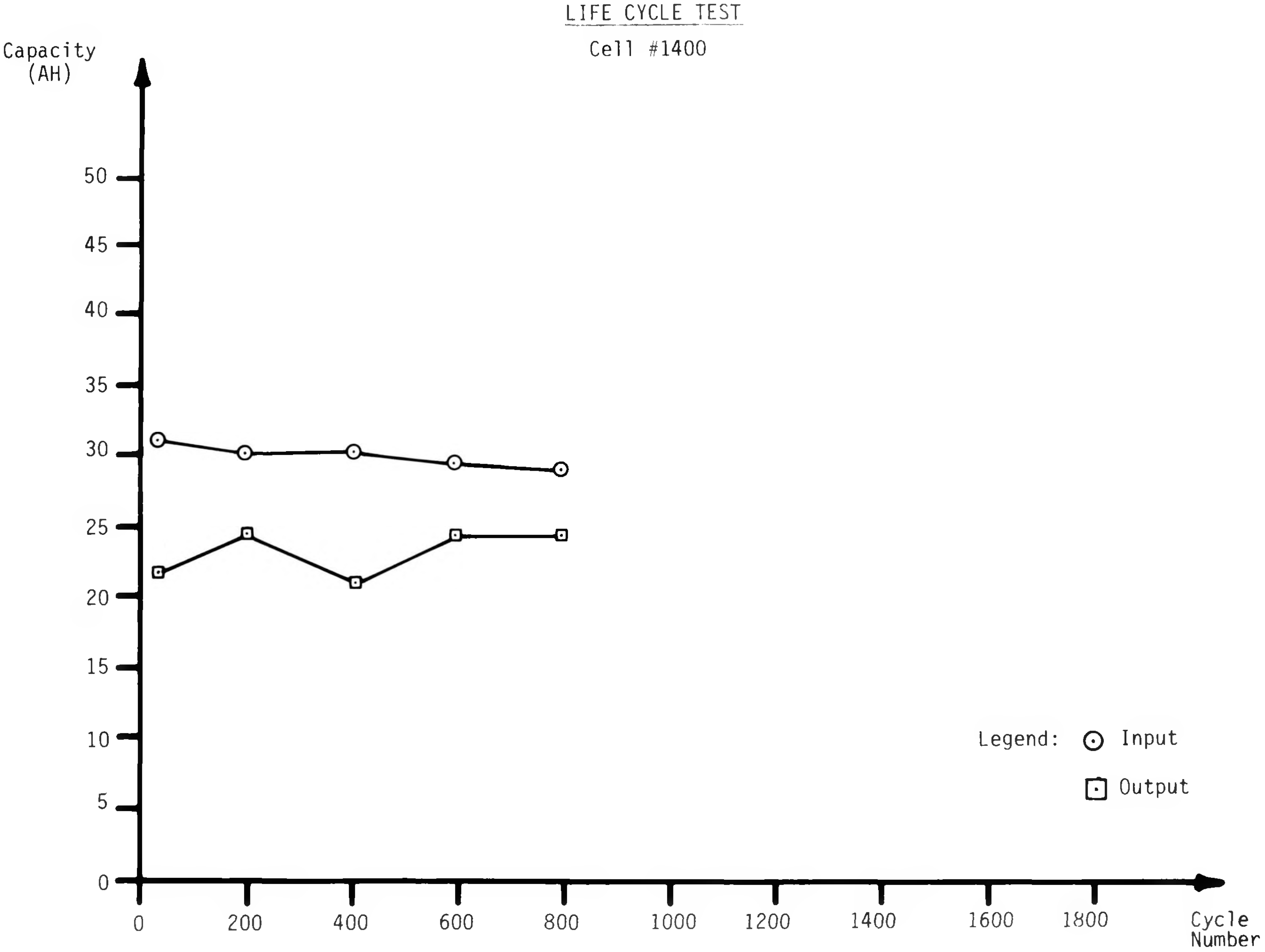
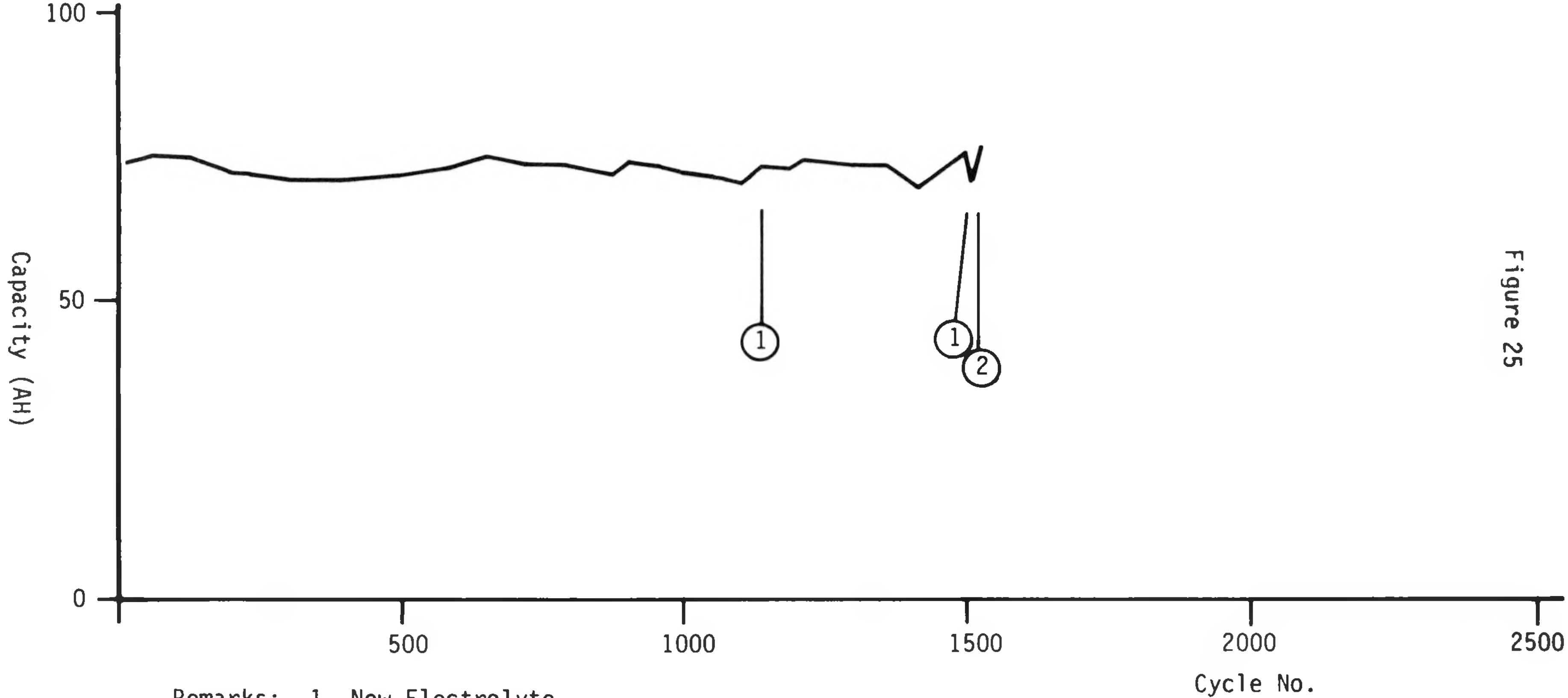

LIFE CYCLE TEST
Cell #1400
Capacity (AH)
50
45
40
35
30
25
20
15
10
5
0
200
400
600
800
1000
1200
1400
1600
1800
Cycle Number
Legend: Input
Output

FIGURE 24

LIFE CYCLE TEST

8 85 AH Cells

Average Capacity



Remarks: 1 New Electrolyte

2 New Separators in Three Cells

Figure 25

LIFE CYCLE TEST

12 300 Ah Cells

Average Capacity

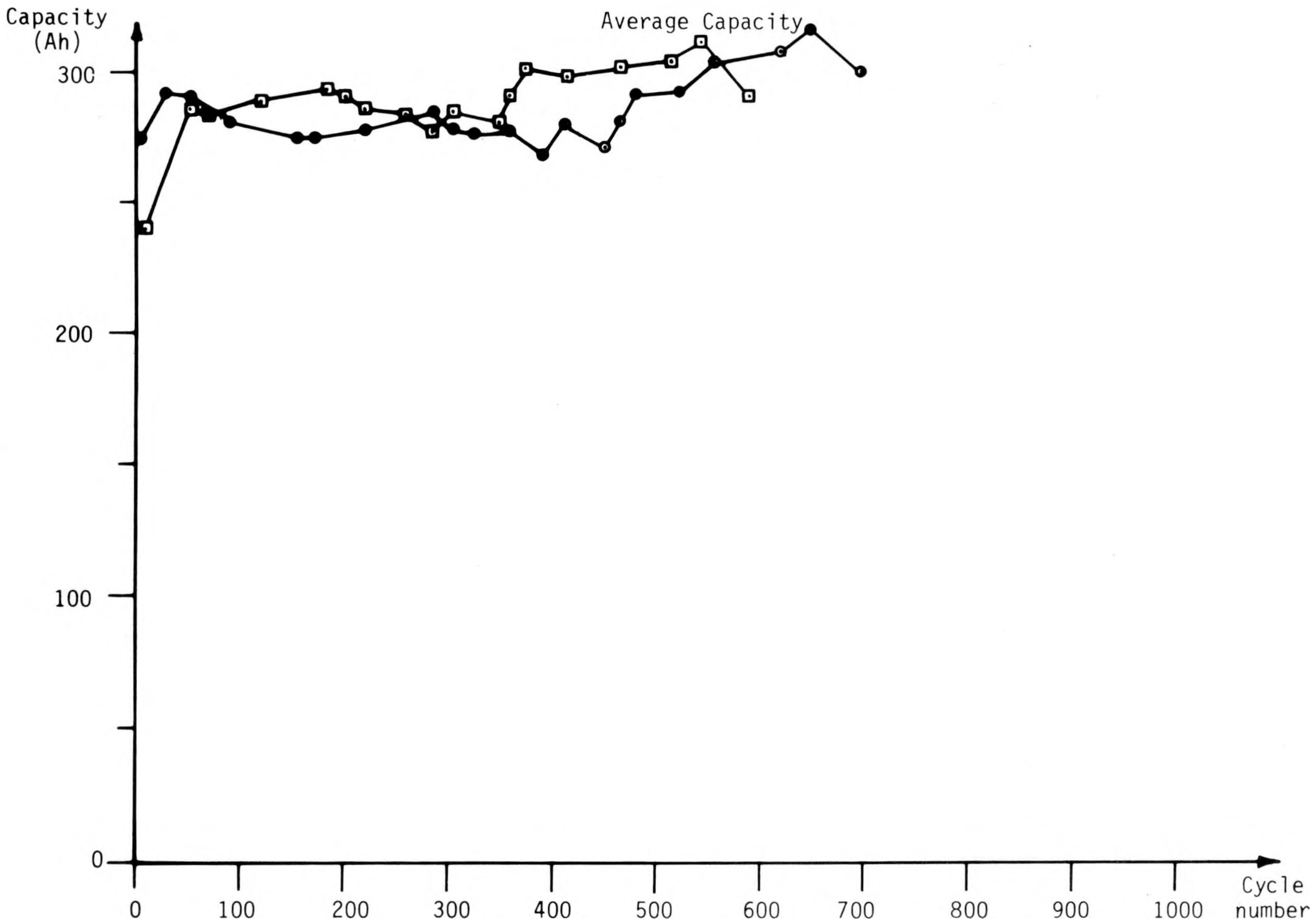
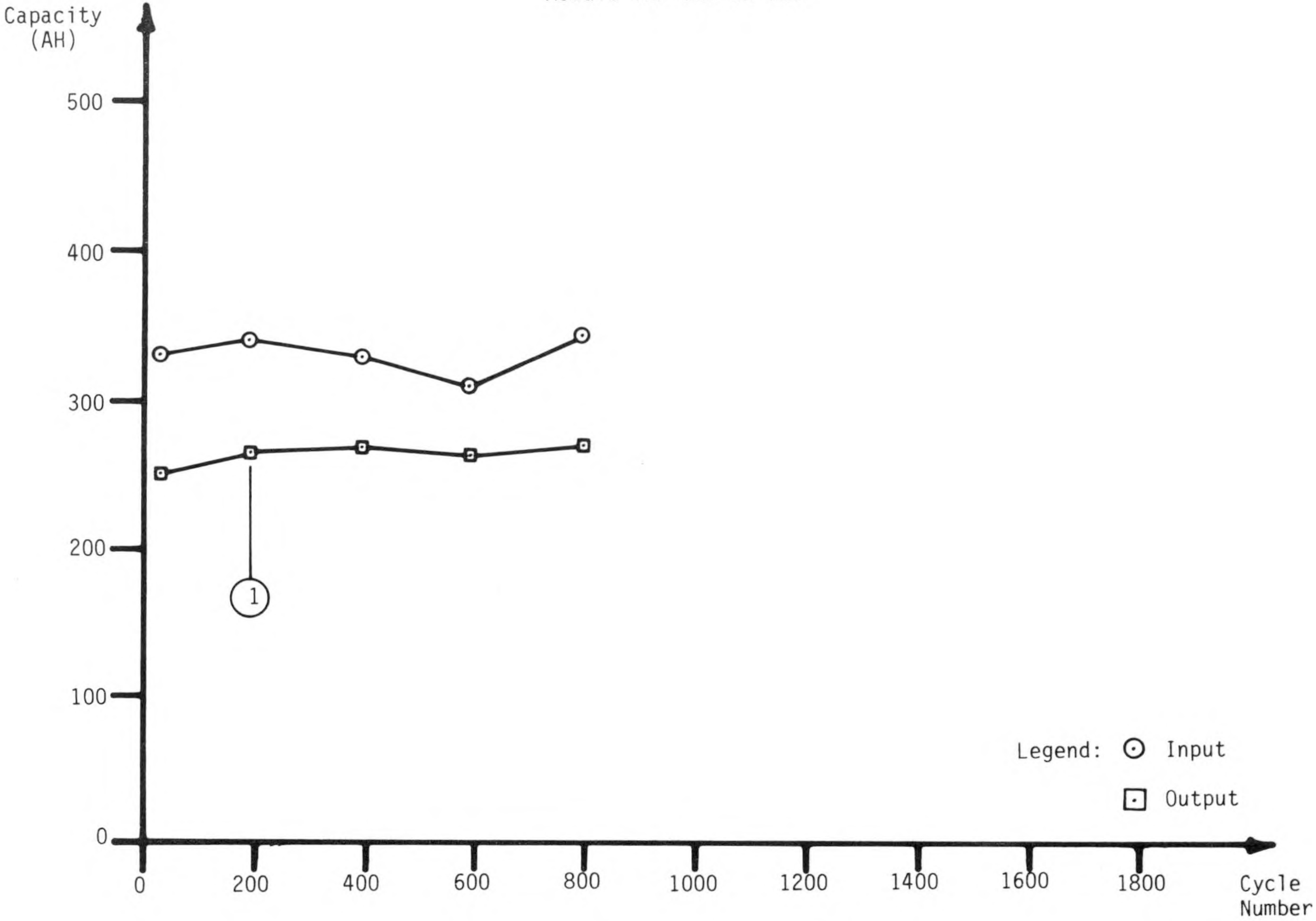


Figure 26

LIFE CYCLE TEST

Module VNF 300-X5-003

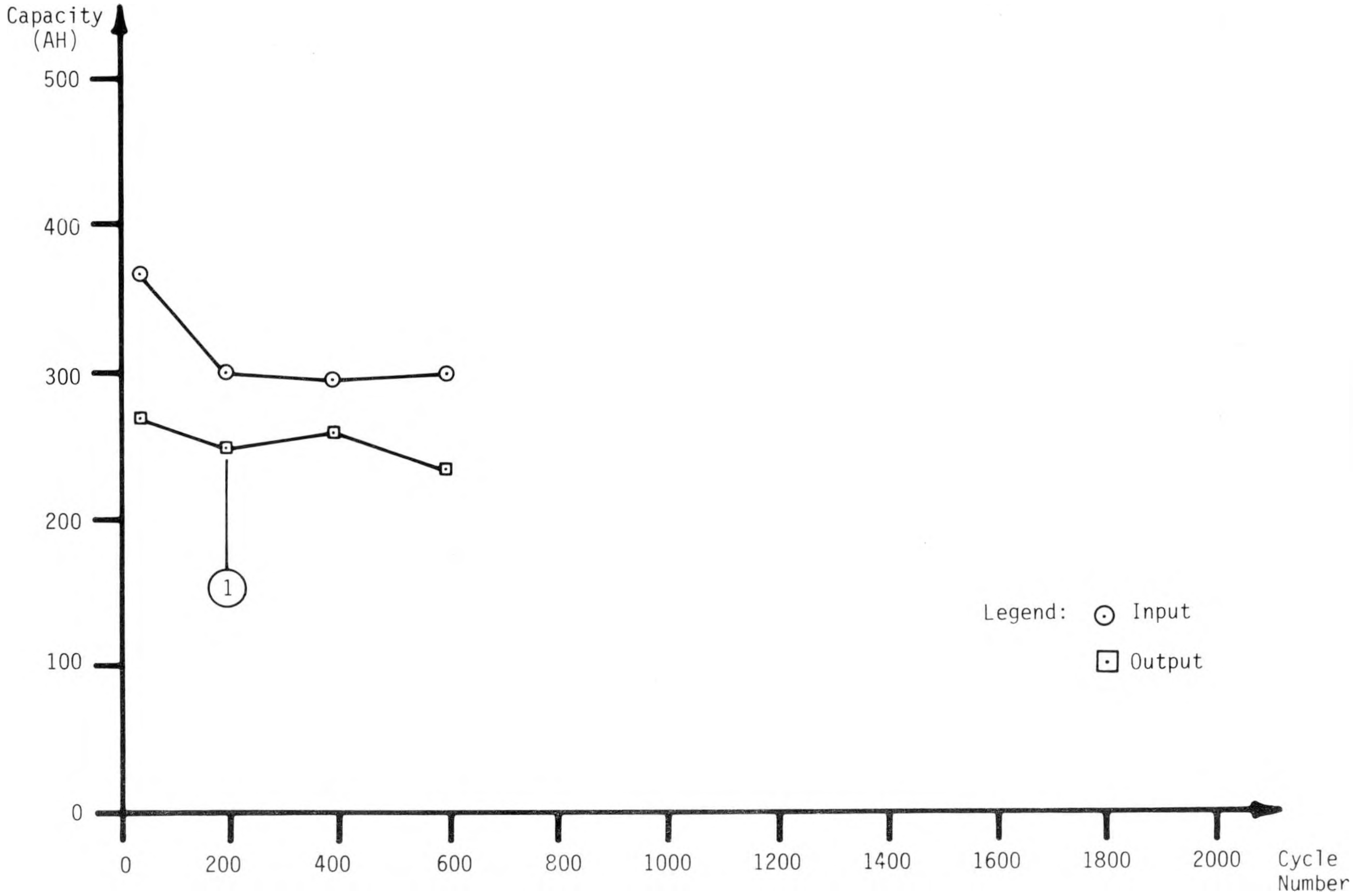


REMARKS: 1 NEW ELECTROLYTE

FIGURE 27

LIFE CYCLE TEST

Module VNF 300-X5-005



REMARKS: 1 NEW ELECTROLYTE

FIGURE 28

3.0 QUALITY ASSURANCE

The Quality Assurance group continued to make significant contribution to the Eagle-Picher Nickel-Iron battery development program. An acceptance weight criterion was developed and refined during the report period for use in the fabrication of positive nickel electrodes. This screening method was demonstrated to be effective within a one-percent (1%) margin of error in selecting plates that, when sintered, exhibited acceptable thickness, porosity and bend strength. The Document Resources Center was instituted, implementing provisions of EPQC-1397 and upgrading the documentation required to assure the retrievability and replication essential in a research and development program. Other important tasks accomplished by Quality Assurance personnel included performance of receiving inspection, maintenance of inventory control and the keeping of materials archives. Quality Assurance activities were in keeping with the Quality Assurance Program Plan set forth in EPQC-100-Ni/Fe. Quality Control functions in the Nickel-Iron development program were the responsibility of the Quality Assurance engineer and a Quality Control technician. The Quality personnel were assigned to the project by the Product Assurance manager to whom they directly reported.

4.0 CONCLUSIONS

The FY-1981 program continued to show significant progress in the drive to meet the established program goals as set forth below.

1. Initial full-scale electrode testing, which reached two thousand three hundred (2,300) cycles in FY-1981, established the capability of the chosen technology to yield the desired cycle life.

2. Plaque processing has now demonstrated significant improvements in the area of higher specific energy with life cycle tests continuing.

3. The separator test cells have demonstrated the sintered PVC separator to be adequate for over 1,900 cycles.

4. Tests on full-scale (270 AH) Cells continued throughout the period. Cells incorporating the latest in electrode technology were placed on test during the year. Initial full-scale cells in the program have now exceeded eight hundred (800) cycles in the continuing life test regimen. Cell #7 output peaked at about 315 AH and 55 WH/Kg specific energy.

5. Five-cell, 6 volt modules, a significant step toward the full-scale battery stage, continued cycling at both the Eagle-Picher test center and the NBTL with some units exceeding seven hundred (700) cycles. All these modules have demonstrated about 45 WH/Kg specific energy. The most recent module to be placed on test at the NBTL incorporated the latest electrode technology.

6. Significant progress was achieved in the development of single-point watering systems. Initially, a prototype watering system is slated for incorporation into a 6 volt module during the proposed FY-1982 program.

5.0 MAJOR PUBLICATIONS

1. Eagle-Picher Industries, Inc., "Annual Report for 1978 on Research, Development and Demonstration of Nickel/Iron Batteries for Electric Vehicle Propulsion," Argonne Report, ANL/OEPM-78-13, October 1979.

2. Eagle-Picher Industries, Inc., "Annual Report for 1979 on Research, Development and Demonstration of Nickel/Iron Batteries for Electric Vehicle Propulsion," Argonne Report, ANL/OEPM-79-13, June 1980.

3. R. Hudson, "Development of the Nickel/Iron Battery System for Electric Vehicle Propulsion," Proc. EVC Expo '80 Conference, St. Louis, May 20-22, 1980, Paper EVC No. 8031, Electric Vehicle Council, Washington, D.C., 1980.

4. R. Hudson, E. Broglio, "Development of the Nickel-Iron Battery System for Electric Vehicle Propulsion," 29th Power Sources Symposium, Atlantic City, New Jersey, June 10-13, 1980.

5. Eagle-Picher Industries, Inc., "Annual Report for 1980 on Research, Development and Demonstration of Nickel-Iron Batteries for Electric Vehicle Propulsion," Argonne Report, ANL/OEPM-80-16, March 1981.

6. K. Gentry, R. Hudson, "Ni-Fe Batteries: An Alternate to Lead Acid Electric Vehicle Propulsion," 16th IECEC 819322, August 1981.

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