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FINAL REPORT

TASKS I AND III - Electrode Improvement and Life Testing
DEVELOPMENT OF FUEL CELL ELECTRODES

by

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CONTRACT NAS 3-9430

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FOREWORD

The work described herein was done at the Parma Technical Center, Union Carbide Corporation, Fuel Cell Department, under NASA Contract NAS 3-9430 with Mr. W. A. Robertson, Space Power Systems Division, NASA-Lewis Research Center, as Project Manager.

ABSTRACT

Research has been conducted over a 22-month period directed toward performance improvement of thin electrodes for use in circulating electrolyte-type fuel cells for aerospace applications. This report covers the electrode improvement program; a separate report NASA CR-72305 describes the results of engineering analysis of the conceptual fuel cell system. Program goals were an operating lifetime of 3000 hours, with an initial voltage above 0.9 volt and a voltage degradation less than 40 millivolts per 1000 hours, at a current density of 200 ASF.

Moderately pressurized cells (30 psia) have demonstrated the feasibility of meeting the voltage goal and voltage degradation rate goal, with a longest operating lifetime (within these specifications) of 2,450 hours.

Principal problem areas were delamination of cathodes, which was eliminated during the Contract effort; and weepage of electrolyte into the oxygen gas passages, which was reduced but not completely eliminated prior to completion of the Contract.

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SECTION I

1.0 SUMMARY

A program of 22 months duration was conducted under Contract NAS3-9430 with the objective of developing high performance electrodes for circulating electrolyte, H_2-O_2 fuel cell systems. Various modifications of the basic Union Carbide thin or fixed zone electrode and commercially available electrodes from other sources were evaluated during the course of the contract. Emphasis in the experimental work was placed upon testing cells with electrode active areas varying from 0.125 ft^2 to 1.3 ft^2 at current densities from 100 to 300 ASF and temperatures of 90 to 100°C. Most of the cell testing work was centered at 200 ASF. At this test center, initial (Task I) program goals of 1) 0.9 volt initial potential and 2) <20 mV decline in 500 hours were established. The program goals were later refined (Task III) to include a voltage decline of a) <40 mV/1000 hours or b) <8 mV/200 hours with a desired lifetime of 3000 hours.

Cell tests are summarized in Table I. This table does not include the numerous quick cell tests (i.e., 2 cm^2 or 0.31 in^2 active area) which were conducted as an adjunct to electrode development and various problem solving studies.

TABLE I

SUMMARY OF CELL TESTS CONDUCTED ON CONTRACT NAS3-9430

Item	Cell Type	No. of Cells	Current Density (ASF)	Pressure (psia)	Purpose
A	1/8 ft^2	31	100-200	15-30	Preliminary Screening Tests
B	1/8 ft^2	35	200	15	Life Tests
		5	200	30	Life Tests
C	1/8 ft^2	4	NASA Cycle	15	Life Tests
		1	NASA Cycle	30	Life Tests
D	1/8 ft^2	3	100	15	Life Tests
		2	300	15	Life Tests
E	1/8 ft^2	3	200	15	T-3N Cathode Development
		1	100	15	T-3N Cathode Development
F	0.325 ft^2	3	200	15	Construction Tests
G	Flight-Size (1.3 ft^2)	2	200	15	Life Tests
		3	200	30	Life Tests
H	Special Cells	5	Various	15	Various

Essentially, all of the "life test" cells were either "hybrid" cells using Union Carbide T-2 anodes (2.5 mg/cm^2 noble-metal catalyst) and American Cyanamid LAB-40 cathodes (40 mg/cm^2 Pt catalyst) or "LAB-40" cells in which both electrodes were identical. Various modifications of the wetproofed backing layer on the LAB-40 electrodes were evaluated in these tests. Considering the 200-ASF life tests as a 45-cell group (i. e., Items B and G, Table I), 17 cells met the Task I Milestone No. 1, and 22 cells met Milestone No. 2. Both milestones were simultaneously realized in 9 cells. In every case where cell operation was at 30 psia, Milestone No. 1 was achieved, since increasing the pressure from 15 to 30 psia resulted in about a 30-mV performance improvement. No cells met the 3000-hour performance goal of Task III. The best demonstrated performance was with Cell No. A074 which achieved a lifetime of 2,450 hours, based on the 40 mV/1000-hour voltage-decay criterion (or 2,100 hours, based on the 8 mV/200-hour criterion).

Two major problems were experienced in the testing program; viz.: 1) delamination of the wetproofing layer from the LAB-40 electrodes, and 2) weepage of electrolyte into the gas passages. Near the end of the program, the delamination problem was solved by modifications in the chemistry and method of application of the LAB-40 backing layer (the so-called C-II modification). Changes in cell construction, however, did not eliminate weepage of electrolyte into the gas passages.

It was generally impossible to establish whether the leakage was through electrode edge seals or through the active area, per se. However, electrolyte weepage through the cathodes was substantially greater than through the anodes. Considering the same group of 200-ASF life tests as above, the average leakage into the anode spaces was 0.6 ml/hr·ft^2, while the average leakage into the cathode spaces was 6 ml/hr·ft^2.

Limited work was done on the development of a high-platinum T-3N cathode which, because of its structure, could be operated at higher gas pressures. In quick-test cells, cathode potentials equivalent to the LAB-40 electrodes were obtained (i. e., +0.010 to +0.020 volt vs. Hg/HgO). Although these best electrodes were not tested in larger cells, limited testing of preliminary versions of the T-3N cathode showed very low weepage in tests of 64 to 240 hours duration.

SECTION II

2.0 INTRODUCTION

This is the final report on NASA Contract NAS3-9430 covering the period from June 30, 1966 to April 30, 1968. The basic purpose of this program was to improve the capabilities of the Union Carbide thin, fixed-zone fuel cell electrodes bringing their electrochemical performance to the high levels required for various space missions. Implicit in the work was use of a low temperature circulating alkaline electrolyte and hydrogen and oxygen as fuel cell reactants.

The program consisted of three tasks. Task I was primarily concerned with electrode development. The goal of Task I was to demonstrate in cell tests during operation at 200 ASF that initial potentials of at least 0.9 of a volt could be obtained and the potential after 500 hours of operation would be no less than 0.88 volt. This involved testing of cells with 1/8 ft^2 and $>$1 ft^2 of active area. The former were designated as "small" cells while the latter were termed "flight-size" cells. Eighteen test stands were constructed to carry out this experimental work; seven of these were capable of operation at pressures of up to 3 atm while the balance were designed for operation at 1 atm.

Many of the cells tested during the Task I program ran well beyond 500 hours with low voltage degradation rates. To provide consistency in testing, operation of the cells which exceeded the program milestones noted above was continued until the voltage decayed to 0.78 volt at which point the tests were terminated.

The objective of Task II was to conduct a preliminary system design of a 5 kW hydrogen-oxygen fuel cell using the various system precepts which had been reduced to practice by Union Carbide including circulation of KOH, and recirculation of reactant gases for by-product water removal. Task II was completed and reported in a separate final report, NASA CR-72305.

In Task III the primary objective was to perform life tests on electrodes selected as a result of the Task I evaluation studies. The target performance for Task III was to obtain 3000 hours life with continuous operation at 200 ASF. In the Task III work, end of life was defined as an average voltage decay of greater than 40 mv/1000 hours or greater than 8 mv in any 200 hour period. As before, the initial voltage milestone was 0.9 volt. Thus, at the end of life the target voltage would be 0.78 volt minimum at 3000 hours.

The Union Carbide thin, fixed-zone electrode as it existed at the outset of this program had been optimized for commercial applications. Typically, the thin electrode consists of a lightly catalyzed, active carbon layer which is backed by a highly wetproofed carbon layer; and this, in turn, is supported on a thin, wetproofed porous nickel substrate. The porous nickel substrate provides mechanical support for the electrode, provides current collection and also provides a diffusion path through which the reactant gases are transferred to the electrochemical reaction zone. Because of the inherent electrochemical activity of the activated carbon, only small quantities of noble metal catalyst are required for reasonable electrode performance. Generally, the noble metal content of Union Carbide Fuel Cell electrodes is less than 2.5 mg/cm^2. Obviously, some performance is sacrificed in the interest of cost by drastically minimizing the noble metal content in the electrodes.

Under Task I of this contract, one of the objectives was to determine ways to increase the catalyst loading of the thin electrodes in order to achieve enhanced electrode performance. A second sub-task was to evaluate replacement of the porous nickel electrode backing and solid nickel current collection tabs with more conductive materials such as silver. A third sub-task was to devise an all noble metal electrode in which the carbon was essentially eliminated, while a fourth sub-task was to examine the problems of increasing the cell operating temperature from 60 to 65°C, typical of the thin electrode optimal performance level to 90-100°C. This change dictated a requirement for improved cell construction materials and also cathodes which were more resistant to oxidation than those containing highly active carbon.

Early in the program work on sub-task I indicated that merely increasing the catalyst loading of standard fixed-zone cathodes was not a fruitful course of action. On the other hand, we found that the standard, commercial Union Carbide T-2 anode gave excellent performance when operated at 90-100°C and current densities of 200 ASF. We also found that the LAB-40* electrode

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* The LAB-40 electrode is a modification to the well-developed AB-40 electrodes produced by American Cyanamid Company in which 40 mg/cm^2 of platinum are bonded to a gold plated nickel current collection screen. The standard AB-40 electrode is then backed with a porous fluorocarbon layer which prevents flooding of the electrode. . This modification was developed specifically for circulating electrolyte systems.

produced by the American Cyanamid Company gave comparable anode performance under the same conditions.

In view of these developments, the bulk of the attention toward improved electrodes was centered on the cathode. The most suitable cathode material found during the course of this program was the LAB-40 electrode. Many cells using this cathode and either of the anodes noted above exceeded the Task I milestones. While promising results were obtained with the Union Carbide T-3N cathode, this work was not carried to the state where a final judgment could be made on this electrode concept.

Approximately ninety 1/8 ft^2, 0.325 ft^2, and flight-size cells were operated during the course of this program. Most of the tests were conducted at a continuous load of 200 ASF. Limited testing was done at 100 and 300 ASF and on a simulated NASA mission profile. Some tests were run at 2 atm pressure, although the bulk of the testing was at 1 atm. Individual performance curves on each of the cells which had been completed in prior reporting periods were covered in the appropriate Semi-Annual Reports and, consequently, are not included in this Final Report. However, in the Appendix to this report are included those performance curves for all cells which were completed since the last progress report was prepared.

To establish the suitability of various materials for potential application as structural members, seals, elastomers, and spacers in cells operating at 90 to 100°C, compatibility tests were conducted by exposing selected items to 12 N KOH at 100°C for one week. Compatibility was assessed by visual appearances, weight change measurements and, in some cases, by analysis of the KOH.

SECTION III

3.0 FACTUAL DATA

3.1 Electrode Studies

At the outset of the program the, then available, standard commercial Union Carbide thin electrodes were designated as Type 2(T-2). These electrodes are approximately 22 mils thick and consist of a 0.007" thick, wetproofed porous nickel backing to which is applied first a layer of inactive carbon (the so-called "D" layer), and then a layer of activated carbon. Typically, this active layer is catalyzed with 1 to 2.5 mg/cm^2 of noble metals after electrode fabrication is complete.

These standard T-2 electrodes were used as a point of departure for the testing work. Initially, four modifications of the standard T-2 were examined as cathodes in 1/8 ft^2 cells, vis.:

1. Standard T-2 (Cells A-001, 004, 009, 010) - 1 mg/cm^2 noble metal;
2. Standard T-2 except pre-catalyzed carbon (Cells A-005, 011, 016) - 5 mg/cm^2 noble metal;
3. T-2 with silver backing (Cells A-002, 003) - 8 mg/cm^2 noble metal;
4. T-2 with silver backing and pre-catalyzed carbon (A-024, 025) - 10 mg/cm^2 noble metal.

Similarly, modifications 1, 2 and 3 above were tested as anodes. These early cell tests established that the cathode potentials of all modifications of the T-2 were unsatisfactory at 200 ASF whereas the anode potentials for the standard T-2 were excellent.

Consequently, attention was directed to modifying for NASA applications new types of experimental cathodes which had been developed by Union Carbide also for possible commercial applications. These electrodes were designated Type 3 (T-3), Type 4 (T-4), and Type 5 (T-5). In the T-3 electrode, the porous Ni layer is fully wetting and faces the electrolyte. Active carbon and wetproofed carbon layers are applied to the porous nickel facing. Since the porous nickel is completely wet by the KOH, the T-3 electrode can operate at substantially higher gas pressures than the T-2 before gas bubbles blow through the electrode into the electrolyte. The capillary forces of the KOH in the fine-pored nickel layer permit the oxygen pressure to be raised from 10-20" w. c. as in a T-2 electrode to ~100" w. c. in a T-3. As a consequence, with the T-3 concept weepage of electrolyte into the gas stream is drastically curtailed.

The T-4 electrode was considered but briefly in this program (Cells A-012, and 030). It is a two-layer electrode with active carbon being applied to a porous Teflon backing. For current collection, metallic mesh or screen is pressed against the active carbon layer. In the T-5 electrode, the current collection screen is embedded in the active carbon layer. Wetproofing is provided by application of a porous Teflon layer to the gas side of the T-5. Three modifications of the T-5 were tested in 1/8 ft^2 cells, specifically:

1. Ni screen, post-catalyzed (Cell A-008)
2. Ni screen, pre-catalyzed (Cell A-017)
3. Ag screen, pre-catalyzed (Cells A-026, 027, 029, 032, 034).

In the initial screening tests, two cathodes available from other commercial sources were also considered. One was the American Cyanamid LAB-40 and the other was the Chemcell H 9454N (Cells A-033 and 035). Based on peak electrode potentials and rate of voltage decay, the LAB-40 cathode appeared to be best suited for further analyses and was, consequently, selected as the cathode for the balance of the small and flight-size cell testing in the Task I program. Comparative performance, based on half-cell potentials, of all these cathodes is presented in Table II.

During the course of the cell testing program in Tasks I and III, two major problems associated with the LAB-40 cathode were encountered; one was delamination of the electrode during operation, and the second was weepage of electrolyte through the electrode into the cathode cavity. Delamination when it occurred was evidenced at the interface between the hydrophobic "L" layer and the AB-40 active layer. Delaminated electrodes would immediately flood in the blistered area and performance would drop drastically. When delamination was first experienced early in the Task I test program (e. g. , Cell A-018), American Cyanamid made modifications in the fabrication process which apparently eliminated the problem. However, there was a recurrence of the problem during the Task III cell testing work, and several additional changes in the chemistry and fabrication process of the LAB-40 backing were evolved by American Cyanamid and tested by Union Carbide. Results of these studies will be described in greater detail under the section entitled "LAB-40 Electrode Evaluation." Thus, throughout the program many modifications of the basic LAB-40 concept were evaluated. The ultimate backing modification designated C-II appeared to be extremely well-bonded to the active layer. With this modification, no evidence of delamination was uncovered, and post-operational peel tests revealed that the bond between the backing and the active layer was actually superior to the intra-active layer bonds.

The other cited problem, weepage into the cathode cavity, was not completely overcome. It should be pointed out that in many instances the magnitude of the electrolyte weepage was insufficient to make a judgment as to whether weepage was through the electrode to cell edge seals or through the electrode, per se. In certain instances unambiguous determinations of the source of weepage were made and are indicated in the cell testing sections of this report.

Because we were not able to develop completely leakage-free cells with the LAB-40 electrodes, a portion of the Task I and Task III effort was devoted to transferring the T-3 electrode concept to a high or all-platinum electrode. With the LAB-40 cathode, blow-through or bubbling of oxygen into the electrolyte stream occurs at about 16" w. c. while with the standard T-3 blow-through does not occur until about 65"w. c. With a LAB-40 electrode the gas pressure is typically less than the KOH inlet pressure and sometimes even less than the KOH outlet pressure. Such a situation can promote leakage, be it through the edge seals or through the electrode structure. With the T-3 concept, on the other hand, oxygen pressure is well above the electrolyte pressure, and a positive force to restrain weepage is thus always present. During the initial electrode selection work (See Table II) very little attention was directed to the possibility of modifying the T-3 electrode to the requirements of the present contract. However, as the work progressed, it appeared that an alternate electrode concept might be required to solve the weepage problem. Consequently, the work described in the next section of this report was undertaken mid-way in the cell testing program. Testing of the T-3N* emphasized small electrodes (~ 2 cm^2 active area) rather than 1/8 ft^2 and flight-size cells.

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*N post-script refers to those modifications relevant to this NASA Contract.

TABLE II

CATHODE SCREENING TESTS IN 1/8 ft^2 CELLS

Cell	Cathode Type	Catalyst Level (mg/cm^2)	Initial or Peak Potential* (mv vs Hg/HgO)
A-001	2-1	1.0	
A-004	2-1	1.0	-0.028
A-009	2-1	1.0	-0.032
A-010	2-1	1.0	-0.011
A-002	2-2	8.0	-0.038
A-003	2-2	8.0	-0.027
A-005	2-3	~5.0	+0.067
A-011	2-3	~5.0	---
A-016	2-3	~5.0	-0.005
A-024	2-4	~10.0	-0.059
A-025	2-4	~10.0	-0.160
A-007	3-1	4.0	-0.099
A-012	4-1	1.0	---
A-030	4-2	2.0	-0.073
A-008	5-1	1.0	-0.050
A-017	5-3	~5.0	-0.015
A-026	5-4	~10.0	-0.007
A-027	5-4	~10.0	-0.015
A-029	5-4	~10.0	-0.010
A-032	5-4	~10.0	-0.030
A-034	5-4	~10.0	-0.043
A-033	Chemcell H9454 N	9.0	---
A-006	LAB-40	40.0	+0.016
A-013	LAB-40	40.0	+0.045
A-014	LAB-40	40.0	+0.019
A-019	LAB-40	40.0	-0.020
A-020	LAB-40	40.0	-0.030
A-023	LAB-40	40.0	-0.036
A-028	LAB-40	40.0	-0.083
A-031	LAB-40	40.0	-0.010
A-036	LAB-40	40.0	+0.035

-indicates no reference data

*IR-free at 200 ASF and 15 psia.

As noted previously, the anode posed lesser problems. Anode potentials for the initial screening tests are noted in Table III. The average anode potential for 15 tests using the T-2 (standard) anode was 0.926 volt vs Hg/HgO . Results of similar screening tests in nine cells using LAB-40 anode showed an average of 0.930 volt vs Hg/HgO. Because the performance of the T-2 and LAB-40 anodes was comparable both were subjected to extensive testing during the balance of the program.

TABLE III

ANODE SCREENING TESTS IN 1/8 ft^2 CELLS

Cell	Anode Type	Catalyst Level (mg/cm^2)	Initial or Peak Potential* (mv vs Hg/HgO)
A-001	2-1	2.5	
A-004	2-1	2.5	0.913
A-007	2-1	2.5	0.910
A-008	2-1	2.5	0.900
A-009	2-1	2.5	0.912
A-010	2-1	2.5	0.907
A-012	2-1	2.5	---
A-016	2-1	2.5	0.932
A-017	2-1	2.5	0.928
A-024	2-1	2.5	0.939
A-025	2-1	2.5	0.938
A-026	2-1	2.5	0.939
A-027	2-1	2.5	0.928
A-029	2-1	2.5	0.938
A-030	2-1	2.5	0.921
A-032	2-1	2.5	0.940
A-034	2-1	2.5	0.948
A-002	2-2	8.0	0.892
A-003	2-2	8.0	0.921
A-005	2-3	~5.0	0.735
A-033	Chemcell H9454N	9.0	---
A-011	Modified AB-40	40.0	---
A-006	LAB-40	40.0	0.933
A-013	LAB-40	40.0	0.920
A-014	LAB-40	40.0	0.929
A-019	LAB-40	40.0	0.941
A-020	LAB-40	40.0	0.948
A-023	LAB-40	40.0	0.932
A-028	LAB-40	40.0	0.952
A-031	LAB-40	40.0	0.920
A-036	LAB-40	40.0	0.898

-indicates no reference data   *IR-free at 200 ASF and 15 psia

3.1.1 High-Platinum Loaded Cathodes

3.1.1.1 Summary

Because of the high voltage requirements specified by the contract, it was suggested that more catalyst must be incorporated into the electrode structure than was present in our conventional electrode types. In late 1966, first attempts were made to raise the platinum level in our active, carbon-containing cathodes to as high as 10 mg/cm^2. This was achieved both by post- and pre-catalyzing methods. Pre-catalyzing refers to addition of noble metal to the active carbon prior to electrode fabrication, while post-catalyzing refers to the addition of noble metals to fabricated electrodes. Significant improvement in cathode potential was achieved, particularly with electrodes prepared with pre-catalyzed carbon. However, the improvement was still less than that required to fulfill the contract voltage requirements. Furthermore, as described previously, incorporating electrodes of this type into larger (1/8 ft^2) cells soon demonstrated severe physical degradation attributable to carbon oxidation at 90° to 100°C.

Consequently, during a seven-month period (April through October of 1967) an attempt was made to develop a high-platinum loaded cathode containing no active carbon. Some very promising results were obtained but, because of funding limitations, a directive was issued by NASA to discontinue work on this project before it had reached completion. It was planned to concentrate most of the work on the T-3N structure; the T-5 (which was more easily made) was intended primarily as a means for screening catalyst-mix variables.

First attempts to make T-3N cathodes involved application of the active mix to the coarse side of a dual-porosity nickel plaque. This particular approach seemed attractive since the coarse porous structure would serve as a good bonding surface. The electrodes of this type were further strengthened by pressing a prerolled, Teflon-bonded, inactive carbon layer to the back of the electrode. This helped force the active mix into the coarse, porous structure, and provided a highly repellent layer on the gas side of the electrode.

It was found that pressing the mix completely through the coarse-pored layer so that it would meet the fine-poored layer and form a proper interface was largely unsuccessful. Typically, electrodes made in this manner showed void areas in the coarse-pored layer between the mix and the fine-pored layer. As a result, excessive gas pressure could degrade performance by driving liquid out of the larger pores. However, in spite of this, several electrodes performed quite well with potentials in or near the American Cyanamid LAB-40 cathode range.

One modification of the metal support structure gave extremely encouraging results. Instead of attempting to bond the active mix to a dual-porosity structure, a special backing was made which consisted of nickel screen bonded to a fine-pored nickel plaque. Application of the mix to the screen side of the plaque permitted it to be pressed directly against the fine-pored layer. Using this method, considerable success was experienced in making electrodes with good working potentials; however, results were not always consistent—as will be discussed later.

Although the nickel-backed electrodes showed promising results on short-term testing, there was concern that nickel corrosion could cause degradation on longer term testing, and consequently we were directed to transfer effort from porous nickel to porous silver. Diferences in the physical properties of silver and nickel were found to greatly alter the fabrication variables, and this particular phase of the work was experiencing difficulty at the time the project was terminated.

The question of "extenders," mixed with the platinum black, was given considerable attention. Straight platinum black without an extender of some sort was found to give poor results. The most satisfactory material tested was carbon black, and this was used extensively throughout the program. Near the end of the program at NASA's request, attempts were made to substitute other materials for carbon black. The most successful substitutions were achieved by co-precipitation of silver and platinum black from ammoniacal solution with KBH_4. Excellent potentials were obtained at 50 ASF, even on porous silver, but not enough work had been done to improve potentials to the point where the electrodes would operate well at 200 ASF.

3.1.1.2 Test Procedures

Before discussing electrode types in more detail, and prior to presenting experimental results, the method of testing electrodes will be described. Small "quick-test" cells of 2 cm^2 active area were employed for each test. Cathodes to be tested were mounted by injection molding into polyethylene frames. A typical cell is shown in Fig. 1. This construction consisted of a Teflon cell body which formed the electrolyte cavity onto which was placed the framed cathode. On the other side was positioned a similarly framed Union Carbide anode which served as a counter-electrode. To complete the cell, machined Lucite pieces were placed against the back of each electrode

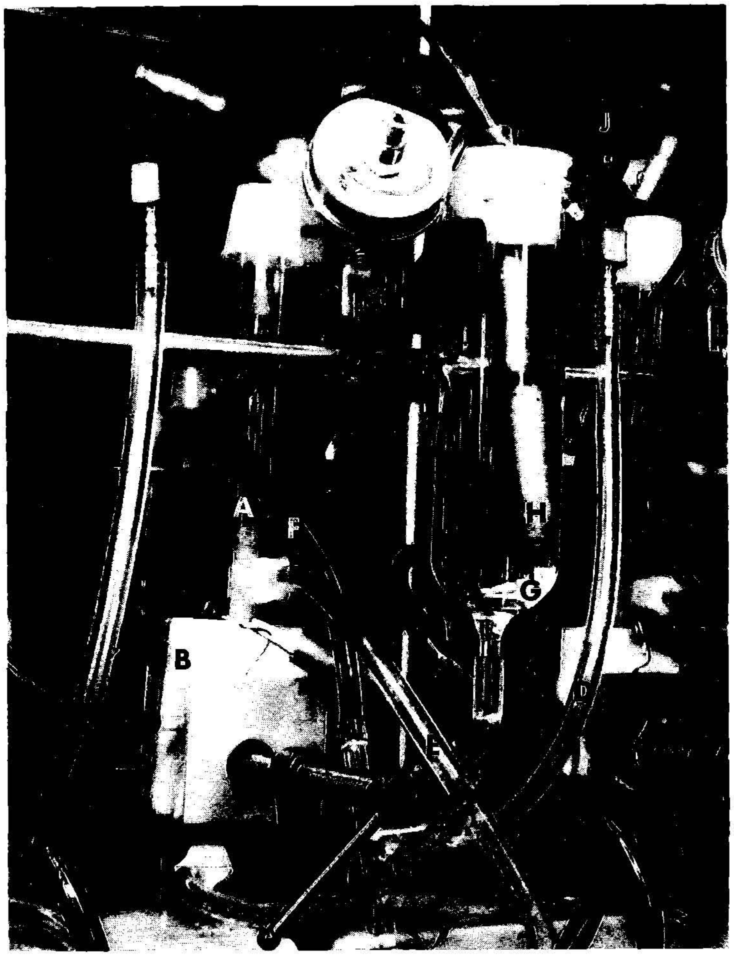

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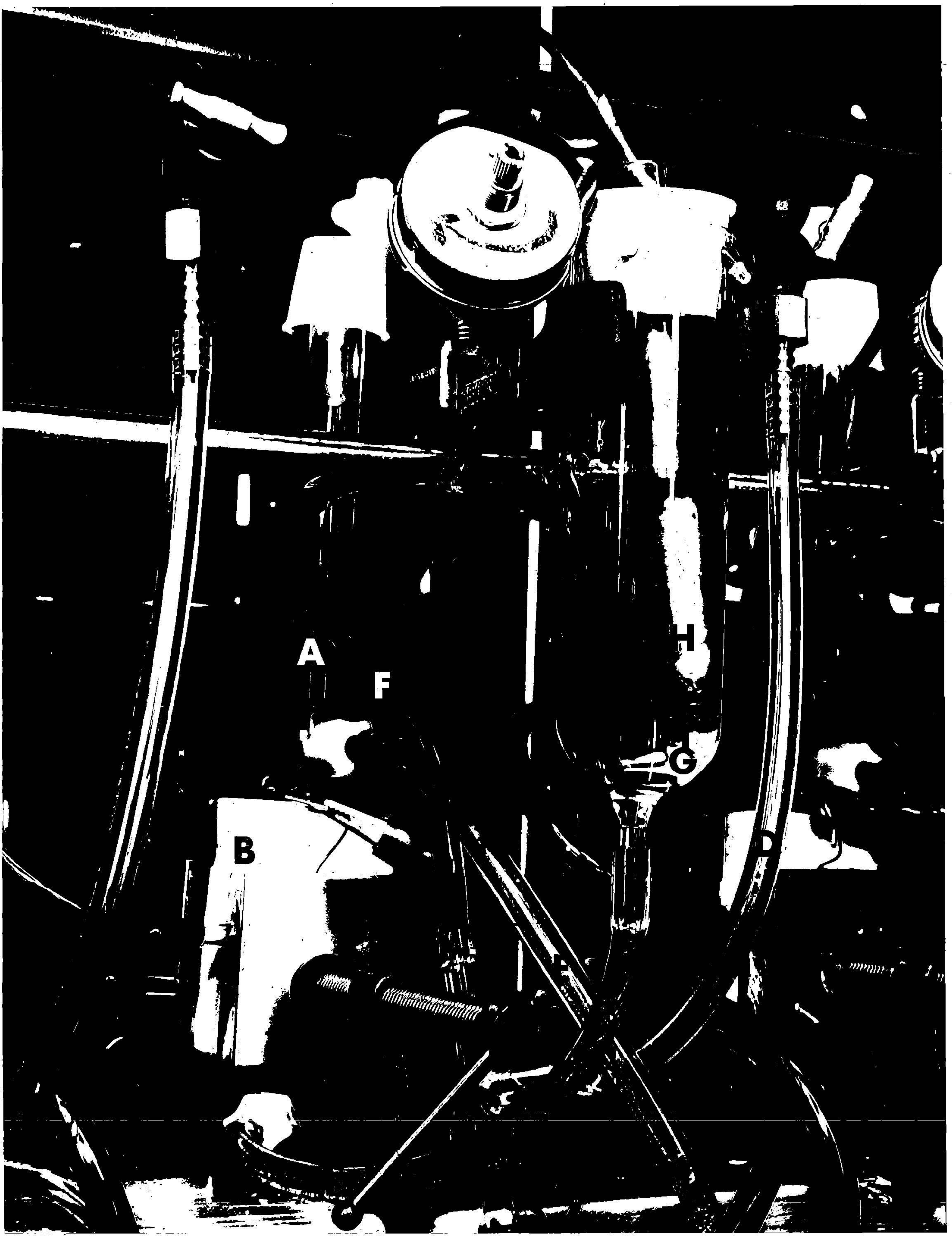
Fig. 1 - Close-up of Single Cell (2 cm^2).

A	KOH Head Tube
B	Cell
C	KOH Inlet Tube to Cell
D	Nitrogen Supply Line
E	Cell Overflow Line
F	Line from Overflow Port to Reservoir
G	KOH Reservoir
H	Heater Sheath
J	Nitrogen Manifold


A
F
H
G
B
D

and the entire unit clamped together. These Lucite pieces served as gas cavities and were provided with inlet and outlet gas tubes. Each cell was provided with a glass electrolyte reservoir into which was inserted a nickel-sheathed cartridge heater. Electrolyte circulation was achieved by means of a nitrogen "bubble-pump" arrangement. A total of 22 test positions (two banks of eleven each) were available for this program, and provisions were made for operating the cathodes under gas back-pressures as high as 5 psig. Since the anode was used only as a counter-electrode, it was normally operated at atmospheric pressure.

In testing electrodes, the cells in each bank were electrically connected in series to a 15-volt power supply which served to "smooth out" current fluctuations. In addition, a switching arrangement was provided so that each cell could be separately isolated from the bank and polarized with a Kordesch-Marko bridge. IR-free cathode potentials were measured versus a reference electrode. Although the larger NASA cells were designed for operation at 90 - 100°C, the glass reservoirs used in these electrode screening setups dictated that a lower temperature be used; thus, a temperature of 80°C was selected for this screening program.

3.1.1.3 American Cyanamid LAB-40 Cathodes

To set a target for potentials to be reached by our experimental high-Pt cathodes, American Cyanamid LAB-40 (40 mg Pt/cm^2) were chosen as a comparison standard. Electrodes of this type, tested under conditions similar to those under which our experimental electrodes were routinely screened, gave values which are summarized in Table IV. Based on these tests, a desired voltage level of +0.010 to +0.020 volt vs. Hg/HgO was selected for the experimental cathodes.

TABLE IV

PERFORMANCE OF AMERICAN CYANAMID LAB-40 CATHODES

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrode)

Series of 4 Tests:	1st Day: -0.008 to +0.018 volt
	Peak: +0.012 to +0.028 volt
	2 Weeks: +0.010 to +0.028 volt

3.1.1.4 Active Carbon Cathodes with High-Platinum Loading

3.1.1.4.1 Post-Catalyzed T-5 Cathodes

First attempts at increasing the platinum level in our cathodes consisted of post-catalyzing them in a standard manner except that larger amounts of platinum were used. A total of ten tests in this experiment are summarized in Table V; seven at a 5 mg Pt/cm^2 level; three at 10 mg Pt/cm^2. Although results at the 5 mg level were good in terms of peak potential values, the improvement observed as compared with cathodes containing less Pt (e. g., 1 mg/cm^2) was not sufficient to make this method appear attractive. Furthermore, in most cases potential degradation was excessive over the test period.

Included in this series were several fabrication variables, including binder level, wetproofing, and active-layer thicknesses. No discernible trends in performance resulted from these variations in fabrication procedure.

3. 1. 1. 4. 2 Rolled, Precatalyzed T-5 Cathodes

Two types of T-5 cathodes were made using platinum precatalyzed carbon—a rolled structure, and a sprayed structure. The former type was prepared by rolling a carbon-Teflon mix into an expanded-metal screen. Fourteen electrodes of this variety are reported in Table V. Fabrication variables included varying Pt:C ratios, binder levels, wetproofing, and presence or absence of a porous-Teflon backing. The platinum level could only be approximated, but was estimated to be between 5 and 10 mg/cm^2, depending upon whether a weight ratio of 1 Pt:2C or 1 Pt:1C was used.

Although results were more promising than those obtained with postcatalyzed electrodes, and several of the electrodes showed good voltage stability over the test period, performance was still below the desired level of +0. 010 to 0. 020 volt.

3. 1. 1. 4. 3 Sprayed, Precatalyzed T-5 Cathodes

Electrodes similar to the preceding type were prepared by spraying a precatalyzed carbon-Teflon slurry onto a base which usually consisted of an expanded-metal grid into which had been rolled a highly wetproofed, inactive-carbon layer. Table V also summarizes the results obtained in 16 tests with electrodes of this type. Variables included in this series were similar to those included in Sect. 3. 1. 1. 4. 2 above. The data show that these electrodes performed, in general, about the same as the rolled type.

3. 1. 1. 5 High-Platinum Loaded Cathodes without Active Carbon

3. 1. 1. 5. 1 All-Platinum, T-5 Cathodes

Initial attempts to make all-platinum cathodes employed a

TABLE V

PERFORMANCE OF T-5 CARBON CATHODES WITH INCREASED PLATINUM LOADINGS

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrode)(1)

Post-Catalyzed Cathodes; 5 mg/cm^2 Level (7 Tests):

All had peak potentials of -0.020 v, or better, vs. Hg/HgO

Three taken off after 3 days because potential dropped below -0.040 v

Remaining four tests: 1st day, -0.026 to -0.004 v
Peak, -0.014 to -0.004 v
2 weeks, -0.059 to -0.020 v

Post-Catalyzed Cathodes; 10 mg/cm^2 Level (3 Tests):

None reached potentials of -0.020 v

Rolled, Precatalyzed Cathodes (14 Tests):

Four never reached potentials of -0.020 v

One reached -0.020 v on only one day during 2-week test period

One dropped to -0.080 v in 3 days

Remaining eight tests: 1st day, -0.025 to -0.002 v
Peak, -0.017 to +0.006 v
2 weeks, -0.044 to -0.002

Sprayed, Precatalyzed Cathodes (16 Tests):

Four never reached potentials of -0.020 v

One reached -0.020 only one day during 2-week test period

One dropped to below -0.400 v in 4 days

One dropped to -0.034 v in 11 days

Remaining nine tests: 1st day, -0.040 to -0.010 v
Peak, -0.016 to +0.007 v
2 weeks, -0.042 to -0.015 v

(1) Goal: +0.010 to +0.020 volt vs Hg/HgO

modified T-5 structure in which the platinum black mix was applied to a support consisting of porous-Teflon bonded to gold-plated nickel screen. Although the T-3 electrode structure was the main objective, it was felt that the preliminary screening of variables could be most conveniently accomplished with the more easily made T-5's.

Numerous mix formulations were tested with consistencies ranging from a paint to a "butter," and the method of application varied accordingly. In the case of mixes to be painted, care was taken to prevent coagulation of the Teflon which was used as a binder, and thickening agents such as sodium carboxymethylcellulose or polyacrylimide were sometimes used. When a spreadable "butter" was desired, the Teflon was deliberately coagulated with a suitable organic solvent.(e. g., "Solvesso" 100, a Standard Oil Product).

Because of the high cost of platinum, very small batches of mix were made; just enough for a 2" x 2" piece. Since the platinum loading was 40 mg/cm^2 in most cases, 1 gram of platinum black was required for each batch to cover a 4 in^2 (26 cm^2) electrode.

With the exception of the earlier tests in which platinum black precipitated from platinum chloride was used (designated as UCC precipitated Pt), electrodes were made almost exclusively with material obtained from Engelhard Industries, having a crystallite size of 90-100 angstrom units. Data from a total of 55 tests were obtained with electrodes of this type. Most of these were single tests, although 20 of them represent duplicate runs on 10 electrodes. Thus, this group represents the testing of 45 different electrode formulations. Table VI summarizes the results.

TABLE VI
PERFORMANCE OF ALL-PLATINUM T-5 CATHODES

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrodes)		
Series of 55 Tests:	29 Never reached potentials of -0.020 volt	
	6 were taken off test in less than a week for various reasons	
	Remaining 20 tests:	1st Day, -0.064 to +0.012 volt
		Peak, -0.016 to +0.012 volt
		1 Week, -0.049 to -0.001 volt
		2 Weeks (7 Tests only), -0.046 to -0.015 volt

Although several tests approached the LAB-40 baseline with respect to peak potential value, they maintained these values for only a short while and were generally unacceptable.

In spite of these mediocre results, several relevant pieces of information were obtained, particularly with respect to type and amount of Teflon binder needed and the cure temperatures. Both FEP and TFE Teflon were tried with almost identical results (See Table VII).

TABLE VII

EFFECT OF BINDER VARIATIONS ON CATHODE POTENTIAL
(T-5 Electrodes, 12 M KOH, 80°C; Results after 2 days on test at 200 ASF)

	IR-Free Potentials vs Hg/HgO
FEP Teflon (Engelhard Pt):	
10% (No. 5-25-67-E)	-0.020 volt
25% (No. 5-25-67-H)	-0.001 volt
TFE Teflon (UCC Precipitated Pt):	
10% (No. 5-19-67-B)	-0.012 volt
15% (No. 5-19-67-C)	-0.004 volt

Various curing temperatures were also tested (See Table VIII).

TABLE VIII

EFFECT OF CURING CONDITIONS ON CATHODE POTENTIAL
(T-5 Electrodes, 12 M KOH, 80°C; UCC pptd. Pt; Results after 2 days on test at 200 ASF)

	IR-Free Potentials vs Hg/HgO
10% TFE (No. 5-19-67-B):	
a. 200°C - overnight	-0.012 volt
b. ~275°C - 1/2 hour in air	-0.024 volt
c. 275°C - 4 hrs. total in N_2 (time from ambient temp. to 275°C. ~2 hrs. in 265° -275°C range)	-0.043 volt
15% TFE (No. 5-19-67-C):	
a. 200°C - overnight	-0.004 volt
b. ~275°C - 1/2 hour in air	-0.007 volt
c. 275°C - 4 hrs. total in N_2 (time from ambient temp. to 275°C ~2 hrs. in 265° -275°C range)	-0.012 volt

These data indicated the 15% binder level to be superior to 10%. Furthermore, the higher binder level permitted a higher curing temperature to be used with less detrimental effect on cathode potential. This was considered to be desirable in terms of electrode strength.

3.1.1.5.2 T-3N Cathodes

The fabrication of T-3N electrodes was found to present unique problems not encountered with the T-5 variety. Two general electrode types were made and tested: (1) the mix was applied to the coarse side of a dual-porosity nickel or silver plaque, and (2) the mix was applied to an alternate porous-metal product in which the coarse-pored layer was replaced by an open-mesh screen bonded to the fine-pored plaque.

(a) Cathodes with Dual-Porosity Nickel

A considerable number of electrodes was made in which dual-porosity nickel was used as the support layer. In many cases, the nickel was gold-plated in an attempt to provide some corrosion stability, but penetration of the gold plate was found to be unsuccessful, and the coverage was largely superficial. Table IX presents a summary of 81 tests, covering 69 different electrodes (only 12 were run in duplicate).

During this time, little success was achieved using only platinum black (40 mg/cm^2) as an active material. Best performance was obtained with electrodes containing carbon black as an extender, with the usual weight ratio being 10 parts platinum and 1 part carbon black. The carbon black is a fully graphitized structure with a "c-axis" spacing of 3.36 Å. It is resistant to oxidation at temperatures well above those experienced in operating fuel cells. The platinum level in all but a few electrodes tested was 40 mg/cm^2.

The general procedure for preparing electrodes of this type was to paint the platinum mix onto the coarse-pored side of the dual-porosity nickel plaque, dry it at a low temperature (ca. 50°C) between coats, and then lay a FEP-painted inactive carbon layer on top of this. The electrode was then pressed, and then cured in a nitrogen atmosphere. In these, and in the following type of electrodes, variables such as Teflon type, pressure, and cure temperature were examined. Details will be discussed subsequently.

The chief disadvantage of the dual-porosity nickel matrix was the fact that it was virtually impossible to press the active mix completely through the thickness of the coarse-pored structure. As a consequence, excessive gas pressure could drive liquid from the resultant void spaces during operation and thus culminate in unusual and unpredictable reactions which normally resulted in performance degradation.

TABLE IX

PERFORMANCE OF T-3N CATHODES WITH DUAL-POROSITY NICKEL MATRIX

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrodes)

<u>All-Platinum Cathodes:</u>

16 Tests (14 different electrodes) were run.

None reached a potential of -0.020 volt.

<u>Platinum Black-Carbon Black Cathodes:</u>

A total of 65 tests (53 different electrodes) was run:

23 Tests (20 different electrodes) never reached a potential of -0.020 volt.

20 Tests (18 different electrodes) reached -0.020 v at least once, but were removed from test in less than 1 week for various reasons.

13 Tests (12 different electrodes) reached -0.020 v, but did not attain the desired level (+0.010 v, or better)

1st Day,	-0.019 to +0.008 v
Peak,	-0.006 to +0.009 v
1 Week,	-0.048 to +0.006 v

8 Tests (8 different electrodes) reached the desired level (+0.010 v or better) at least once:

1st Day,	-0.001 to +0.017 v
Peak,	+0.010 to +0.022 v
1 Week,	-0.006 to +0.012 v

1 Test reached the desired level (+0.010 v, or better) for one week*:

1st Day,	-0.053 v
4th Day,	+0.016 v
12th Day,	+0.016 v

* Potentials above +0.010 v were obtained from the 4th day through the 12th day.

A further disadvantage resided in the nonuniformity of the dual-porosity nickel itself. Consequently, a fabrication pressure suitable for one particular lot might not be satisfactory for the next.

(b) Cathodes with Nickel Screen-Porous Nickel Base

Because of the foregoing disadvantages, a modified nickel matrix was developed which consisted of a nickel screen sintered to the fine-pored nickel layer. Electrode fabrication procedures were similar, but these electrodes proved to be distinctly superior in that the mix could be pressed down to contact the fine-pored layer. Bonding (particularly to the wires) was not always as strong as desired, and oxygen-backpressure sensitivity was not entirely consistent, but the operating voltage level of electrodes in this group showed considerable improvement over those discussed in the above.

Table X summarizes results obtained on electrodes of this type. All electrodes reached potentials of better than -0.020 volt—even several of the all-platinum ones—and a significant number performed as well as LAB-40 cathodes during their one-week test period.

Because of the very encouraging results of these tests, a more-detailed program for screening fabrication variables was set up and run. These tests were carried out with electrodes using the nickel screen-porous nickel substrate (gold plated). Major variables tested were: fabrication pressure (see Table XI); type and amount of fluorocarbon binder (see Table XII), and cure temperature (see Table XIII).

Reproducibility in these tests was poor, probably because of areas of poor screen attachment to the porous nickel (a problem which was later solved). In spite of this, several important conclusions seemed justified:

1. Results indicate the superiority of a 2000-psi fabrication pressure as compared to 4000 psi.

2. TFE appears to be superior to FEP as a binder, although the amount (in the 10-20% range) does not appear critical.

3. The injurious effect of too high a cure temperature is strongly suggested.

TABLE X

PERFORMANCE OF T-3N CATHODES WITH SCREEN* ON POROUS NICKEL MATRIX

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrode)

All-Platinum Cathodes:

A total of 6 tests (4 different electrodes) was run.
All achieved peak potentials between -0.012 to +0.006 volt but were on test less than one week.

Platinum Black-Carbon Black Cathodes:

A total of 42 tests (21 different electrodes) was run:

8 Tests (5 different electrodes) achieved peak potentials between -0.005 to +0.013 v but were on test less than one week.

16 Tests (8 different electrodes) reached -0.020 volt but did not attain the desired level:

	1st Day,	-0.014 to +0.009 v
	Peak,	-0.014 to +0.009 v
	1 Week,	-0.070 to +0.004 v

8 Tests (5 different electrodes) reached the desired level at least once:

	1st Day,	+0.008 to +0.016 v
	Peak,	+0.010 to +0.016 v
	1 Week,	0 to +0.008 v

10 Tests (3 different electrodes) reached and maintained the desired level for one week:

	1st Day,	+0.010 to +0.027 v
	Peak,	+0.016 to +0.027 v
	1 Week,	+0.010 to +0.018 v

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* Gold-Plated nickel screen.

TABLE XI

EFFECT OF FABRICATION PRESSURE ON CATHODE POTENTIAL

15% TFE-bonded Pt black-carbon black
electrodes (10:1);
12 molar KOH; 80°C; 200 ASF
O_2 back pressure = 2 psi

Fabrication Pressure (psi)		1 Day on Test IR-Free Potentials vs Hg/HgO (volts)	7 days on Test
2000	Cell 1	+0.005	+0.004
	Cell 2	+0.011	+0.004
4000	Cell 1	-0.003	-0.008
	Cell 2	-0.50 (Drifting)	-1.03 (H_2 evolution)

TABLE XII

EFFECT OF TYPE OF FLUOROCARBON ON CATHODE POTENTIAL

Fabrication Pressure: 4000 psi
Platinum black-carbon black mix (10:1)
12 molar KOH; 80°C; 200 ASF
O_2 back pressure = 2 psi

	IR-Free Potentials vs Hg/HgO (volts)					
	10 Wt. % Fluorocarbon*		15 Wt. % Fluorocarbon*		20 Wt. % Fluorocarbon*	
Fluorocarbon Type	No. of Days on Test 1	7	No. of Days on Test 1	7	No. of Days on Test 1	7
TFE - Cell 1	+0.002	+0.006	-0.003	-0.008	+0.012	+0.010
Cell 2	+0.009	+0.008	---	---**	0	0
FEP - Cell 1	-0.012	-0.009	-0.020	-0.010	-0.014	-0.004
Cell 2	-0.014	-0.010	-0.016	-0.010	-0.050	-0.042

*Percentages by weight based upon platinum plus Teflon content only; other ingredients being ignored.

**Questionable replicate.

TABLE XIII

EFFECT OF CURE TEMPERATURE OF FLUOROCARBON TYPES UPON CATHODE POTENTIAL

Fabrication Pressure: 4000 psi
Platinum black-carbon black mix (10:1)
12 molar KOH; 80°C; 200 ASF
O_2 back pressure = 2 psi

	IR-Free Potentials vs Hg/HgO (Volts)					
20 Wt. % Flurocarbon Type	225°C Cure Temp.		275°C Cure Temp.		325°C Cure Temp.	
	No. of Days on Test 1	7	No. Days on Test 1	7	No. of Days on Test 1	7
TFE - Cell 1	+0.004	+0.004	-0.003	-0.008	-1.03 (H_2 evolution)	--
Cell 2	+0.003	-0.006	--	--*	-0.062	-1.02 (H_2 evolution)
FEP - Cell 1	-0.009	-0.006	-0.020	-0.010	-0.034	-0.026
Cell 2	-0.028	-0.068	-0.016	-0.010	-1.04 (H_2 evolution)	--

* Questionable replicate.

(c) Cathodes with Silver Screen-Porous Silver Base

Attempts to make electrodes using the "standard" platinum black-carbon black mix on silver were not successful. The chief physical differences between this support and the nickel support resided in hardness of material and geometry of the structure. The latter included differences in mesh and wire sizes of the screens used as well as pore size of the fine-pored plaques.

In view of these facts, the chief variable tested was fabrication pressure. Electrodes were pressed in the range of 500-4000 psi without success. Other variables considered included the method of mixing the "paint," the drying of the paint coats between applications, and the addition of porous Teflon as a backing. In a group of 14 tests, only three reached potentials in the range of -0.100 to -0.020 volt versus Hg/HgO. No satisfactory explanation for the poor performance was obtained prior to termination of this portion of the work.

(d) Filtered Electrodes

The idea that a workable T-3N cathode could be made by pressure and/or vacuum filtration of a suitable mix into the structure of a dual-porosity silver plaque was investigated to a limited extent. A special filtration fixture was built which permitted the simultaneous application of vacuum below the porous-metal piece and pressures as high as 60-psi above it.

It was found that a slurry of platinum black (or Pt black + carbon black) could be filtered fairly well; however, difficulty in ending up with the active material in the body of the coarse layer in contact with the fine-pored layer was experienced, and necessitated use of a plaque with a very coarse layer sintered to the fine-pored structure.

The most successful electrodes were prepared by pre-wetproofing the platinum black-carbon black mix with FEP Teflon prior to filtration. The use of FEP instead of TFE Teflon permitted the wet-proofed material to be repulverized so that a filterable slurry could be made. For these electrodes, the slurry was prepared by mixing 1.2 g of the wet-proofed material with 25 cc of H_2O and stirring at high speed with a "VirTis" homogenizer. The slurry was then further diluted to 500 cc, stirred moderately, and filtered through a dual-porosity silver plaque. A pressure differential of 75 psi was used during filtration. As a final step, a layer of FEP-painted carbon was pressed over the active mix using a 4000-psi pressure. After air drying overnight, the piece was cured at 250°C in a nitrogen atmosphere.

Although 200 ASF potentials were poor, peak potentials for these two tests (+0.032, +0.045 at 50 ASF, and -0.009, +0.010 at 100 ASF) indicated the ultimate feasibility of this method.

(e) Tests with Noncarbon Extenders

At the termination of this phase of the effort, work was being concentrated on the use of noncarbon extenders with platinum black, with particular emphasis upon silver. Both nickel and silver substrates were used, and the extenders tested included (in addition to silver) TiO_2 and ZrO_2.

A series prepared by using the nickel screen-porous nickel substrate with different pressure and binder variables gave no usable electrodes at 200 ASF. In most cases potentials were erratic, and only the peak values are reported in Table XIV.

TABLE XIV

POTENTIAL OF CATHODES USING NON-CARBON EXTENDERS WITH PLATINUM BLACK-NICKEL SCREEN-POROUS NICKEL SUBSTRATE

12-M KOH; 80°C; 200 ASF (Potentials Referred to Hg/HgO Electrodes)

Extender	Extender/Pt Ratio	% TFE	Pressure (psi)		Peak Potentials
Ag powder	10:6	15	1000	(Test 1)	-0.032
				(Test 2)	-0.030
Ag powder	10:6	20	500	(Test 1)	-0.011
				(Test 2)	-0.024
Ag powder	10:3	15	250		-0.036
			500		-0.020
			1000		-0.025
Ag powder	10:3	20	250		-0.028
			500		-0.018
			1000		-0.022
TiO_2	10:2	15	1000	(Test 1)	-0.028
				(Test 2)	-0.022
TiO_2	10:2	20	500	(Test 1)	-0.168
				(Test 2)	-0.128
TiO_2	10:1	15	250		-0.013
			500		-0.119
			1000		-0.115
TiO_2	10:1	20	250		-0.102
			500		-0.115
			1000		-0.058
ZrO_2	10:3	15	1000	(Test 1)	-0.020
				(Test 2)	-0.024
ZrO_2	10:3	20	500	(Test 1)	-0.036
				(Test 2)	-0.032

In spite of the poor results, several of the tests merit further attention, particularly those with TiO_2 and ZrO_2. It is believed that considerable improvement is possible if particular attention is given to optimum particle size (it is surmised that that used was too coarse) of the extenders, in combination with other fabrication variables.

An interesting use of silver as an extender gave electrodes with excellent potentials at 50 ASF, and certainly deserves further study. This method consists of co-precipitating silver and platinum black with potassium borohydride. Data obtained with electrodes of this type on a silver substrate are presented in Table XV.

TABLE XV

POTENTIALS OF ELECTRODES PREPARED WITH CO-PRECIPITATED SILVER- AND PLATINUM-BLACK SILVER SCREEN/POROUS SILVER SUBSTRATE

12-M KOH, 80°C; Potential Referred to Hg/HgO Electrode;
All Electrodes Contain 40 mg (Pt + Ag)/cm^2

Pt:Ag Ratio	% TFE	Pressure (psi)		Peak Potential at 50 ASF	Indicated Stability
1:1	15	1000		+0.053	Good
1:1	15	4000		+0.012	Poor
1:1	25	1000		+0.060	Good
10:1	25	1000	(Test 1)	+0.031	Good
			(Test 2)	+0.015	Poor

Test periods were short (3 days maximum) but several of the best electrodes appeared to be quite stable over this period at the 50 ASF level. It is interesting to note that decreasing the Pt:Ag ratio from 10:1 to 1:1 (using the same total amount of catalyst in the electrode) actually increased the electrode potential at 50 ASF.

3.1.2 LAB-40 Electrode Evaluation

The LAB-40 electrode has been described previously (see footnote on page 4). Early versions of the LAB-40 electrode when tested in 1/8 ft^2 cells showed delamination of the electrode backing (e.g., Cell A-018). This problem was discussed with American Cyanamid who instituted several changes in the composition and/or bonding procedure for attaching the "L" fluorocarbon layer to the AB-40 active layer. These various modifications are described by a letter (A, B, or C) indicative of the chemistry of the backing, a Roman numeral (I or II) indicative of the processing procedure and some times an Arabic numeral 1, 2, 3 or 4 describing minor fabrication process modifications. These changes were initially successful in eliminating delamination. For example, Cell A-051, whose performance will be discussed in detail subsequently, was subjected to 32 thermal cycles from 35 to 90°C with no evidence of backing failure. Later in the Task III work, a recurrence of this problem was experienced as documented in Table XVI, and additional testing work was undertaken both by Union Carbide and American Cyanamid to understand and eliminate the problem. Ultimately, the so-called C-II backing evolved which in its final modification proved to be excellently bonded to the active layer. Peel tests of electrodes after extended operation in cells indicated that separation of this backing from the active layer did not occur. Rather the electrode tore apart within the active layer generally along the current collection screen.

Analysis of the data summarized in Table XVI indicates that the A-II-2 backing modification used in both 1/8 ft^2 cells and in larger cells (e. g., A-214 and A-215) was particularly prone to delamination. Delamination occurred in practically every instance where this material and fabrication treatment were employed. In contrast, the B-II* backing modification used in small cells was generally adequately bonded (Note Cells A-116, A-118 and A-123). However, the similar B-II-4 backing used in Cells A-109, A-110, and A-117 was variable in quality. These B-II-4 electrodes were prepared by cutting down ~7-1/2 x 7-1/2 electrodes originally procured for flight-size cells to the 4-1/2 and 5" size required in the 1/8 ft^2 cells. Since the B-II backing prepared specifically for 1/8 ft^2 did not delaminate while the B-II-4 backing intended for the flight-size cells did, the variability is likely associated with process scale-up.

This hypothesis was further confirmed by an experiment involving cells A-109 and A-110. The cathodes for these two cells were selected on the basis of visual appearance. The cathode in A-109 was judged to be well-bonded based on the observation that the pattern of the metal current collection screen was obvious in the porous fluorocarbon backing suggesting that the electrode has been adequately pressed during fabrication. In A-110, on the other hand, the pattern

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*According to American Cyanamid, B-II and B-II-4 are essentially identical.

TABLE XVI

OBSERVATIONS ON THE BOND INTEGRITY IN CELLS WITH LAB-40 ELECTRODES

Cell	Anode	Cathode	Comments	Separator	Total Hours on Test
A-105	LAB-40 (A-II-2,S7332 103-13)	LAB-40 (A-II-2,S7332 102-6)	Autopsy indicated delamination of electrodes in isolated areas.	Serpentine	1034
A-107	T-2	LAB-40 (A-II-2*,S8333-119-9)	Observed cathode delamination at 385 hrs. Confirmed by autopsy.	Serpentine	507
A-109	T-2	LAB-40 (B-II-4,568-1D)	Cathode appeared okay.	Serpentine	1648
A-110	T-2	LAB-40 (B-II-4,568-2B)	Observed cathode delamination after 600 hrs.	Serpentine	1445
A-111	LAB-40 (A-II-2,S8333-119-4)	LAB-40 (A-II-2,S8333-119-8)	Pressure cell suspected delamination of anode by 48 hrs and cathode by 380 hrs. Autopsy showed delamination of both electrodes.	Flattened TFE Mesh	752
A-112	LAB-40 (B-II-4,565-5A)	LAB-40 (B-II-4,565-7B)	No test, shorted electrodes.	Flattened TFE Mesh	0
A-113	LAB-40 (A-II-2,S8333-119-1)	LAB-40 (A-II-2,S8333-119-5)	Cathode delamination during first 24 hrs. (Autopsy confirmed this and also showed anode delamination).	Serpentine	119
A-212	T-2	LAB-40 (B-II,S8333-12)	Autopsy showed delamination of cathode	Serpentine	1985
A-214	LAB-40 (A-II-2,541-9B)	LAB-40 (A-II-2*,540-2A)	Delamination of both electrodes in 3 hrs. Autopsy confirmed this.	Serpentine	3
A-215	T-2	LAB-40 (A-II-2,541-9A)	Observed cathode delamination 400 hrs. Autopsy confirmed this.	Serpentine	436
A-114	T-2	LAB-40 (A-II,S8333-119-3)	Observed cathode delamination at 658 hrs.	Serpentine	912
A-115	LAB-40 (A-II-2,S8333-119-6)	LAB-40 (A-II-2,S8333-119-2)	Autopsy shows anode delamination. Cathode okay.	Serpentine	1846
A-116	T-2	LAB-40 (B-II,S8333-120-1)	No observable delamination.	Serpentine	1222
A-117	LAB-40 (B-II-4,565-7A)	LAB-40 (B-II-4,565-3B)	Autopsy showed anode backing very poorly bonded. Cathode okay.	Serpentine	338
A-118	T-2	LAB-40 (B-II,S8333-120-2)	No observable delamination.	Serpentine	72
A-119	LAB-40 (C-II,S8505-105-2)	LAB-40 (C-II,S8505-105-3)	No observable delamination.	3 pc flattened TFE	1024
A-120	LAB-40 (C-II,S8505-106-3)	LAB-40 (C-II,S8505-106-2)	No observable delamination.	3 pc flattened TFE	1351
A-121	LAB-40 (C-II,S8505-111-1)	LAB-40 (C-II,S8505-111-2)	Cell shorted during subassembly. Electrodes used for blowthrough tests.	3 pc flattened TFE	0
A-122	LAB-40 (C-II,S8333-121-2)	LAB-40 (C-II,S8333-121-3)	No observable delamination.	3 pc flattened TFE	1392
A-123	T-2	LAB-40 (B-II,S8333-120-3)	No observable delamination.	3 pc flattened TFE	1292
A-125	LAB-40 (C-II,S8806-20-3-1)	LAB-40 (C-II,S8806-20-1)	No observable delamination.	Horizontal Serpentine + Polysulfone Mesh	386
A-126	LAB-40 (C-II,S8806-20-3-3)	LAB-40 (C-II,S8806-20-2-2)	No observation possible.	Horizontal Serpentine + Polysulfone Mesh	44
A-127	T-2	LAB-40 (C-II,S8806-20-2-4)	No observable delamination	Horizontal Serpentine + Polysulfone Mesh	(1100)

*Note: The electrodes in Cells A-107, A-111 and A-113 were procured on Union Carbide P.O. 742-06601 after July 1, 1967. These electrodes were verbally described by the vendor as having the generic designation A-II and that the Arabic postscript had been deleted. However, the electrodes as received were actually designated as A-II-2. The "A-II-2" electrodes in Cells A-214 and A-215 were procured prior to July 1, 1967 on Union Carbide P.O. 742-07660 and presumably do not reflect process improvements incorporated into the later A-II electrodes.

of the screen was not visible in backing. As can be seen in Table XVI, cell A-109 operated for 1,648 hours without delamination, while backing separation occurred in the Cell A-110 cathode after 600 hours on test.

No evidence of delamination occurred in any of the cells using various lots of electrodes with the C-II backing modification. Since all testing was conducted on 1/8 ft^2 electrodes, the one question not answered in the course of this work is whether or not significant variability would exist in flight-size electrodes with the C-II backing.

During the course of this evaluation, some attention was also directed to the possibility that the design of the gas space separators might be contributing to delamination. Consequently, cells were constructed both with serpentine separators and with plastic-mesh separators in the H_2 and O_2 spaces. With the plastic mesh, the supported area on the backing is much greater than with the serpentine. Although the comparative tests are not completely conclusive, separator design does not appear to be a major factor. Cells with mesh separators delaminated (e.g., A-111), while cells with serpentine separators survived the testing regimen intact (e.g., A-109).

A supplementary experimental program was instituted in Task III in an attempt to delineate some of the causes of LAB-40 electrode delamination and to develop possible quality control tools which would reduce process variability. To this end, American Cyanamid devised a peel test in which the load required to strip the "L" backing from the LAB-40 active layer was quantitatively measured in an Instron tensile machine. This approach did not appear to provide any correlation with service performance or fabrication history. In almost every instance, the peel test failure occurred within one of the layers rather than at the interface between the backing and the active layer.

Concurrently, Union Carbide developed a simple blister test. In this test a 5/8" disk of the LAB-40 electrode was clamped in a fixture and gas pressure was applied from the active side of the electrode. The pressure was gradually increased in 2-psi increments until delamination occurred. In this test delamination almost invariably occurred at the interface between the active layer and the backing. Various electrodes were tested: 1) in the as-received condition; 2) after a one or two week soak in 100°C KOH; 3) after various thermal cycles in air to 100°C, and 4) in some cases after operation in small (2 cm^2)

electrochemical cells. Data accumulated from these tests are summarized in Table XVII. From the data it may be observed that the blister or delamination

TABLE XVII

BLISTER PRESSURE OF ELECTRODES(1)

Electrode No.	As Rec'd	KOH Soak at 100°C 1 wk	KOH Soak at 100°C 2 wk	Thermal Cycles 100°C for 1 Hour, Room Temperature for 1 Hour 5 cycles	Thermal Cycles 100°C for 1 Hour, Room Temperature for 1 Hour 10 cycles	Cells Operating at 200 ASF, 80°C 1 wk	2 wk	3 wk	4 wk	5 wk
565-3B(2)	14	4		12	20					
(B-II-4)	14	2		12	20					
	14	4								
565-7-A(3)	12	2		16	15					
(B-II-4)	17	4		16	15					
	14	10								
	22					8	10	10	10	10
	22					8	8	8	10	12
541-8C	10		5	14	13					
	12		10	14	15					
568-2B	14		--	--	--					
(B-II-4)										
565-5A	16		11	20	22					
(B-II-4)	18		15	18	18					
565-7B	10		9	8	16					
(B-II-4)	10		10	10	18					
S8505-105	18	4								
(C-II,	20	4								
Lightly	17	10								
Pressed Backing)										
S8505-107	16	12								
(C-II,	19	12								
Medium	18	12								
Pressed Backing)										
S8505-106	46	19								
(C-II,	40	14								
Heavily	44	22								
Pressed Backing)										

(1) Pressure in psig

(2) Cathode in Cell A-117

(3) Anode in Cell A-117

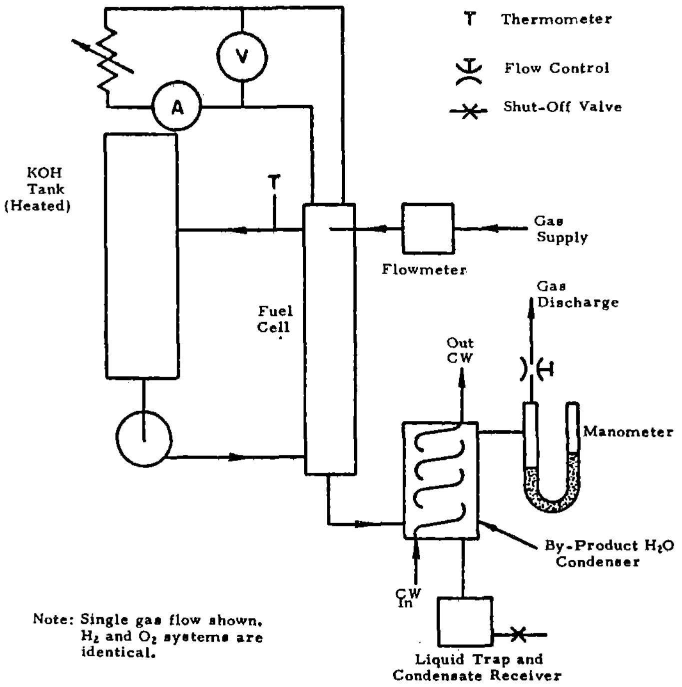
pressure was reduced substantially by exposure of the electrode to hot KOH in both static tests and in electrochemical cells. In contrast, thermal cycling in air, produced slight increases in blister pressure. Further testing is required before this procedure can be used for quality control purposes. However, based on the successful performance of cells with the C-II backing modification, it appears that blister pressures of > 20 psi should provide delamination-resistant electrodes.

3.2 Cell Testing

3.2.1 Facilities

Under the initial terms of Contract NAS3-9430 Union Carbide agreed to provide 18 single cell test positions of which 12 were to accommodate small cells (defined as approximately 3" x 3" or 6" x 6") and six were to take so-called flight-size cells which were to have at least 1 ft^2 of active electrode surface. This commitment required fairly extensive modification of the existing test facilities and considerable new construction, because the smaller size is not widely used in normal Union Carbide programs, and the larger size, though generally used, is not usually operated at the high current densities required here. Furthermore, normal Union Carbide operations are carried out at or below 70°C, which permits the use of polypropylene electrolyte tanks and plumbing, while the 100°C requirements of this program necessitated the use of nickel as the construction material.

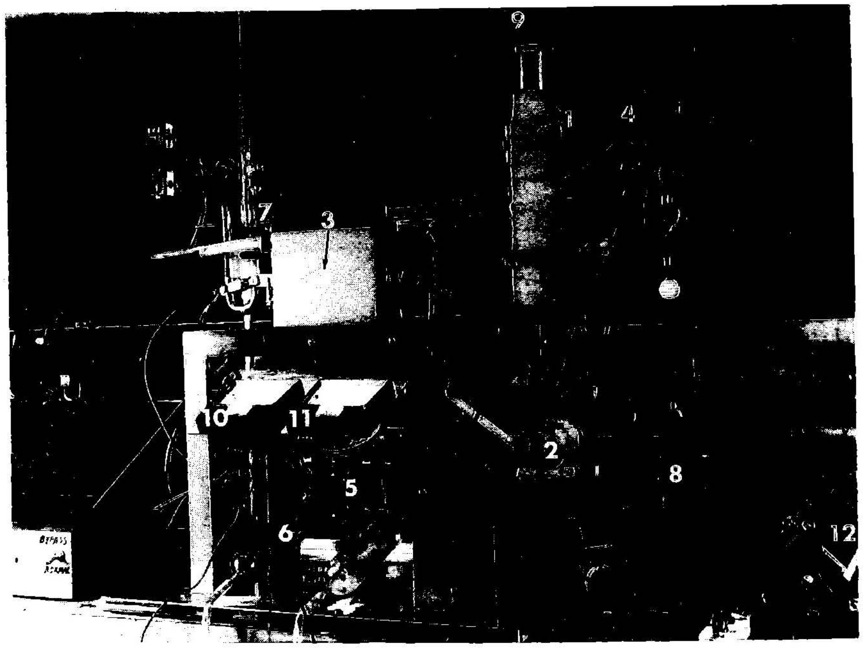
Initially, six small test stands were activated and used in the early stages of the program. Some of this equipment is illustrated in the schematic diagram of Fig. 2, and the photograph of Fig. 3.



Note: Single gas flow shown, H_2 and O_2 systems are identical.

D-3122

Fig. 2 - Schematic Diagram of NASA "Small" Cell Test, Original Design, for Atmospheric Pressure Operation.

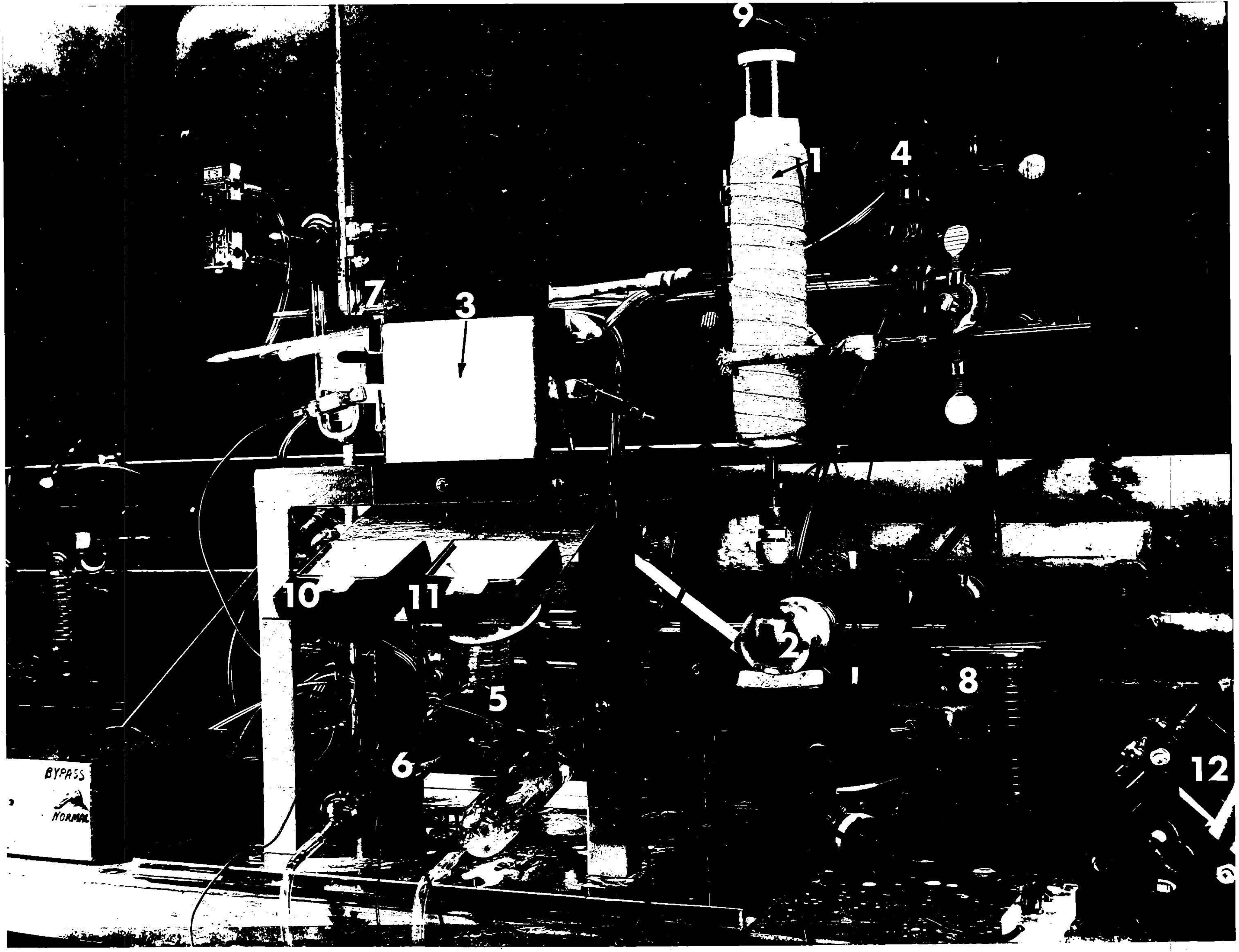


D-3119

Fig. 3 - NASA "Small" Cell Test, Original Design.

In this arrangement the KOH reservoir (1) is constructed of nickel, and heated by means of heating tape. The electrolyte is circulated with a centrifugal pump (2), entering the bottom of the cell (3), and exiting at the top from where it returns to the reservoir. Since both gas circulating systems are identical, only one is shown in the schematic, and only the oxygen loop is identified in the photograph. Each gas first passes through a flowmeter (4), then enters the cell at the top. All excess gas is purged from the system (i. e., no recirculation is provided), and leaves the cell from the bottom, the exit port being positioned diagonally across from the inlet port. The exit gas passes through a water-cooled condenser (5) which serves to remove a major portion of the water, which then collects in the condensate receiver (6). The latter also acts as a trap to detect the presence of electrolyte leakage should it occur. The exit gas finally passes through a control valve positioned on one leg of a U-tube containing mercury (7). This permits control of the gas pressure in the cell.

Also shown in the photograph is the Variac (8) used for adjusting electrolyte temperature, an electrolyte level probe (9) which automatically shuts off the hydrogen supply and puts the cell on open circuit in the event that the electrolyte level were to become dangerously low, and the components of the load circuit; voltmeter (10), ammeter (11), and rheostat (12).


9
1
4
7
3
10
11
2
8
5
6
12
BYPASS
NORMAL

In addition to five test positions of the foregoing type, one position suitable for operating at pressures up to 45 psig was built. While the contract had not specifically contemplated testing of cells significantly at very elevated pressure, early work indicated that single cell voltage gains on the order of 30 mv could be obtained by increasing the operating pressure to ∼30 psia. The schematic and photograph of this system are shown in Figs. 4 and 5. Pressure balance is achieved by simultaneous pressurization of the elctrolyte with nitrogen as the hydrogen and oxygen pressures are increased. This is easily accomplished by feeding the nitrogen to one side of the diaphragms of both the hydrogen and oxygen regulators Item 1, Fig. 5. This forces the pressure of these gases to "follow" the nitrogen pressure. As before, only one gas system is indicated in the figures. The hydrogen and oxygen pressures are maintained at a value slightly above that of the nitrogen by means of springs in the regulators. The electrolyte reservoir (2) and pump (3) arrangement is similar to that described for the previous system.

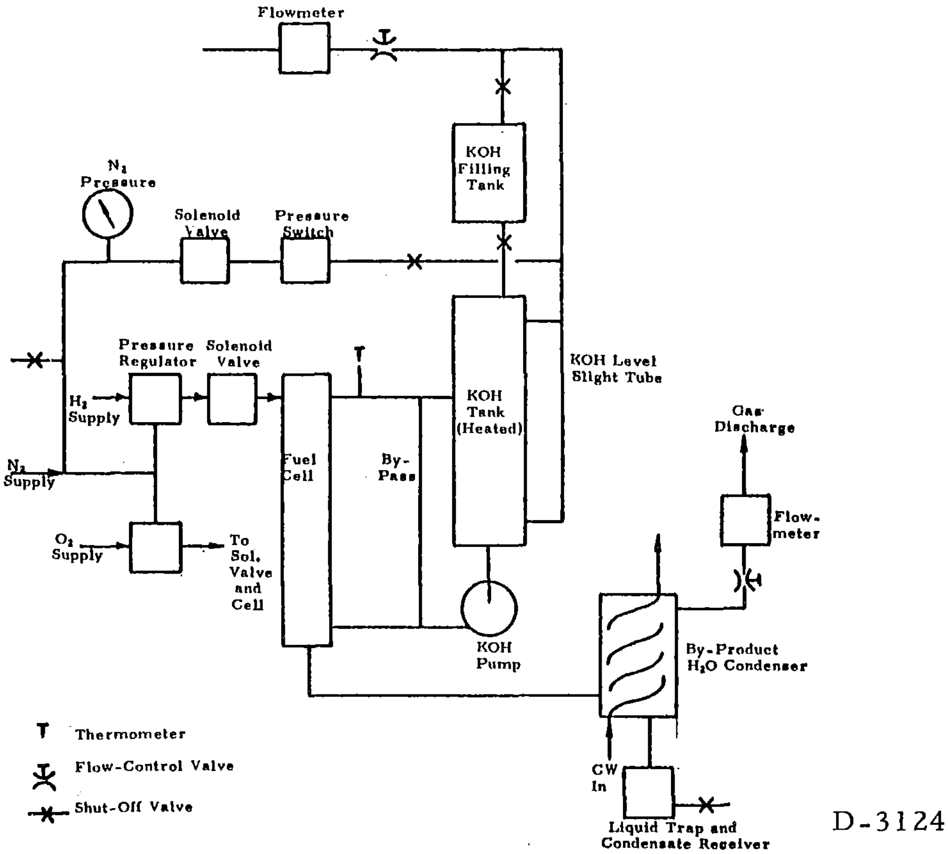
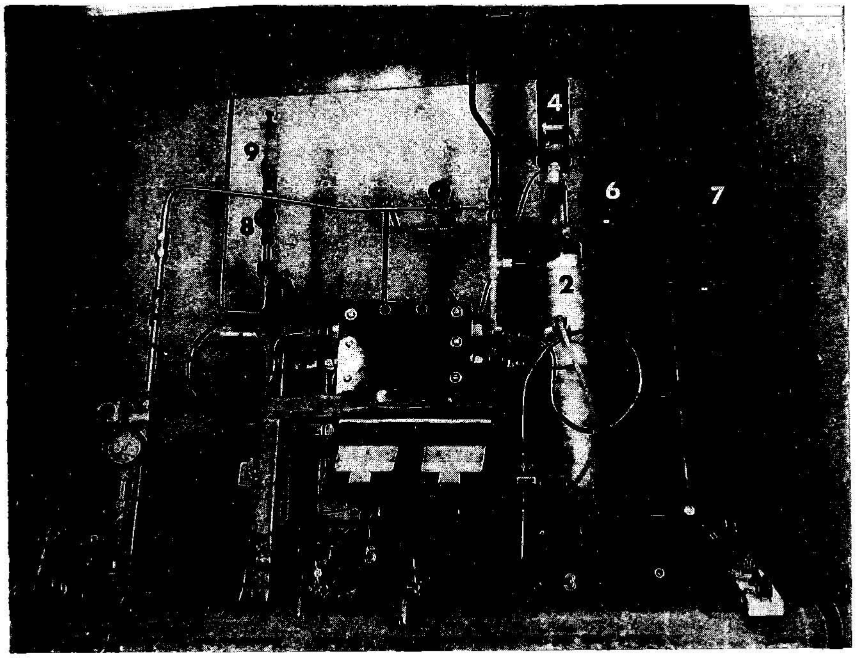


Fig. 4 - Schematic Diagram for "Small" Pressure Cells, Original Design.

In addition, a second tank (4) was installed above the main reservoir. This can be isolated from the system by means of valves above and below, and provides a means for adding electrolyte to the pressurized system, if desired. Both of these tanks are provided with service openings (normally plugged). When reference electrode readings are required, the electrode (a Zn wire) is inserted directly into the main KOH reservoir.



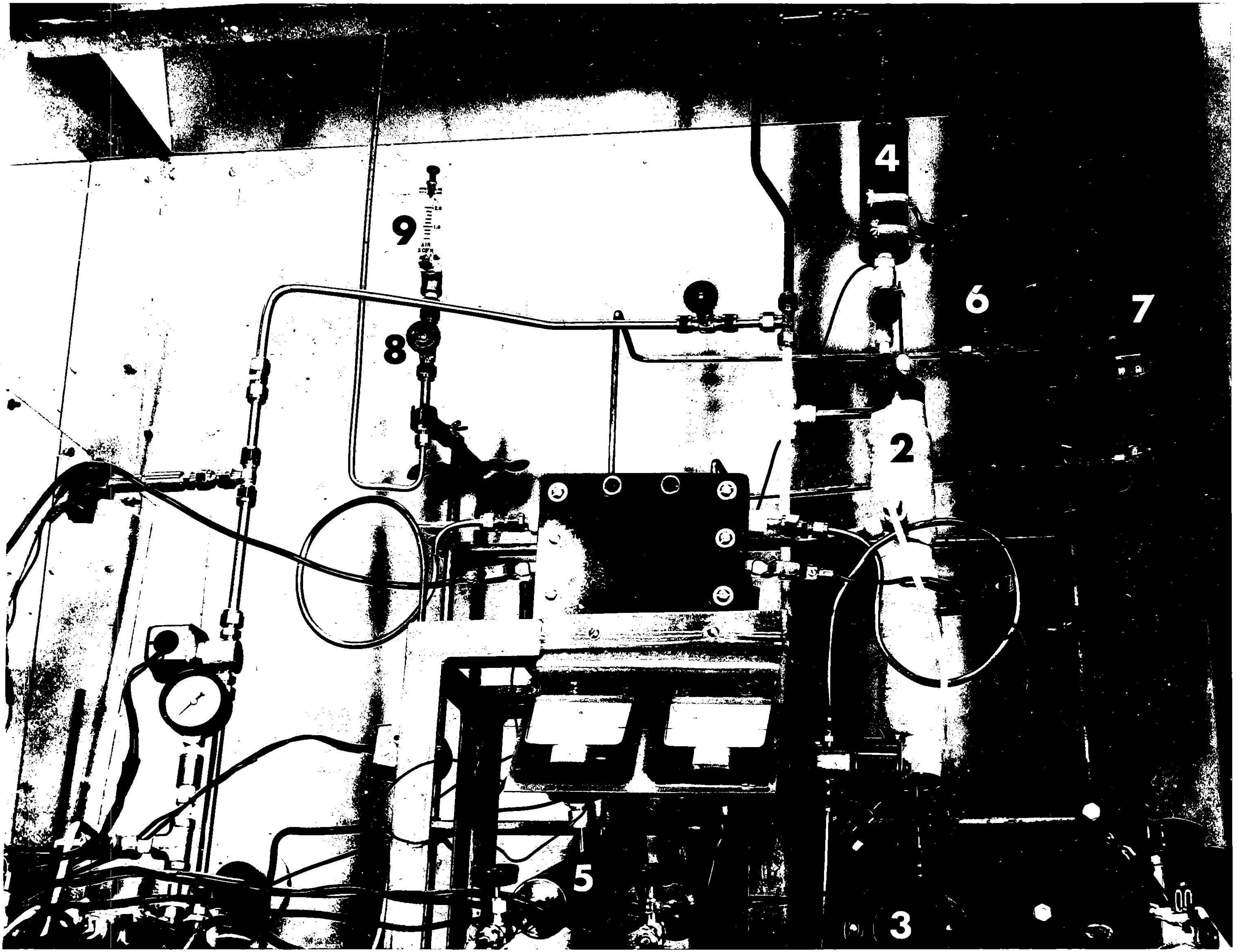
D-3118

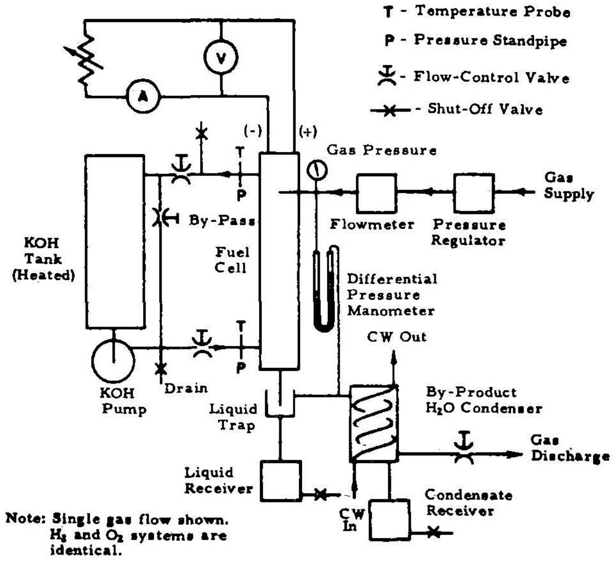
Fig. 5 - NASA Pressure Cell Test Position for "Small" Cells, Original Design.

Each exit gas passes through a condenser and trap (5)-stainless steel- in a manner similar to that already described. The flow rate is adjusted by means of a control valve (6), and measured by a flowmeter (7) at the discharge end of line. A continuous purge of nitrogen is also maintained through valve (8) and flowmeter (9) which prevents the accumulation of hydrogen and oxygen above the electrolyte.

Safety features were provided to completely shut down the system, in the event of loss of nitrogen pressure or drop in electrolyte level, by means of a pressure switch in the nitrogen line, solenoid valves in all of the gas lines, and a probe in the electrolyte reservoir.

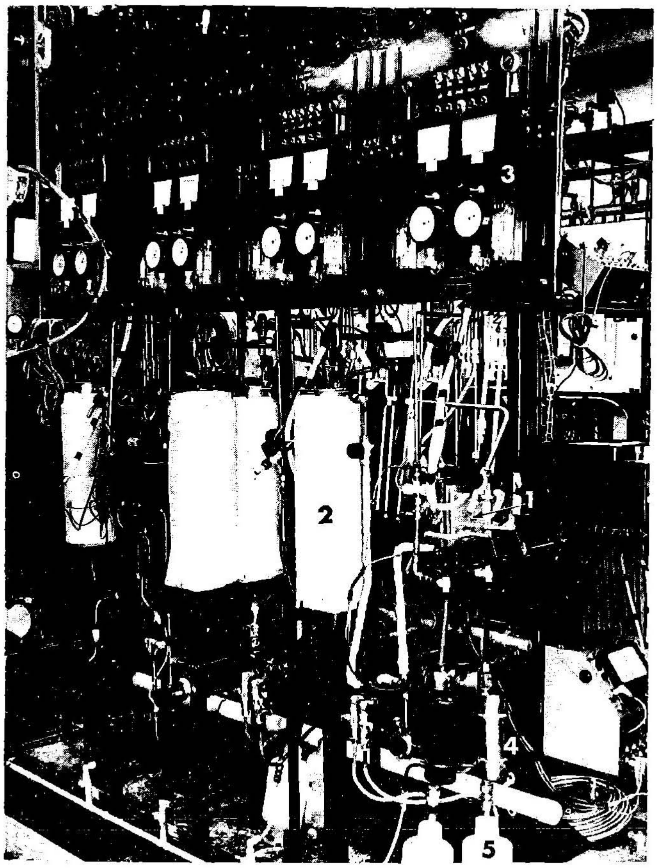
An additional six small test stands, more highly instrumented and capable of more precise control and monitoring, were subsequently constructed, These stands were designed for 15 psia operation. Figs. 6 and 7 show the schematic drawing of a position and photograph of this facility. Identifying numbers in Fig. 7 are as follows: (1) fuel cell; (2) heated electrolyte reservoir; (3) instrument panel containing pressure regulators, flowmeters, gages, voltmeter, and ammeter; (4) condenser (exit gas line), and (5) condensate receiver.


4
9
6
7
8
2
5
3



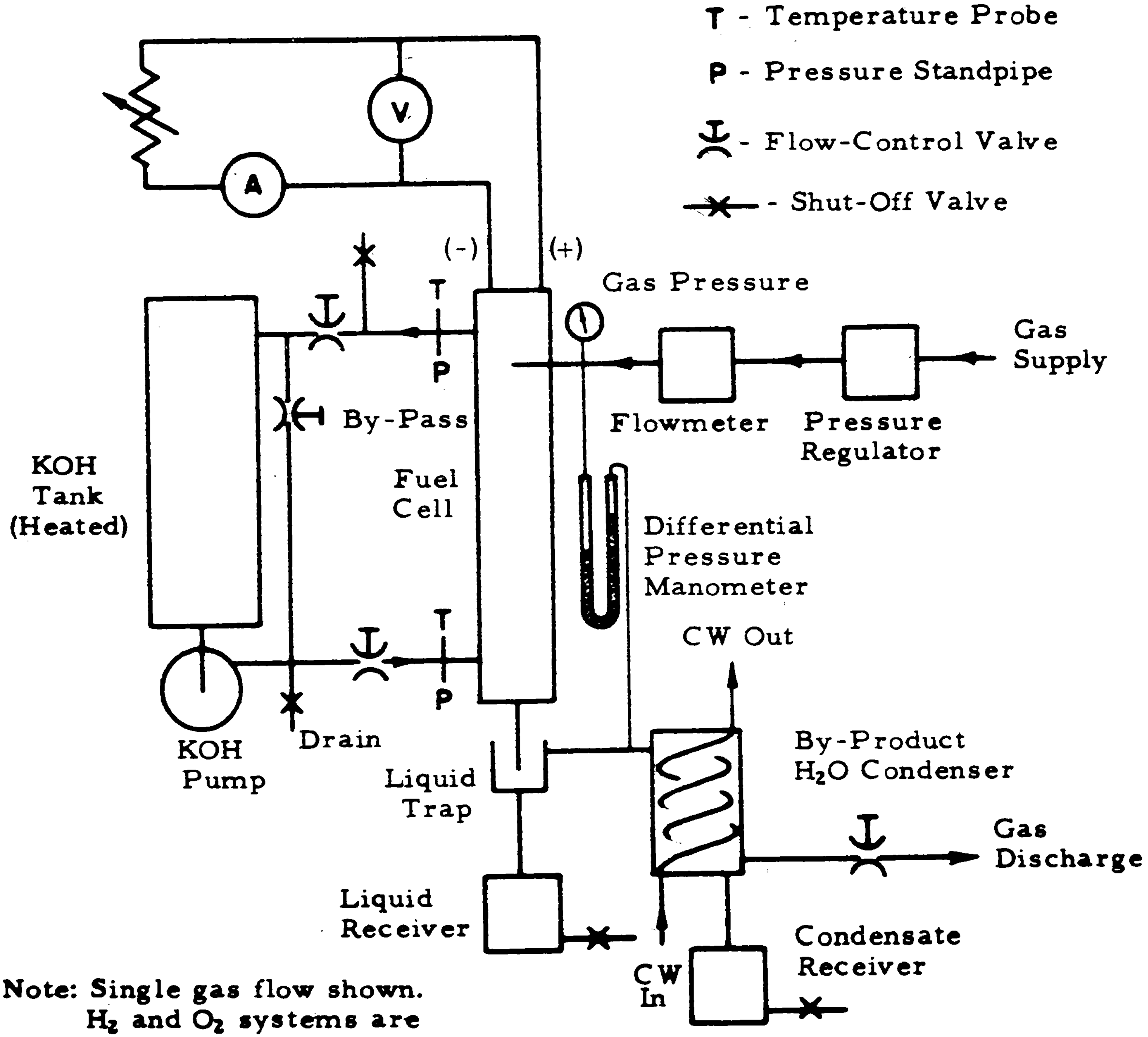
D-3123

Fig. 6 - Schematic Diagram NASA Single Cell Test, Improved Design.

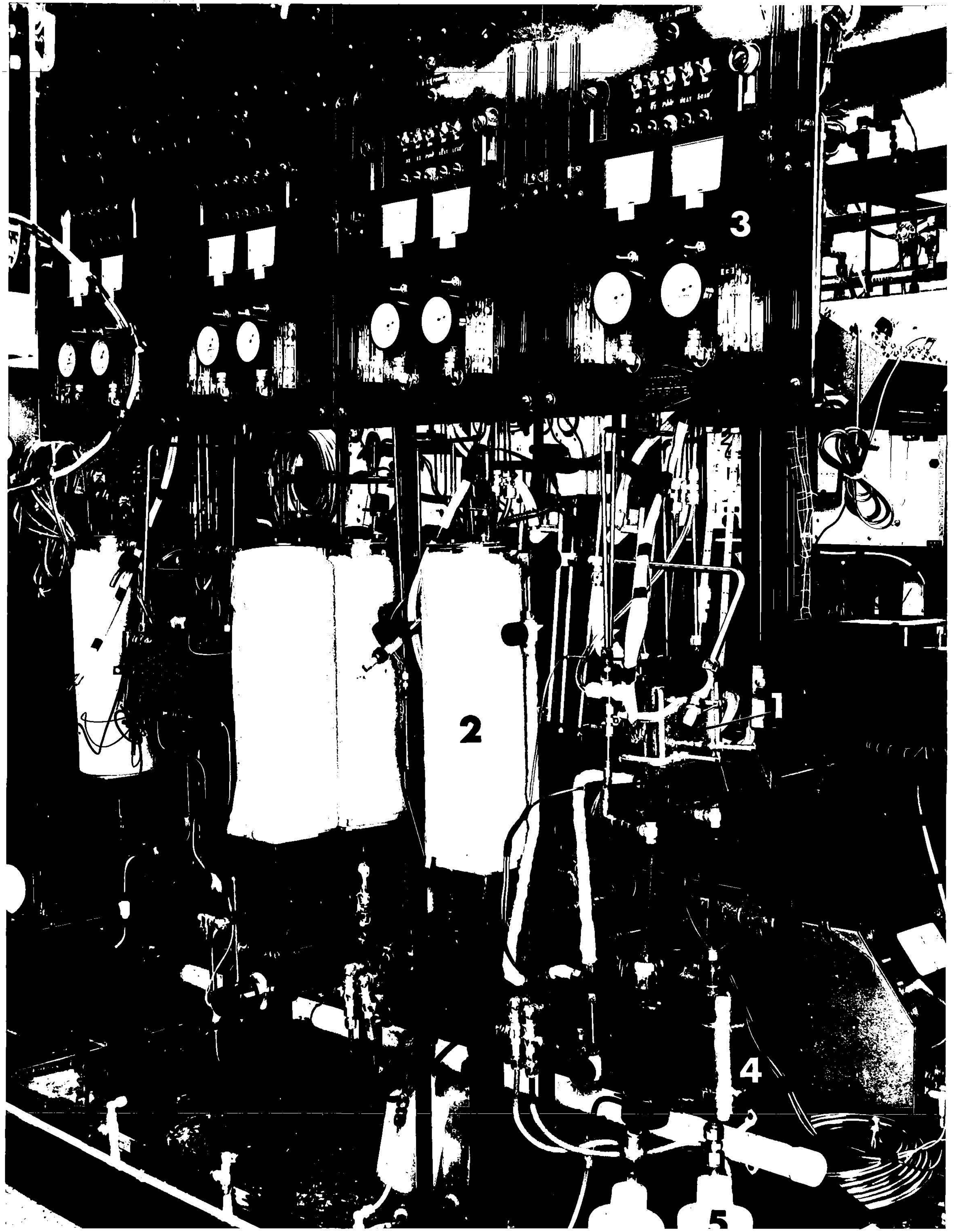


D-3120

Fig. 7 - NASA Single Cell Test Positions, Improved Design.



Note: Single gas flow shown.
H_2 and O_2 systems are
identical.


3
2
1
4
5

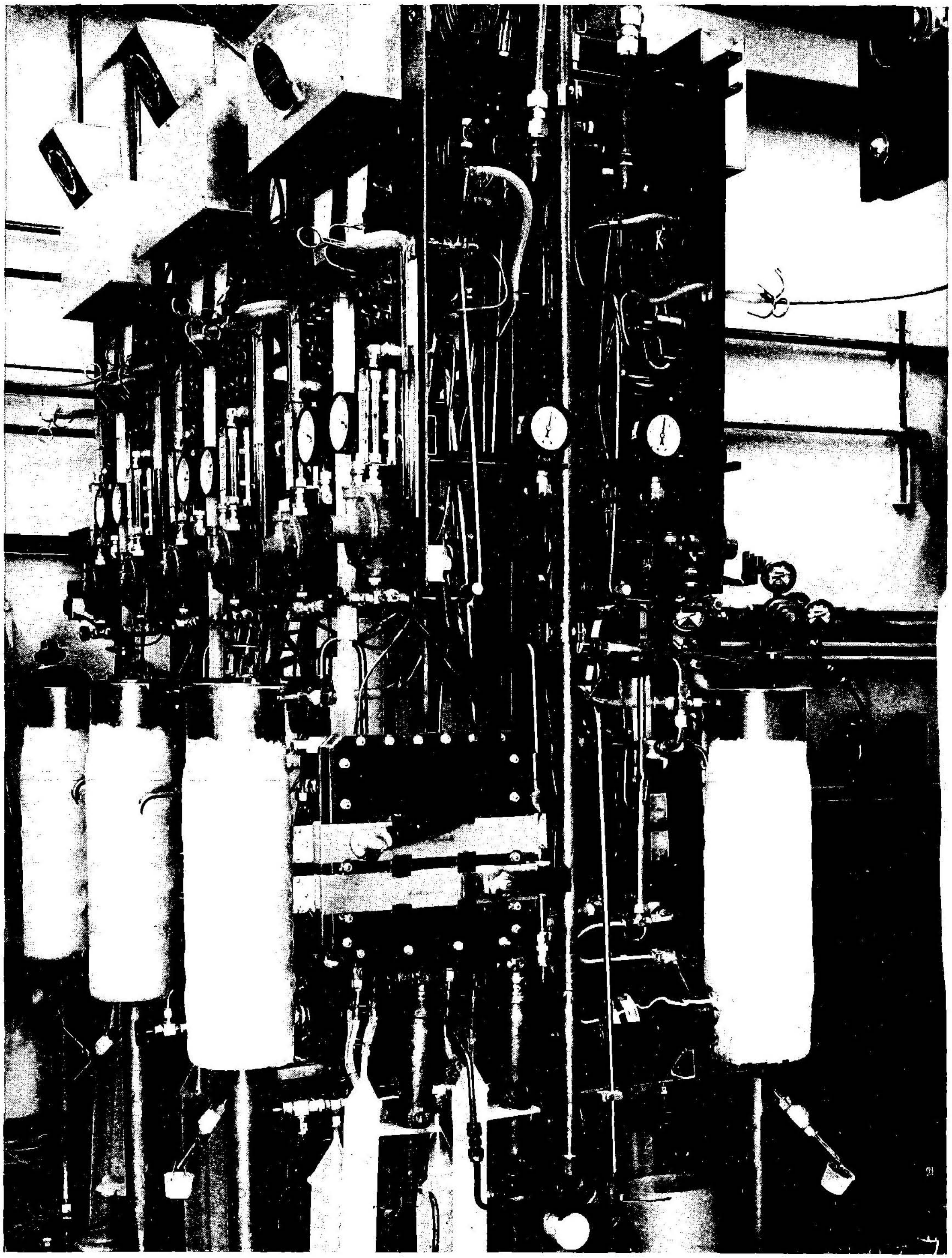
For the Task I program, six large test stands for the flight-size cells were also built. Of these, four were for operation at 15 psia (normal atmospheric pressure) and two for operation at 30 psia or higher. The 15 psia stands are substantially similar to the small stands except for such obvious modifications as larger electric loads and leads, larger fluid ducts and meters, and larger water collection devices. The large 15 psia stands are shown in Fig. 8. The 30 psia stands are depicted in Figs. 9 and 10. The large test stands could also be used for small cells as the situation required, in which case the electrical systems were altered.

During Task III, four of the original five small, atmospheric pressure stands were decommissioned and replaced with new pressure stands capable of accepting either flight-size or small cells. In addition, the first pressure stand was extensively upgraded in terms of instrumentation and control equipment.

3.2.2 Test Procedures

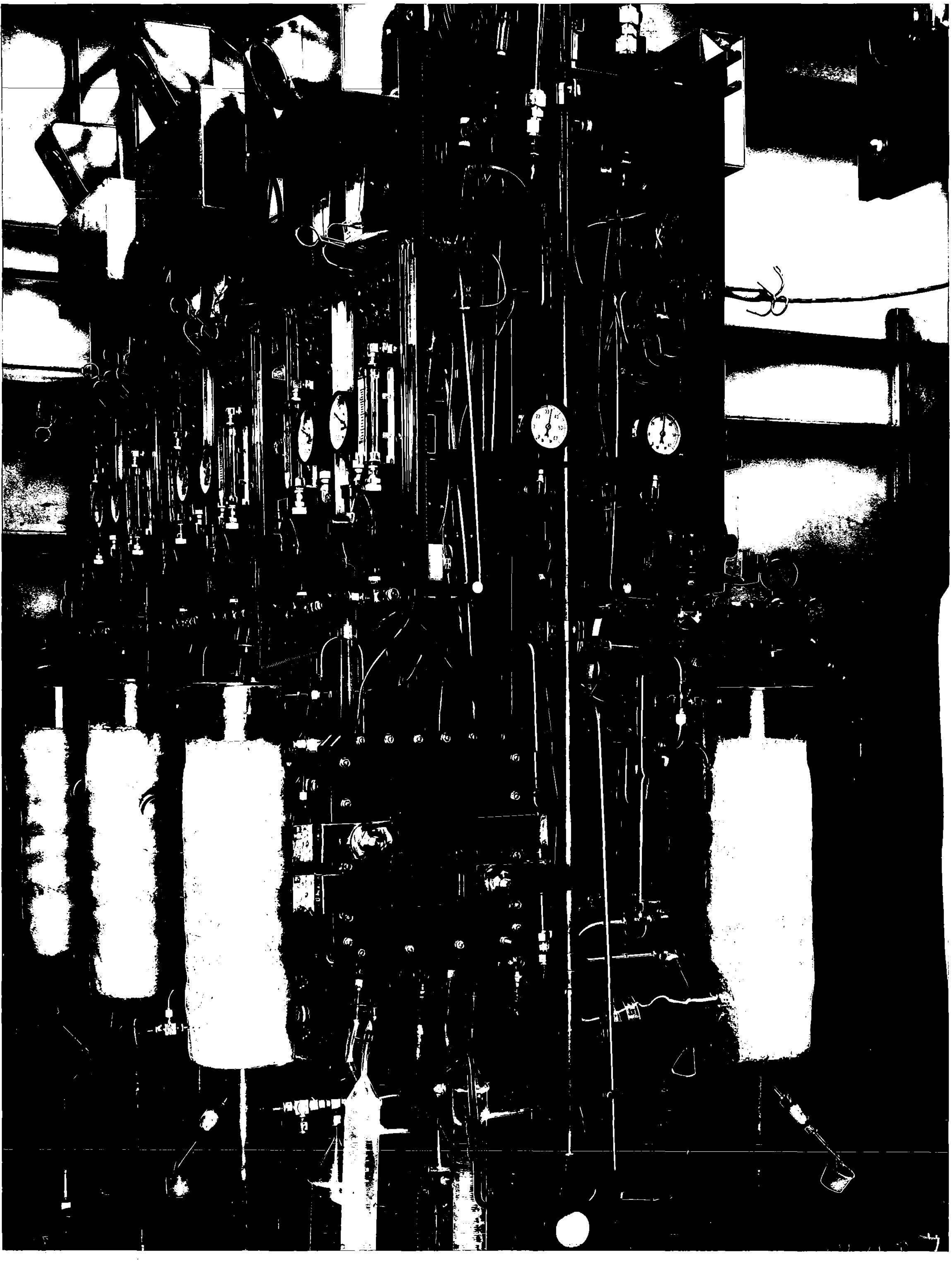
Most of the tests described in this report were life tests at 15 psia conducted on small cells. For these, the following procedures were generally used; The electrolyte was 14 N KOH, heated in the electrolyte tank to give a cell outlet temperature of 90 ± 2°C. The nominal electrolyte circulation rate through the cell was 124 ±10 ml/min. Sometimes this flow rate was exceeded because the nominal 124-ml flow could only be achieved by very nearly closing the flow control valve—an unsafe condition for cell operation, since the nearly closed valve was prone to possible blockage caused by small dirt particles. In practice, the KOH inlet pressure was generally 16.5" of KOH; the pressure drop through the cell was 3 - 4". A determination of the electrolyte flow as a function of pressure drop was part of the normal start-up procedure and was repeated periodically. Usually no significant flow degradation was found. The pressure drop at a flow rate of 125 ml/min of hot 14 N KOH (per electrode pair) is included in most of the performance studies so that the KOH flow characteristics of the various cells may be compared.

Hydrogen and oxygen were not recirculated, but instead were passed through the stacks once and then exhausted. The flow rates were adjusted to give water balance; i. e., to maintain the electrolyte concentration at the desired point as far as possible. This proved difficult, and in most instances the electrolyte tended to concentrate, so that water was added from time to time. This explains the recorded fluctuations in electrolyte normality. The gas flows were measured on Brooks air purge meters. For the small stacks these were generally kept at



D-3437

Fig. 8 - Test Stands for Flight-Size Cells Operated at 15 psia.



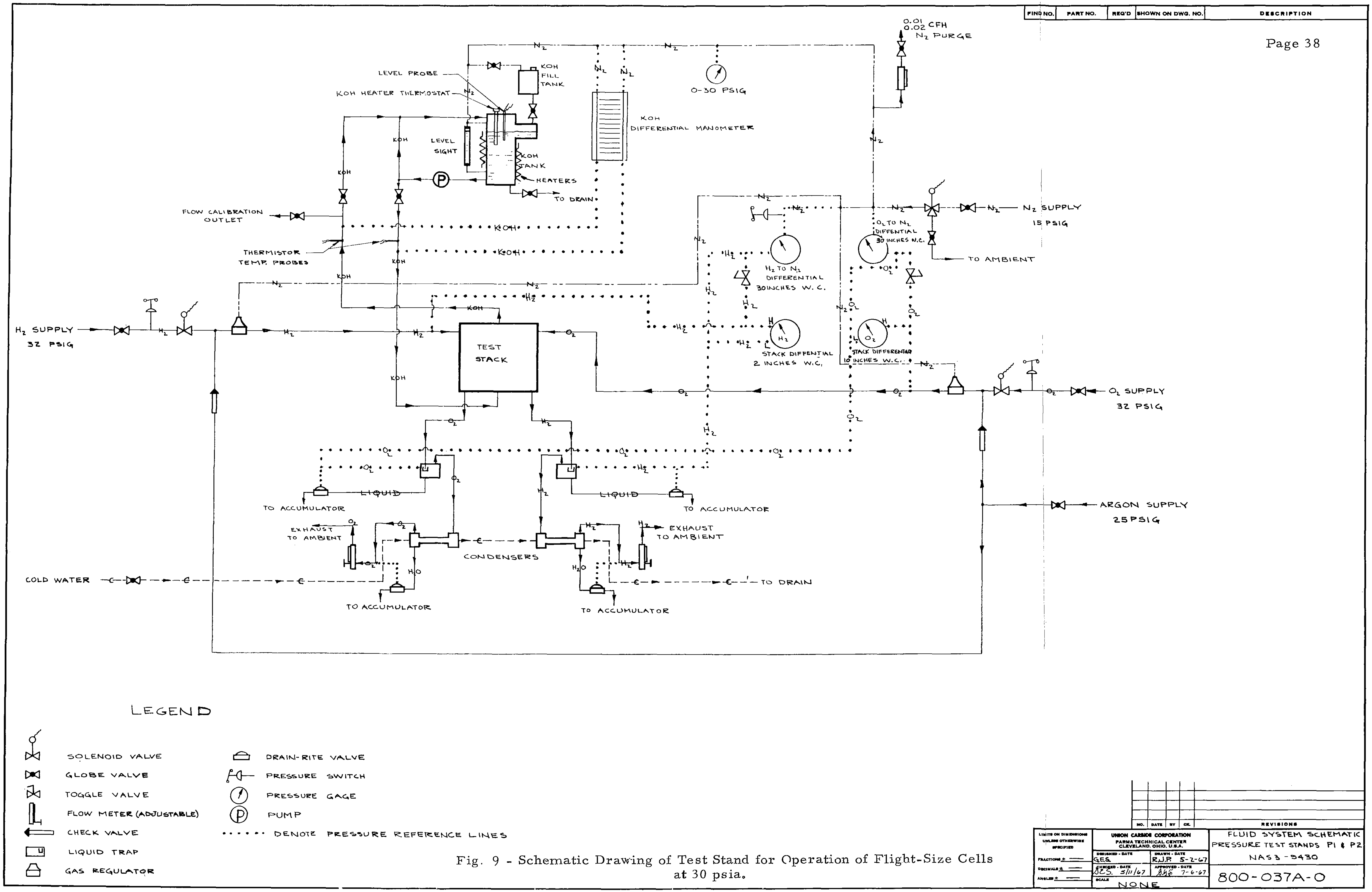
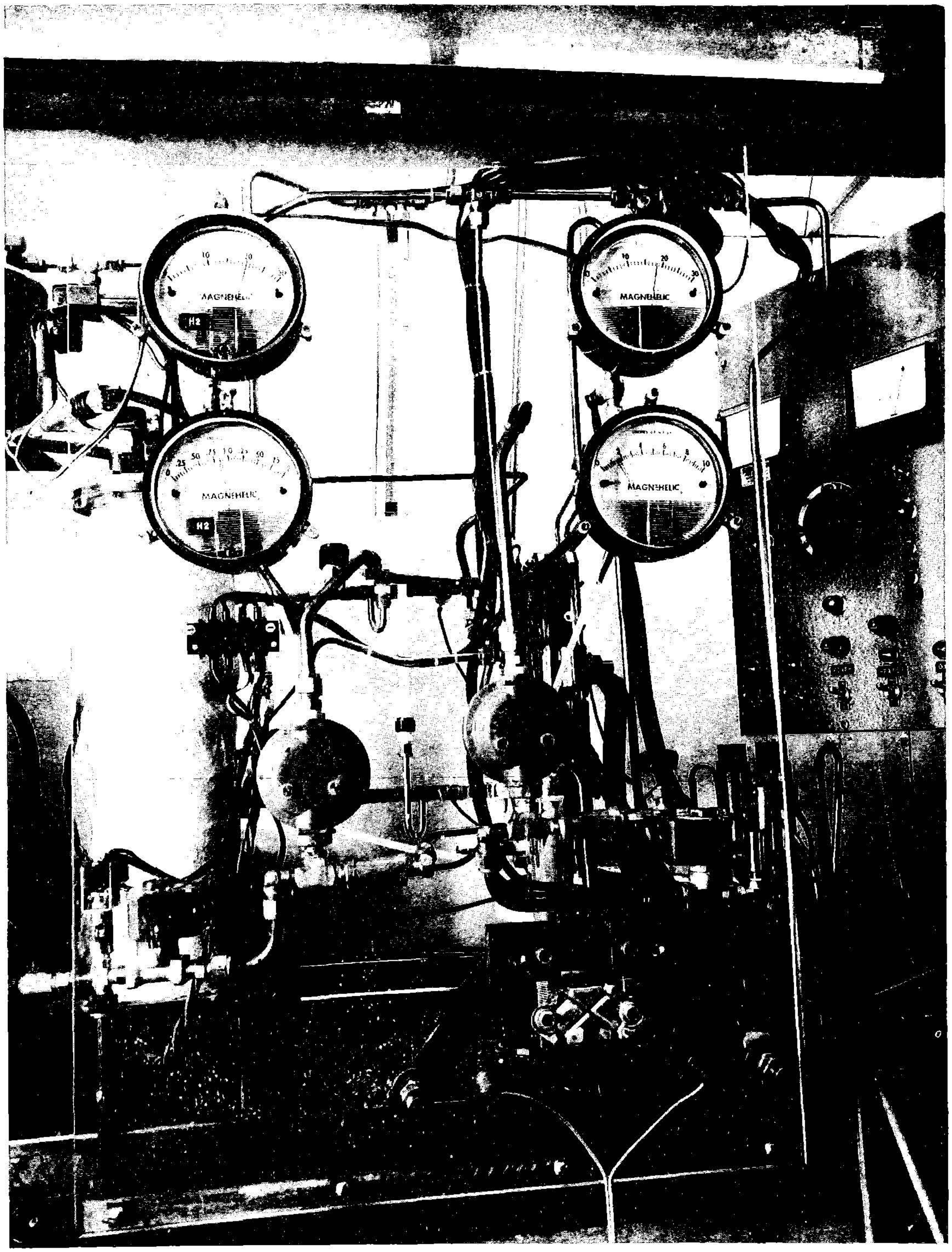
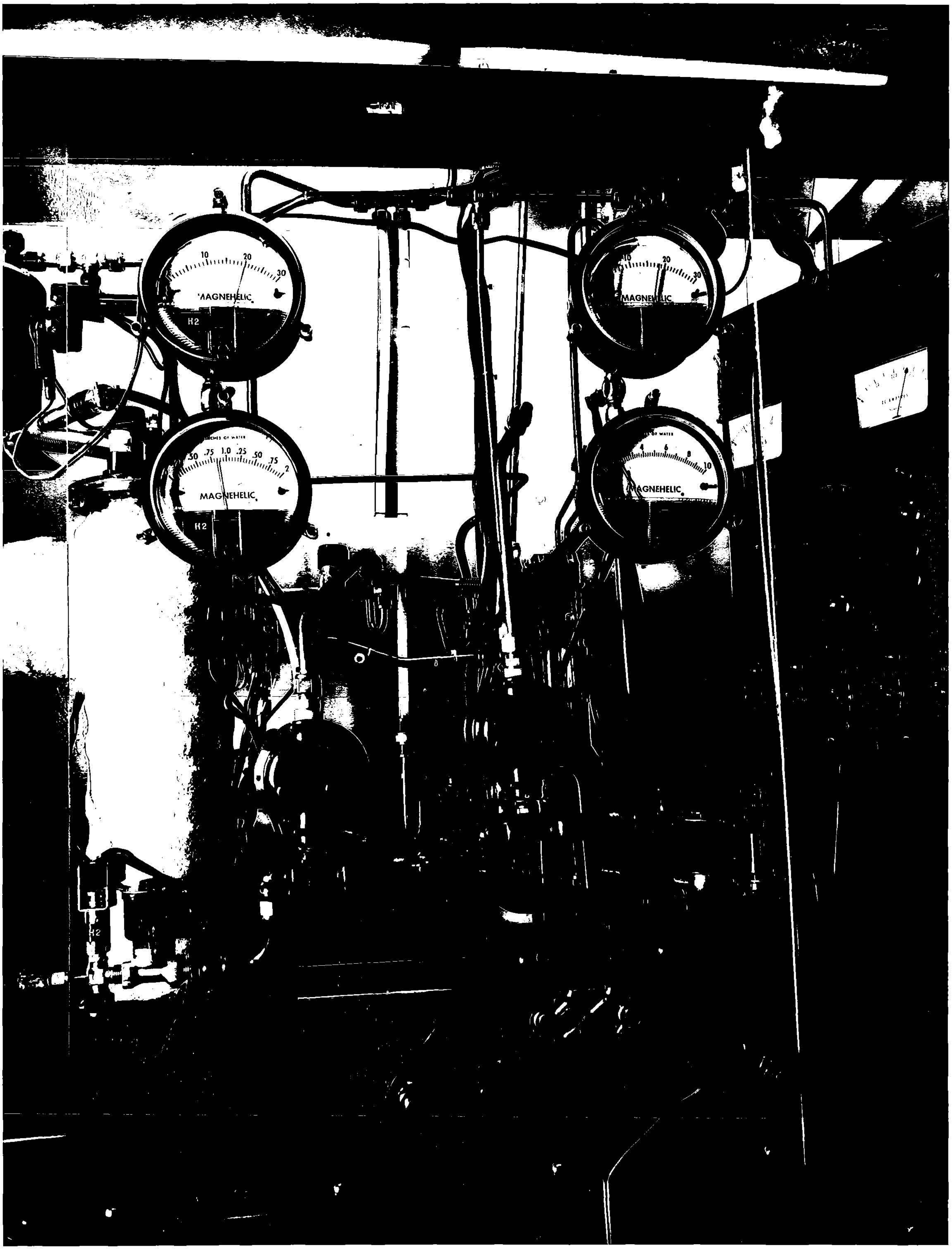


Fig. 9 - Schematic Drawing of Test Stand for Operation of Flight-Size Cells at 30 psia.



D-3446

Fig. 10 - Test Stand for Flight-Size Cell Operation at 30 psia.


10 20 30
MAGNEHELIC
H2
INCHES OF WATER
.50 .75 1.0 .25 .50 .75 2
MAGNEHELIC
H2
10 20 30
MAGNEHELIC
4 6 8 10
MAGNEHELIC
H2

what is equivalent to 0.4 scfh of air for H_2 and 2.0 scfh air for O_2, corresponding approximately to 1.56 scfh H_2 and 1.82 scfh O_2. At 200 ASF a 1/8 ft^2 electrode actually requires 0.40 scfh H_2 and 0.20 scfh O_2, so that for most tests the systems were run at what would correspond to a recirculation rate of 4X use rate on the hydrogen side, and 9X use rate on the oxygen side. These flow rates generally require about 0.3 - 1.0" water equivalent ΔP for O_2 and 0.1 - 0.2" for H_2. Gas inlet pressures were set regularly to be 1" below the blow-through pressure for O_2, and 2" below blow-through for H_2. In addition, the difference between the pressures of the two gases was not permitted to exceed 10". Actual inlet pressures were usually in the 15 - 30" range.

The electrolyte temperatures of 90 ± 2°C at the outlet were measured with a thermistor system from Yellow Springs Instrument Co., Yellow Springs, Ohio. Most test stands were equipped with probes both on the inlet and outlet, and in general the temperature difference between the two was about 2°C, the inlet being higher. The heat lost from the stack was greater than that produced by the electrochemical reaction even after the stacks were insulated with either 3/8" Plexiglas plates over the cell end plates or with Styrofoam. Temperature was maintained by heating the electrolyte tank externally with a heating tape controlled by a Ni-plated Fenwall thermoregulator immersed in the electrolyte tank. Electrolyte pressures were measured in standpipes at the inlets and the outlets.

Current, voltage, temperature, gas flows, and electrolyte concentration were checked daily. Condensed water was removed and measured daily; leakage, if any, was measured as needed. The alkalinity of all liquids collected was checked and recorded if any was found. In general, polarization curves were run on all cells once a week using a Marko-Kordesch interrupter, unless special situations required more frequent data taking.

The Marko-Kordesch Interrupter [J. Electrochem. Soc. <u>107</u>, 480-483 (1960)] is an instrument which permits measuring the voltage of a battery or fuel cell stack eliminating the ohmic voltage drop. This is called the IR-free reading, denoted by the letter "F" in all our polarization curves. The polarization curve obtained as usual by taking the terminal voltage was also measured with the same instrument and is shown on the same graphs, unmarked, called the IR-included curve. Thus, the two curves may be compared directly.

Cells operated at 30 psia were first started in the standard fashion at 15 psia, 90°C, and 200 ASF. Particular attention was paid to blow-through pressures and electrolyte flows which could not be checked after pressurization. Then, with the stack on open circuit, the solenoid controlling the flow of nitrogen-- the pressurizing gas-- was opened. Nitrogen pressure was applied uniformly to O_2 and H_2 regulators and to the entire electrolyte system, so that all portions of the fuel cell system experienced the rise in pressure simultaneously. The H_2 and O_2 purges were watched carefully and adjusted to maintain the correct purge rate.

As far as data taking was concerned, the same procedures were followed and the same data taken as for 15 psia operation except: a) electrolyte flow could not be checked; b) N_2 pressure and purge were checked daily, and c) the KOH normality was checked daily by titration of a small sample, instead of the specific gravity method usually used.

The procedures used for the flight-size cells were basically the same as those outlined above for the small cells with such obvious modification as having the gas flows, etc. about ten times larger.

Task II of the contract called for the design of a 5 kW system capable of performing under a certain load profile. Several tests were run to establish performance of cells under a load profile similar to that specified for the system. These tests, which will be discussed subsequently, differed from all other tests in that the electrolyte was only 11.5 N KOH. All liquids removed from the stack were collected in just two bottles, one in the anode-exit side, and one in the cathode exit. The normality of the liquid was determined and the equivalent electrolyte leakage, if any, calculated therefrom. Gas inlet pressures were checked daily. The pressures were set at 1" below blow-through pressure at a fairly low current (ca. 60 ASF), and were not changed, even though the pressure dropped (for the same gas flow rate) at the higher current densities. These tests were carried out on the original design, less highly instrumented, group of test stands. The actual load cycle used in the tests differs in detail from the load cycle specified for the 5 kW system. It has the same percentage of the operating time under the same loads as the system specification requires, but the exact timing was rearranged for convenience of single-shift operation.

3.2.3 Cell Design and Construction

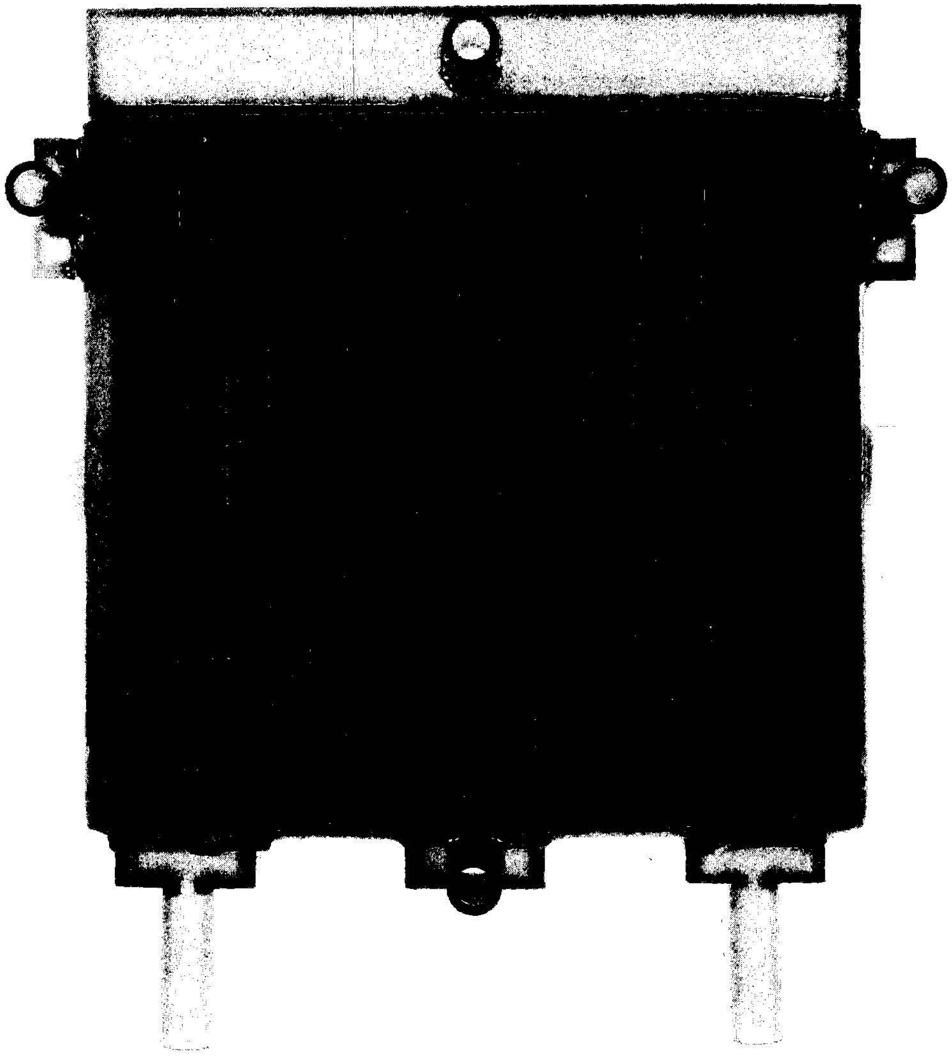
All of the cells tested in this program used the same basic cell design and construction procedure. This involved sandwiching an anode and a cathode between two end plates with appropriate gas and electrolyte separators and sealing the unit with epoxy. Cells are generally characterized as hybrid, meaning that a T-2 anode was coupled with a LAB-40 cathode, or as LAB-40 meaning that this electrode was used both as anode and cathode. Various plastics in various geometries were used for the fluid separators. As the program progressed, details of construction were continually evolving to correct for various problems uncovered during testing. During Task I only minor construction modifications were made; but during Task III, major redesign was undertaken.

A typical small cell used for atmospheric pressure testing during Task I is depicted in Figs. 11 and 12. The active area of each electrode in this design is nominally 1/8 ft^2, with the electrodes separated from each other by the electrolyte separator specified in Table XVIII. Initially, an expanded-nylon separator (S-2) was used, but autopsy of several long-lived cells after failure showed that the nylon separator had disintegrated. This permitted the electrodes to touch and short out.

Similarly, autopsies showed that the woven Lamport's polypropylene separator (S-1) was not useful when exposed to O_2 at 90°C for long periods, and consequently both the electrolyte and cathode separators were changed to expanded-mesh Teflon materials. The S-1 polypropylene separator was satisfactory in the H_2 atmosphere of the anode compartment.

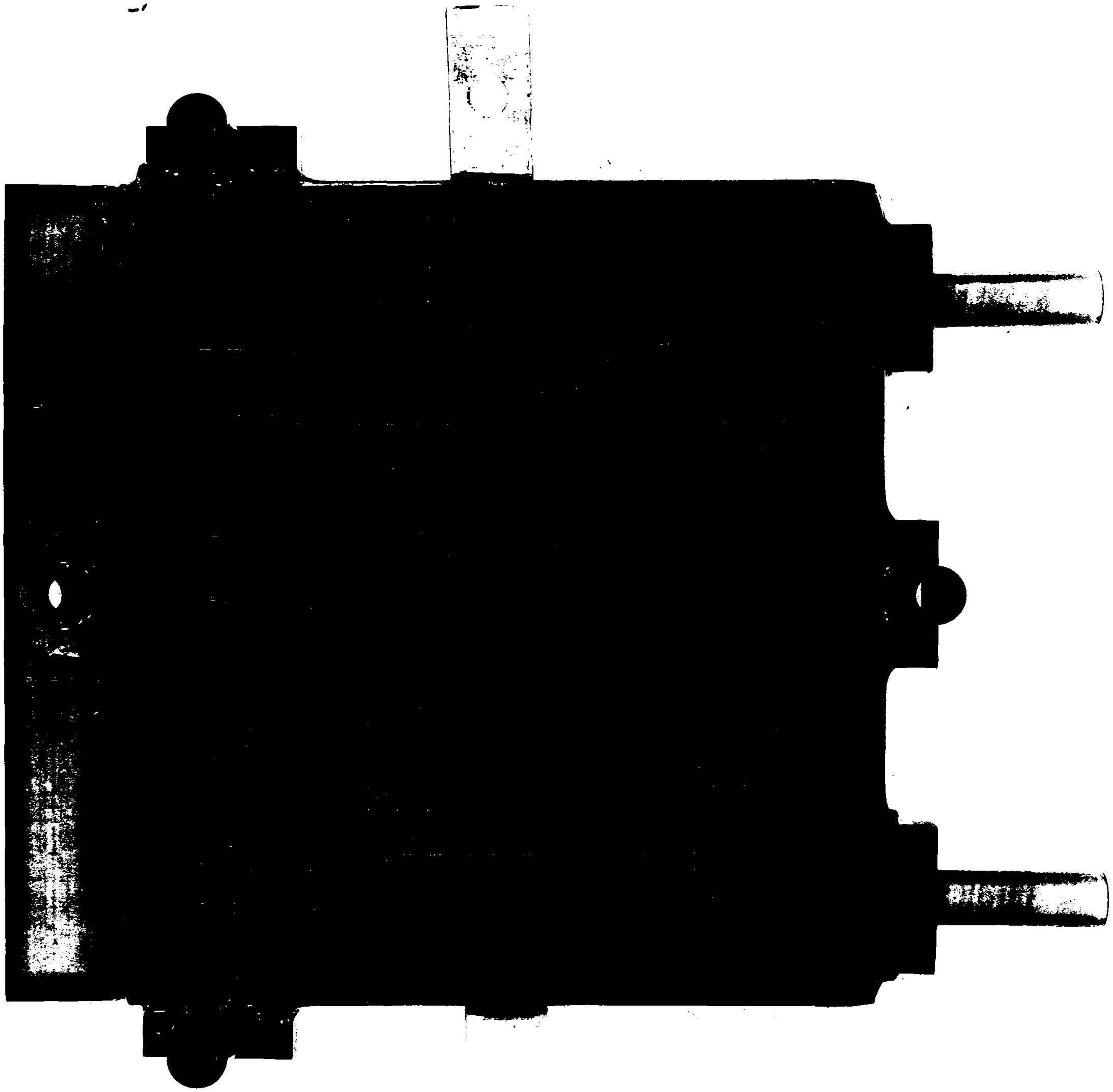
The electric current produced was collected by framing each of the electrodes in folded 0.005" silver foil, 3/16" wide after folding. The silver was sealed to the LAB-40 electrodes on the gas side with clear epoxy, and on the electrolyte side with silver epoxy. For the T-2 electrodes, this arrangement was just reversed, so that the silver epoxy was on the nickel side of the anode. Furthermore, along one edge of each silver frame (usually on the gas side) a 0.025" thick silver strip (3/16" wide) with an integrally welded tab was attached to bring the current out of the cell. Tabs from anodes and cathodes were on opposite sides of the cell.

The housing as a whole was made of polysulfone and epoxy. Polysulfone



D-3393

Fig. 11 - Small Cell for Operation at 15 psia, (Task 1 Design) 1/8 Ft^2 Active Electrode Area.



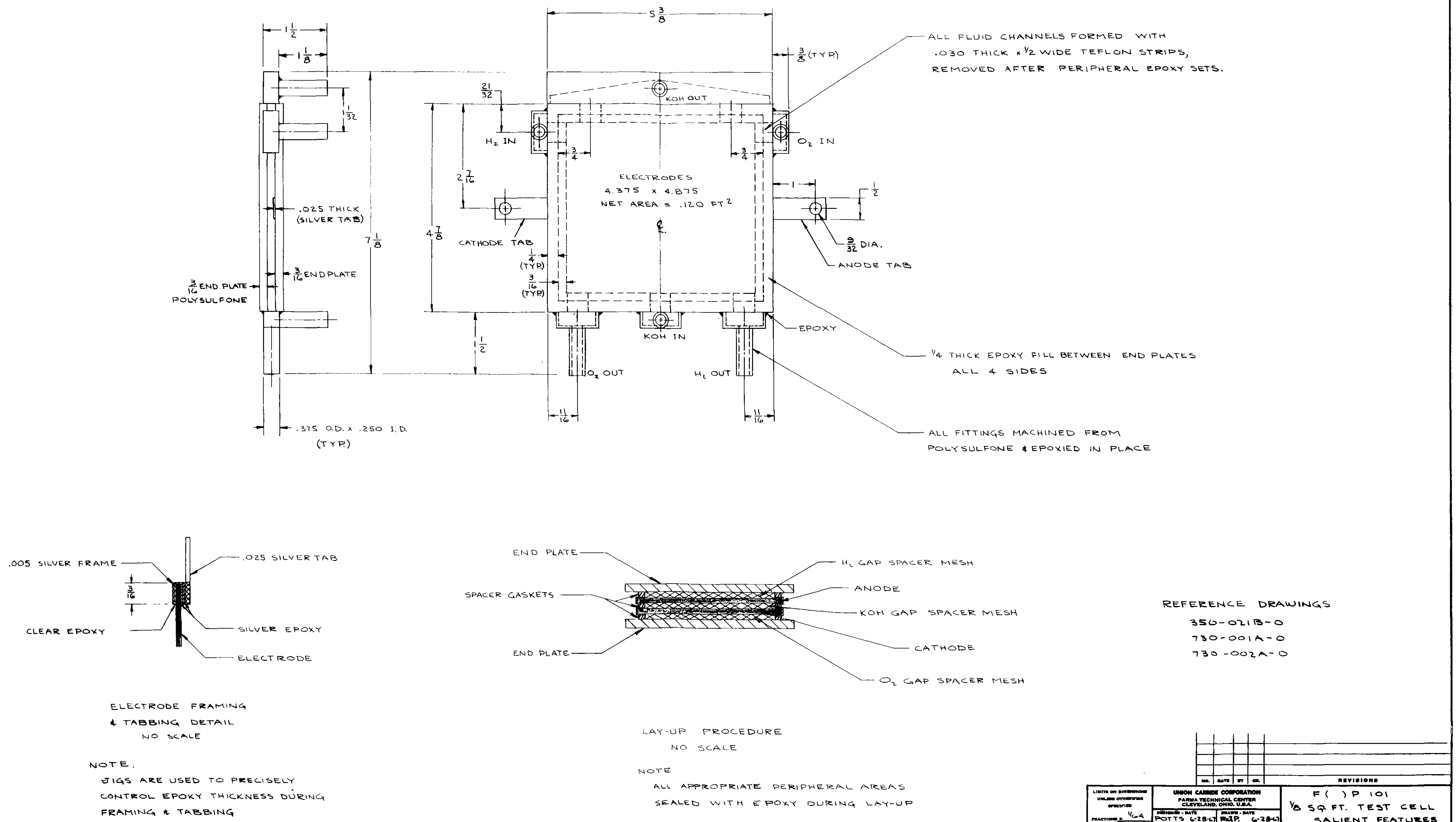


Fig. 12 - Schematic Drawing of Small Stack.

TABLE XVIII

Identification of Separators

S-1	Lamport Style 7700 polypropylene, nominal thickness 0.093."
S-2	Exmet code 10 expanded nylon 25-1 (stablized).
S-3	Webril SM 91-oriented fiber-nominal 6 mil polypropylene.
S-4	Exmet expanded polypropylene 25-1.
S-5	Polypropylene - DuPont 30 PDS 89 - stretched.
S-6	Exmet expanded Teflon 25-1, flattened. A single piece is flattened to 0.015"; 3 pieces are used and arranged so that the axis of the center piece is at right angles to the axes of the end pieces. Number preceding S-6 designation indicates number of individual 0.015" thick pieces used.
S-7	Exmet expanded Teflon 25-1, heat treated but not flattened, 0.045" nominal thickness.
S-8	Polysulfone serpentine stick - full length vertical.
S-9	Polysulfone serpentine stick - single ended vertical.
S-10	Polysulfone serpentine stick - full length horizontal.
S-11	Expanded Polysulfone mesh.

end plates, with the electrodes and separating gaskets held between them, were joined by an epoxy bridge. The whole unit was sealed with Union Carbide Type-2774 epoxy with 2793 hardener in the ratio of 3:1, and oven cured at 50°C. The H_2 and O_2 inlets were at opposite top corners of the same face, the gas outlets at the diagonally opposite bottom corners. There were no orifices. Fluid slits 0.030" thick x 0.05" wide were provided as the electrolyte and oxygen inlets and outlets in the epoxy edge fill. Hydrogen slits were 0.040" thick to accommodate the thicker anode separator. The KOH inlet was at the bottom center with one inlet slit into the electrolyte space. At the top, two slits (one near each corner) opened into a plenum chamber which had one oulet in the center, with the top of this plenum being "gabled" to encourage egress of any trapped gas. All the inlet and outlet tubes were polysulfone of 3/8" O.D., with 0.032" wall thickness.

The frame gaskets spacing the electrodes from each other and from the end plates were generally 3/16" wide, and of such thickness as to accommodate the separator properly. The gaskets were made from a special Neoprene rubber, treated with H_2SO_4, or from polysulfone. Both types were glued to the mating faces with epoxy in the cell subassembly. These gaskets and Teflon plugs (later pulled out to form the fluid passages) provided a seal during the successive pouring of the 1/4" thick epoxy along the four edges of each cell.

Shortly after initiation of the Task III program, several long duration cells (i. e., $>$2000 hours on test) remaining from Task I were removed from test and destructively examined. From these tests it became apparent that the existing cell construction would not reliably operate for the 3000-hr lifetime goal of Task III. In particular, the silver epoxy joints which bonded the silver current collector strips to the electrodes had become severely debilitated during extended exposure to 90-100°C KOH. It was believed that the breakdown of this electrically conductive epoxy joint was responsible, at least in part, for the weepage that had been observed in many of the test cells. Consequently, several major changes in cell construction were instituted. For example, the silver epoxy joint was replaced by a welded joint with the current collector strip being resistance seam-welded directly to the electrode. In a few cells (i. e., A-101, A-102, A-105 and A-212) nickel was used as the current collector material. However, the remaining cells constructed during Task III utilized silver as the electrode bus. It was also decided to replace the epoxy adhesive used previously (i. e., Union Carbide 2774 resin with 2793 hardener) with alternate formulations -- the objective being to obtain a joint which could accommodate the thermal stresses resulting from differential expansion between the electrodes and the plastic structural members. The 2774/2793 combination was felt to have inadequate elasticity over the operating range to result in reliable edge seals. Consequently, a series of screening tests were instituted on several commercially-available resins. These screening tests included the following:

1. Stress and Strain: Stress and strain values were determined by measuring the proportional limit in a compressive load versus deflection test.

2. Adhesion: To evaluate adhesion, the various epoxies were used to bond strips of electrode to polysulfone sheet. Four samples were prepared with each epoxy, using the electrode structures below:

a. LAB-40 gas side;
b. LAB-40 KOH side;
c. Union Carbide T-2 gas side;
d. Union Carbide T-2 KOH side.

After soaking samples in a 100°C oven for 4 hours, they were each subjected to a peel test. A bond failure within the electrode structure as opposed to bond failure between the electrode and polysulfone was used as the acceptance criterion.

3. KOH Compatibility Test: Samples of each of the epoxies were soaked in 12 N KOH at 100°C for a period of one week. Except for slight discoloration and changes in weight, none of the samples appeared to have suffered any adverse effect from the KOH treatment.

4. Thermal Cycling: To evaluate the ability of the epoxy to tolerate thermal cycling, structures representative of those found in a typical cell were constructed. After several thermal cycles in air at 100°C, the structures were subjected to a peel test and checked to see whether they were still leak-tight to KOH.

5. Viscosity: The potting technique used to assemble cells requires an epoxy of low viscosity so that it will flow into all interstices. Viscosity was judged by visual observation of the flow properties of the various epoxies.

Results are summarized in Table XIX. On the basis of this evaluation, the D. Ring Chemical Company No. 101 Resin with the "F" Hardener (2:1) was selected for further evaluation in test cells. This adhesive was used in Cells A-109, A-110, A-111, A-112, A-126, A-127, A-214, and A-215. At this juncture in the cell construction work, the seal area (i. e., the amount of electrode encased in epoxy) on the electrodes was increased about 1/4 inch on each edge of the electrode. However, the selection of the Ring 101-F combination coupled with increased electrode seal area did not solve the cell weepage problem; and, consequently, further alternatives for the cell adhesive were considered. One of the problems with the Ring epoxy was that it contained substantial quantities of trapped air bubbles which were difficult to remove and could possibly contribute to imperfect edge seals.

One cell (A-107) was constructed with a proprietary adhesive prepared by Hysol, three cells (A-118, A-123 and A-125) used an adhesive* formulated

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*70 parts Union Carbide phenoxy resin PKHH and 30 parts toluene diisocyanate.

TABLE XIX

EVALUATION OF CANDIDATE EPOXIES FOR NASA CELL CONSTRUCTION

Sample No.	Manufacturer	Base	Hardener	Ratio Base/Hardener	Stress (psi)	Strain (in/in.)	Adhesion	KOH Compatibility	Thermal Cycling	Viscosity
1	Union Carbide Corporation	2774	2793	3:1	11,400	0.030	A	A		
2	Union Carbide Corporation	2774	2793	2:1	8,850	0.033	A		$2^{(a)}$	$2^{(b)}$
3	D. Ring Chemical Company	101	E	2:1	6,600	0.033	U	A		
4	D. Ring Chemical Company	101	F	2:1	7,700	0.035	A	A	1	1
5	D. Ring Chemical Company	100	E	2:1	9,300	0.034	A	A	3	3
6	D. Ring Chemical Company	100	F	2:1	7,900	0.034	A	A	4	4
7	D. Ring Chemical Company	100	A	2:1	8,000	0.034	U			
8	Kunststoff Chemical Co.	Rezolin A	916AD	12.5:2.4	15,100 11,200	0.042 0.038	U U	A A		
9	Kunststoff Chemical Co.	Rezolin L930	L930H	10:6						
10	Shell Chemical Co.	815		92:8						
	Armstrong Products Co.		A		23,400	0.0056	A	A	A	2
11	Union Carbide Corporation									
	50%	2774	0822	10:3	21,500					
	50%	2774	2793	4:1		0.0135	A	A	A	1
12	"	2774	ZZLB-0325	7:3	5,880	0.0492	A	A	A	3
13	"	2774	ZZLB-0340	7:3	6,890	0.0339	A	A	A	5
14	Allaco Products	All Bond	All Bond	1:1	16,180	0.0047	A	A	A	4

(a) Ranked in order of preference

(b) lowest viscosity

A = Acceptable

U = Unacceptable

by Bjorksten Research Laboratories, Inc. and one special cell was assembled from two electrodes which first had an elastomer molded around the perimeter by Gulton Industries, and these were cemented together with a proprietary cement.

Excellent adhesion of the various cell components was obtained with the Bjorksten adhesive. Seven additional cells were potted with a Rezolin epoxy which had been previously used by Union Carbide Corporation on a company-supported program. The particular epoxy was Rezolin "A" resin with XC2-11-2 hardener (4:1). With each different adhesive, details of the cell fabrication procedure were varied to accommodate the particular characteristics of each bonding agent.

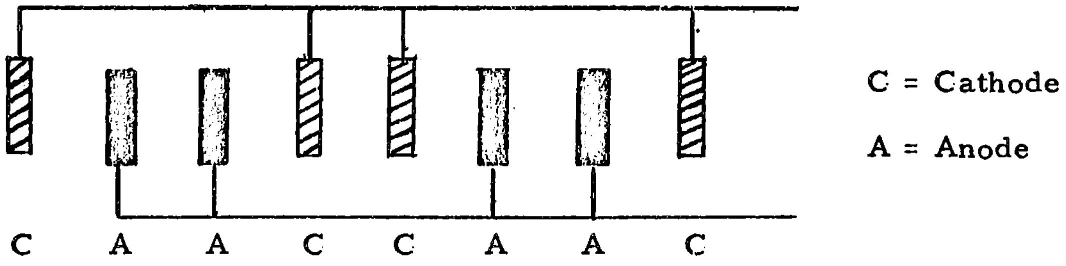
Many of the cells tested early in the Task III program used the S-7 expanded Teflon mesh separator in the fluid spaces. Flow distribution problems suggested that this type of separator was subject to creep and collapse during operation. Consequently, two alternate types of separators were used in many of the Task III cells. One design used the so-called S-6 separator described in Table XVIII. This separator consisted of multiple layers of flattened Teflon mesh. While the dimensional stability of this separator was excellent, the pressure drops were rather high. The other design was the serpentine stick separator which channels flow in a tortuous path across the face of the electrode. Serpentine stick configurations employed either vertical or horizontal sticks. (Fig. 13). Extensive flow testing indicated that the horizontal serpentine not only resulted in excellent flow distribution of electrolyte across the face of the electrodes but also tended to easily disgorge any gas bubbles from the cell. The final separator configuration evolved in the program employed an expanded polysulfone mesh in the gas spaces and a horizontal serpentine of polysulfone in the electrolyte space. No problems were encountered with this configuration.

3.2.3.1 Small Stacks for Use at 30 psia

The construction of the pressure cells was identical with that of the regular stacks except for the addition of a reinforcing shell. This shell consisted of 1/4" Panelite end plates bolted together over the basic stack, and potted on all four sides with epoxy. A typical unit is shown in Fig. 14.

3.2.3.2 Flight-Size Stacks

The so-called flight-size stacks are basically scaled-up versions of the small stacks, i.e., they are not prototypes of the probable building blocks for an aerospace system. Front and rear views of the stack are shown in Fig. 15.; details of construction are shown in Fig. 16.; and a unit installed in a test stand in Fig. 17. The stacks were of Type-404, meaning there were four pairs of electrodes electrically parallel, as shown below:



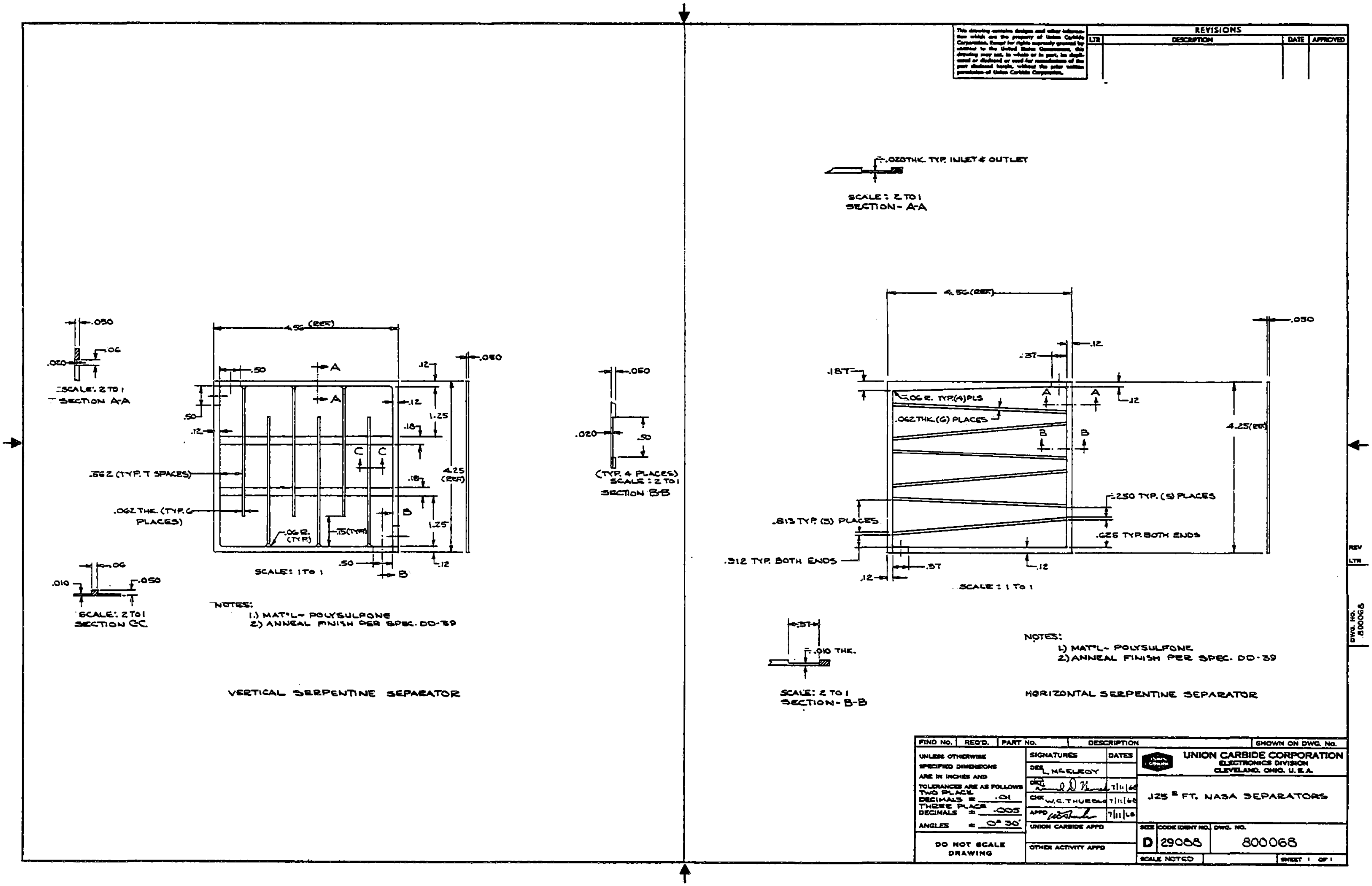
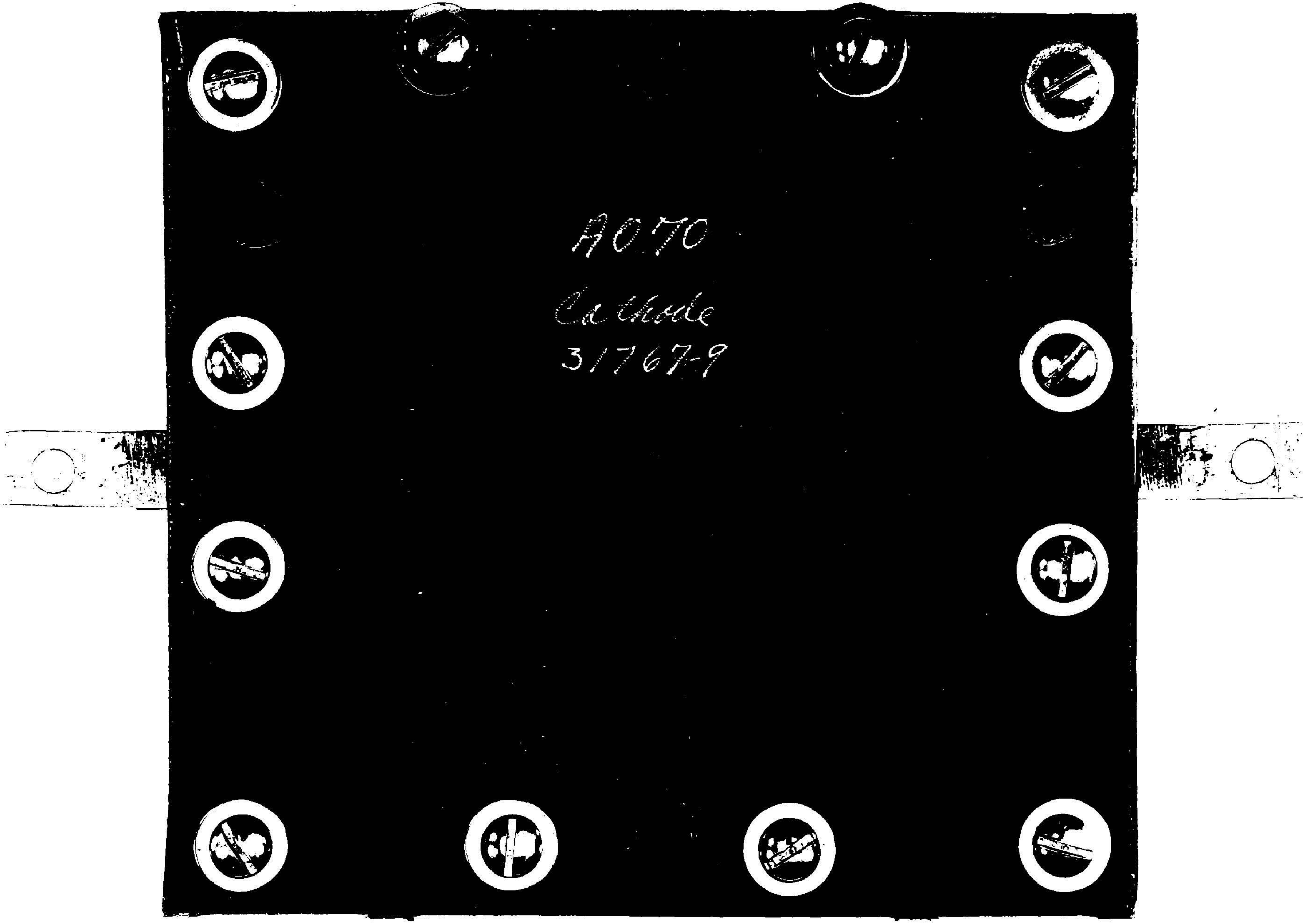
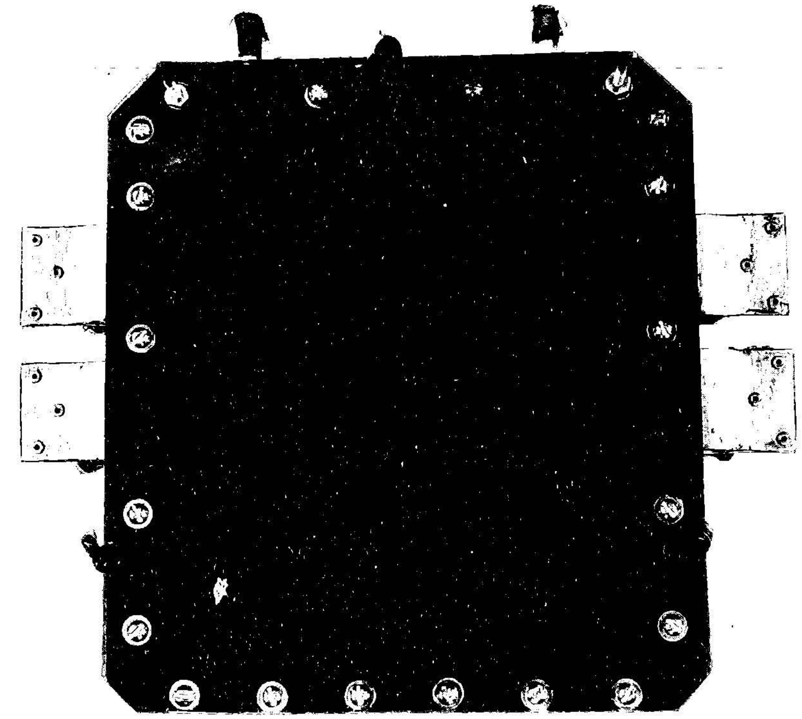


Fig. 13 - Serpentine Separator Designs

D-3394

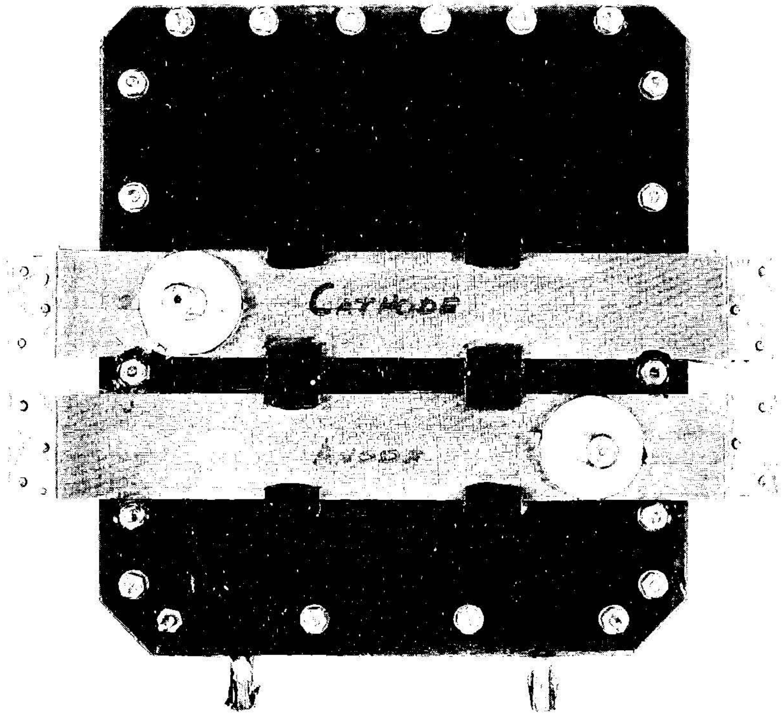
Fig. 14 - Small Stack for Operation at 30 psia; Finished Cell is about 6-1/2" x 7".


A070
Cathode
31767-9



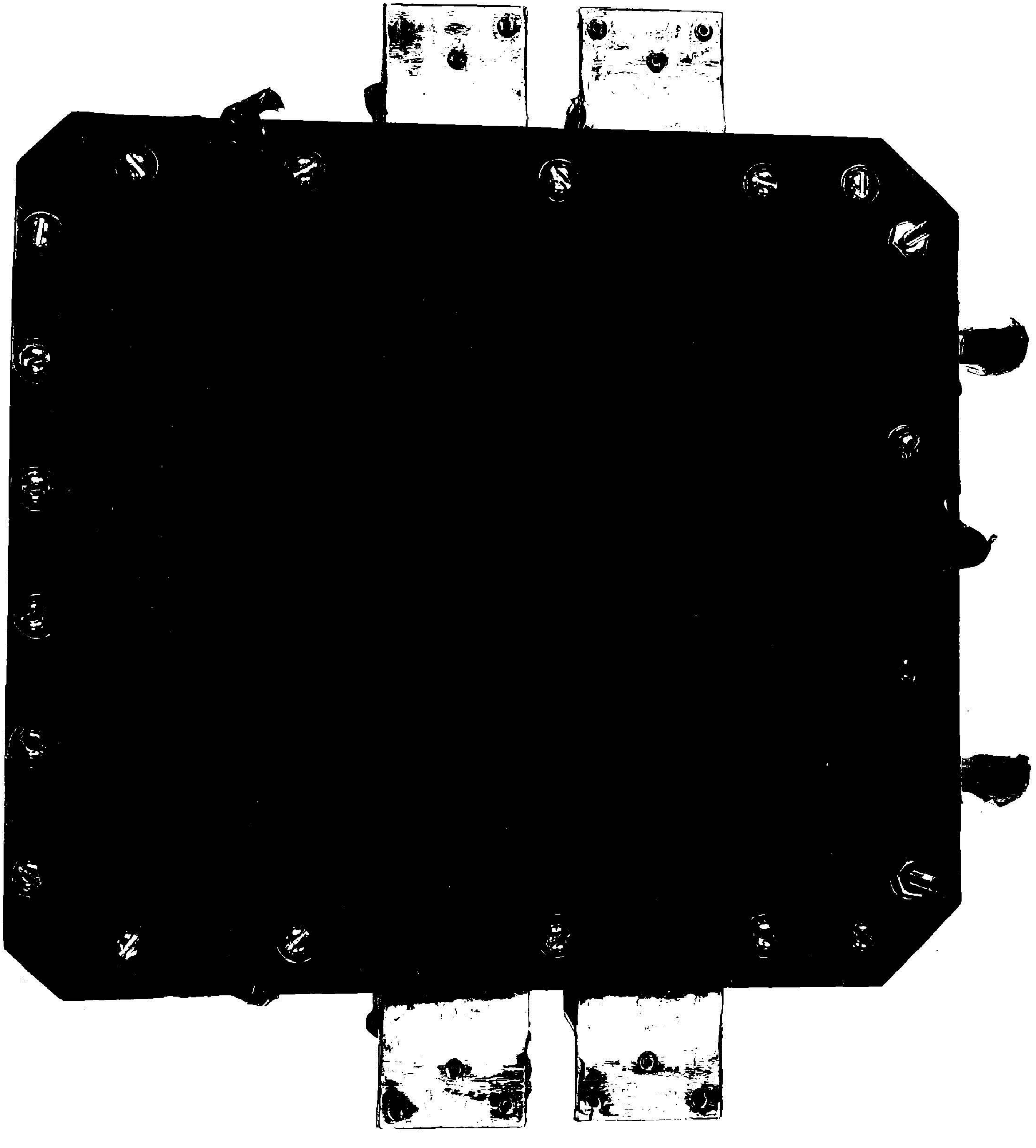
D-3403

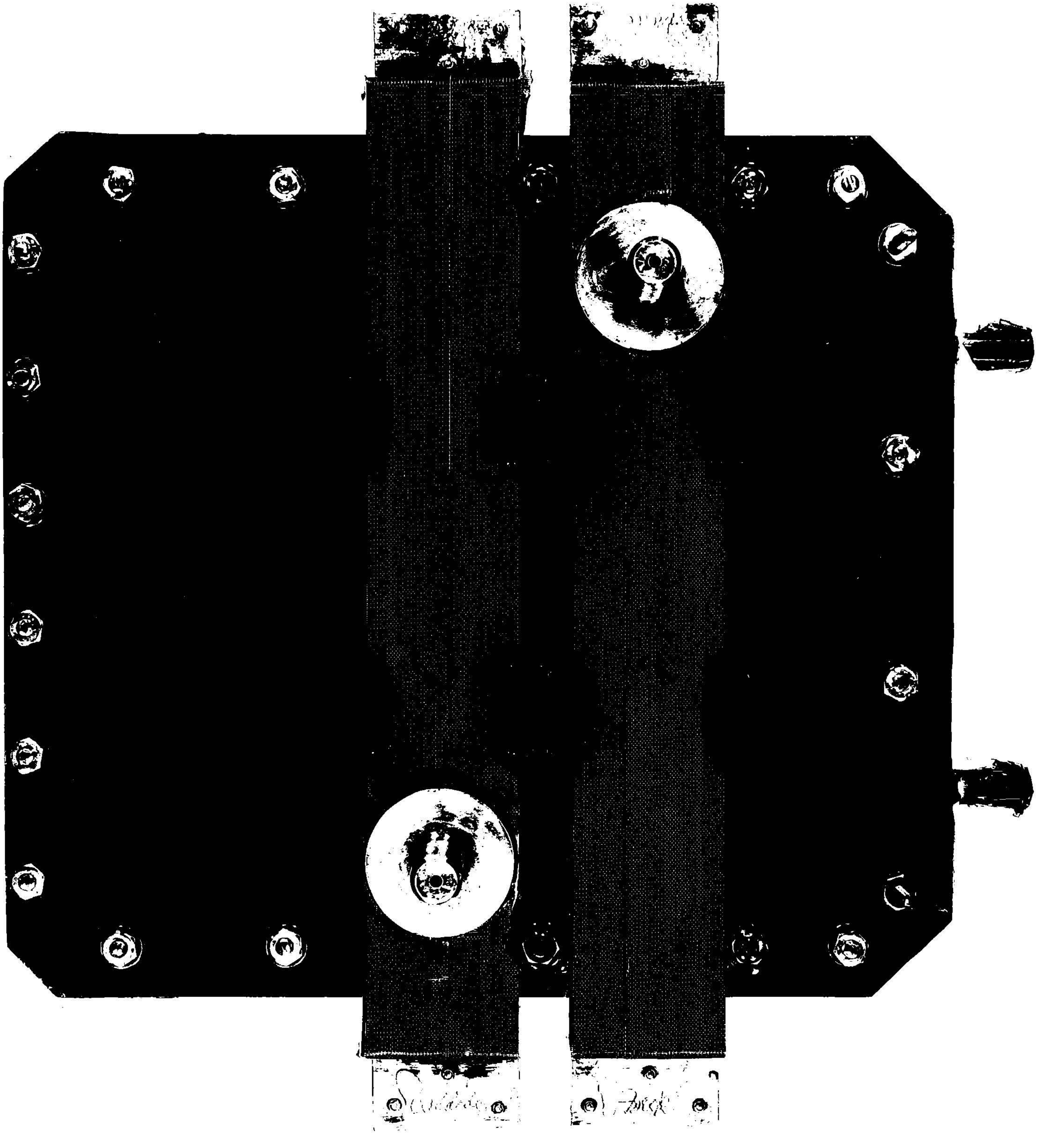
Fig. 15 a- Flight-Size Test Stack; Front View. (Finished Stack is about 8-7/16" x 9-7/16".)



D-3402

Fig. 15b- Flight-Size Test Stack; Rear View Showing Bus Bars.





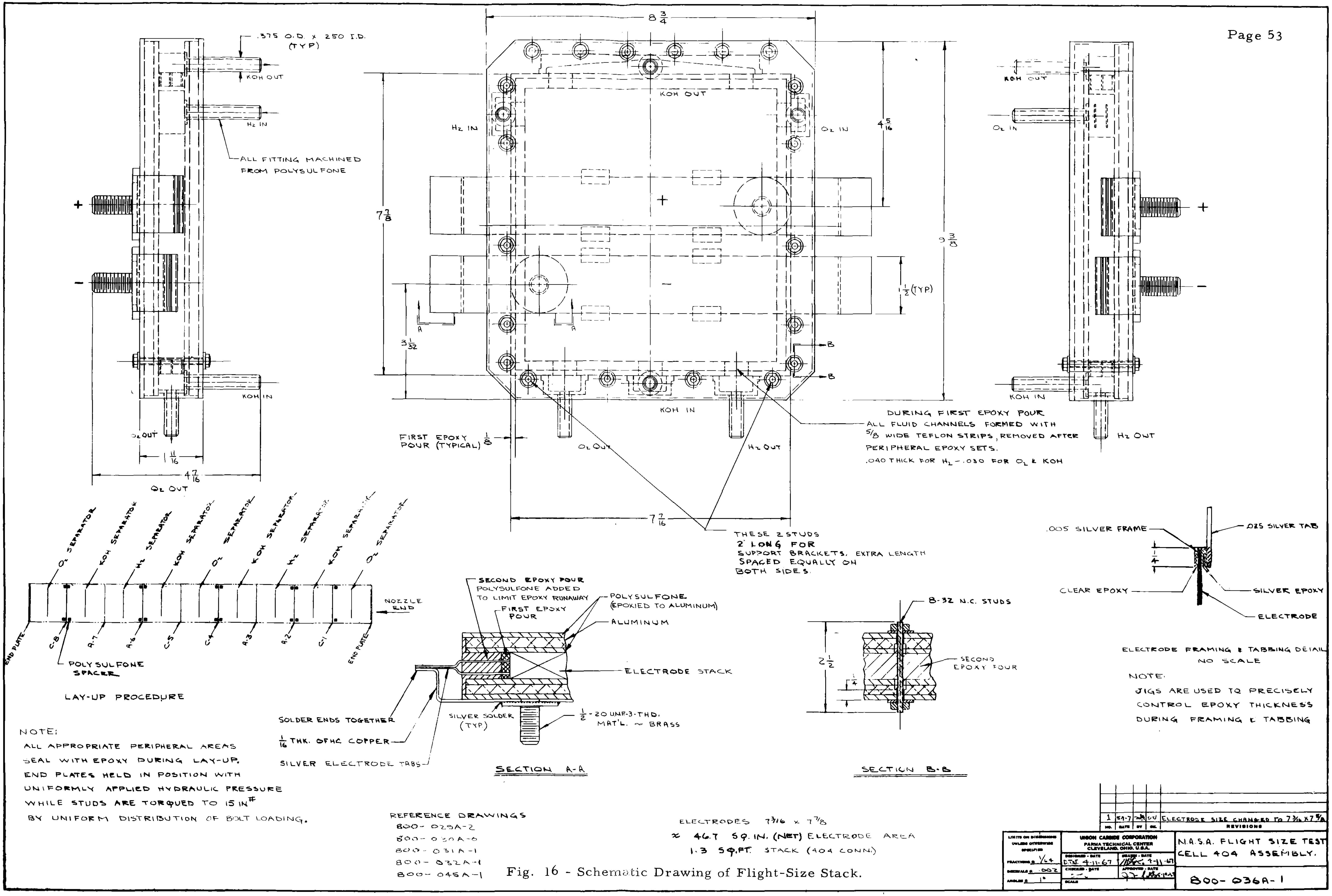
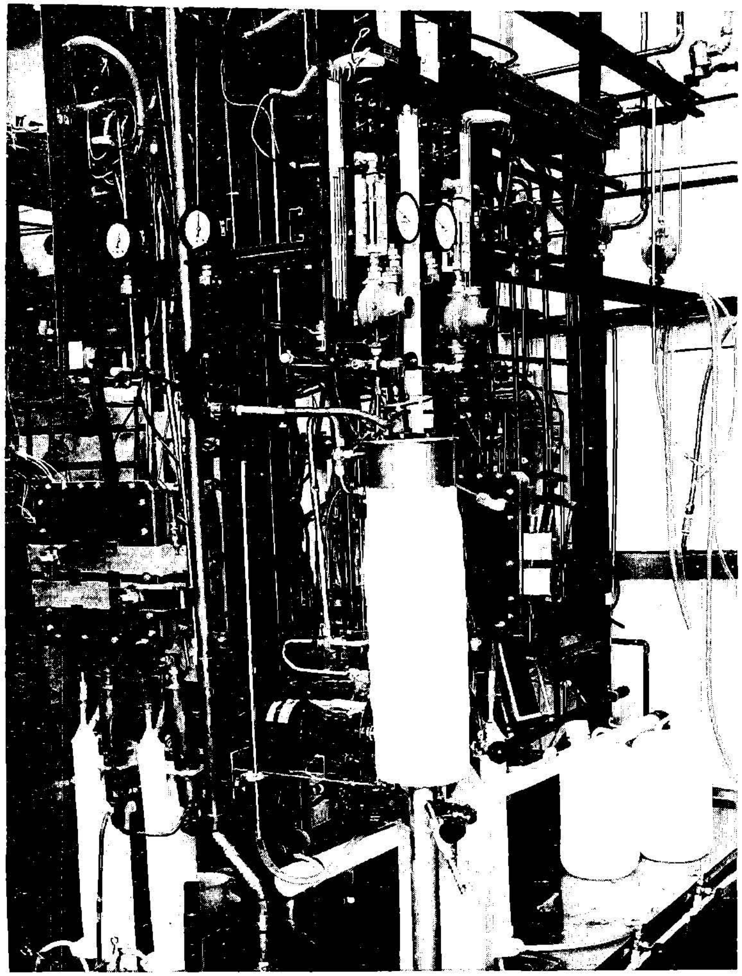
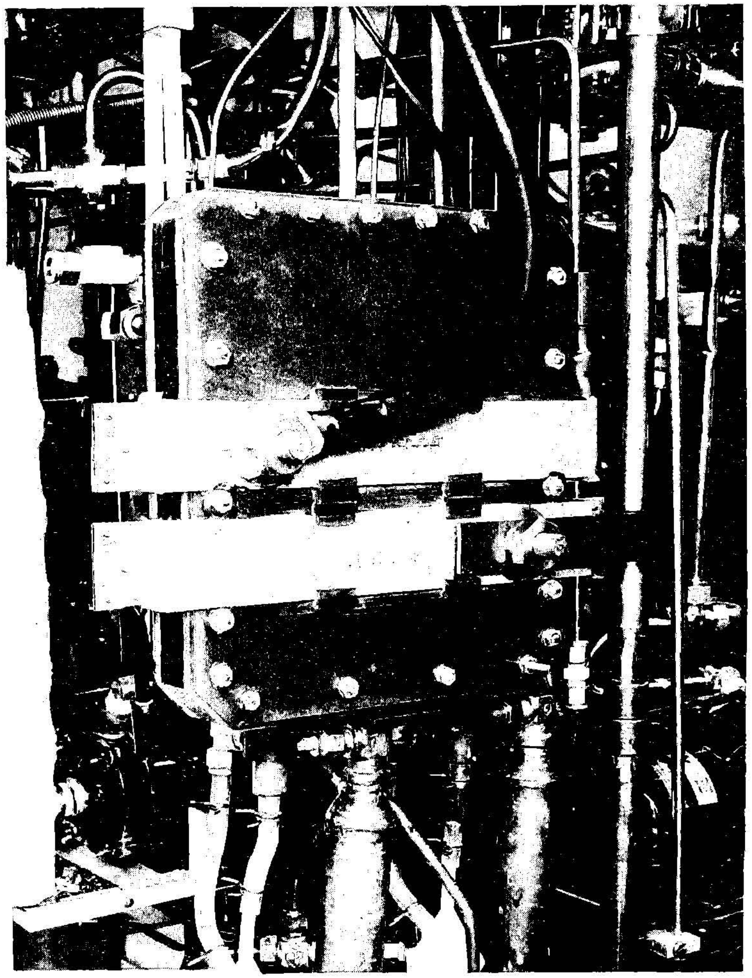


Fig. 16 - Schematic Drawing of Flight-Size Stack.



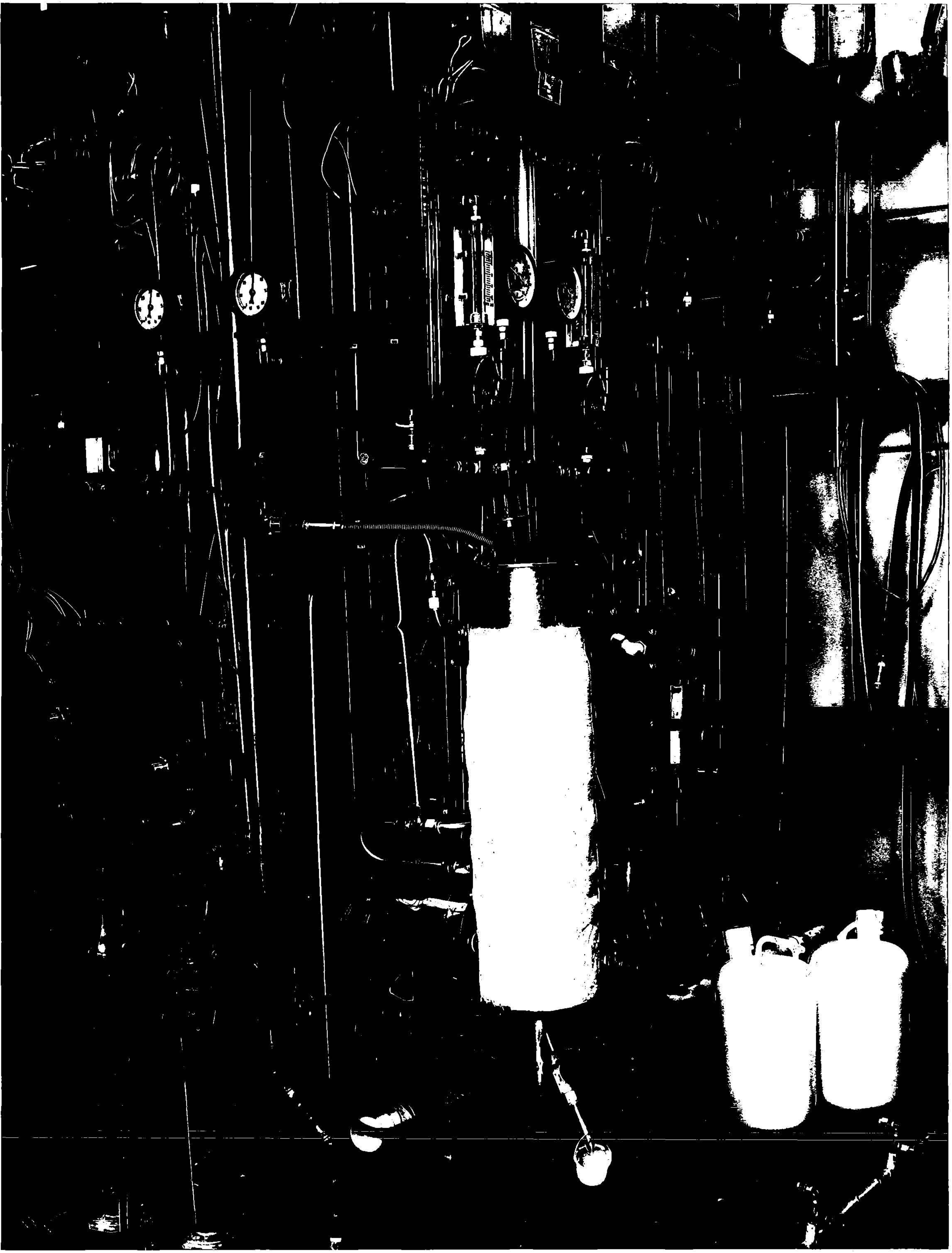
D-3436

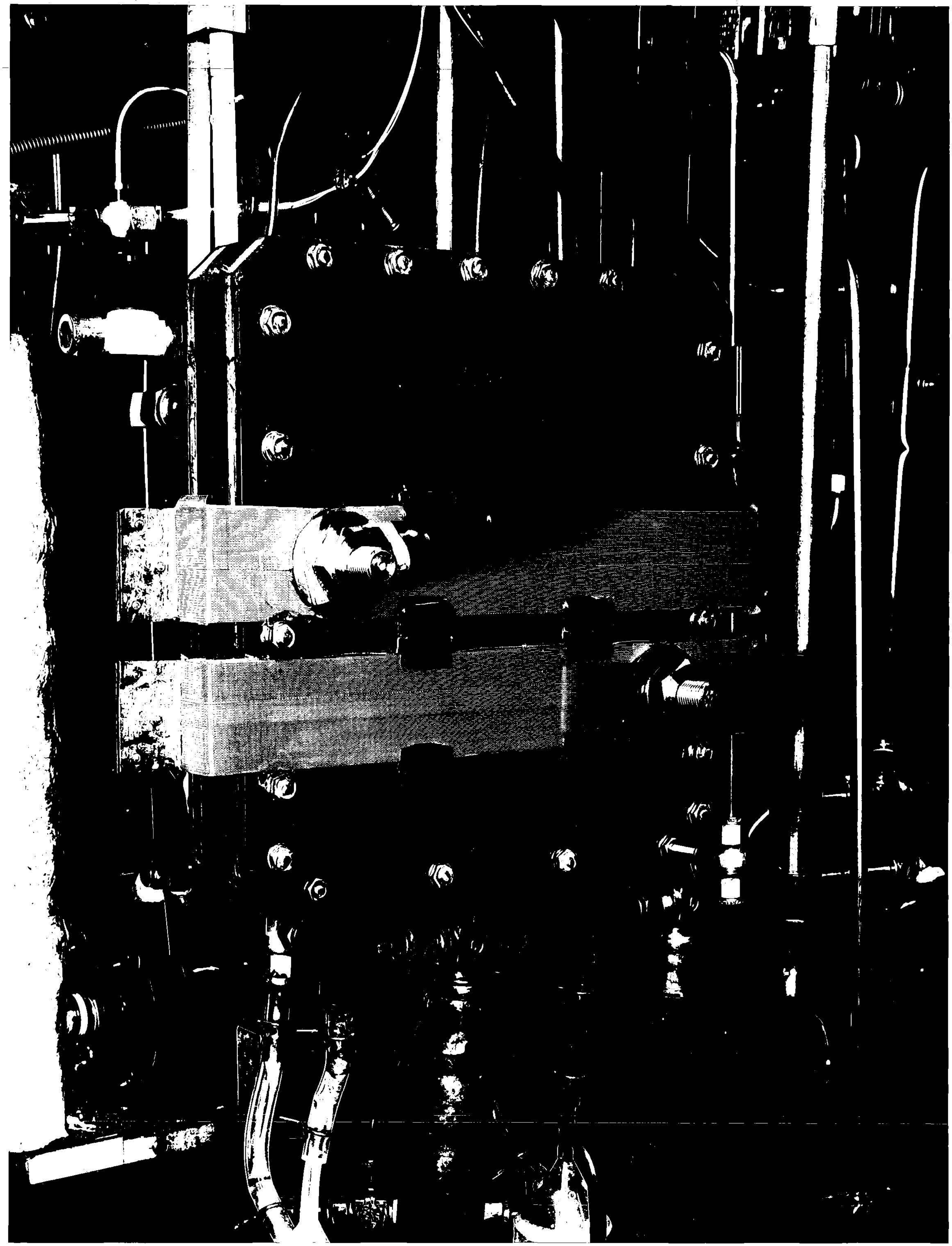
Fig. 17a- Front View of Flight-Size Stack in Test Stand C-10, Showing Panel and Electrolyte Tank.



D-3438

Fig. 17b- Rear View of Flight-Size Stack in Test Stand C-3.





This configuration was adopted to reduce the current per tab by providing an increased number of current collectors. Each electrode had an active area of 46.7 in^2, so that the cell had an active area of 1.3 ft^2; the contract called for a minimum of 1.0 ft^2. Even though operation of these cells at the specified 200 ASF required only 260 amperes, they are capable of handling 500 amperes.

The electrodes were framed in 0.005" silver foil, 1/4" wide on each side after folding. The silver was sealed to the LAB-40 electrodes on the gas side with clear epoxy, and on the electrolyte side with silver epoxy. For the Type-2 (T-2) electrodes this arrangement was again reversed, so that the silver epoxy was on the nickel (or gas) side of the anode. Furthermore, along one edge of each silver frame -- usually on the gas side -- was a 0.025" thick silver strip, 1/4" wide, with an integrally welded tab 1.5" wide to bring the current out of the cell. Tabs, later to be paralleled, were brought out on opposite sides in deference to the matching up of edge spacer thickness with fluid gap mesh thickness. The tabs were joined externally to appropriate copper bus bars, as shown in Fig. 15. There were also peripheral spacing strips of 3/16"-wide-polysulfone. This design does not incorporate fluid orifices; slits with the dimensions shown in Fig. 16 served as gas and electrolyte passages.

The end plates consisted of 1/4" aluminum sheets sandwiched between two 1/8" polysulfone sheets, glued together with epoxy. After the stack was aligned and assembled in a jig, with epoxy applied to all the appropriate mating surfaces, the stack was compressed slowly and uniformly with hydraulic pressure to assure uniform and continuous bonding by the adhesive. While the pressure was on the stack, bolts were inserted and tightened following a uniform torquing scheme, up to an applied torque of 15 inch-pounds per bolt. After bolting, the sides of the stack were potted with 2774/2793 epoxy. All nozzles were polysulfone.

3.2.4 Types of Cell Tests

The cell tests conducted under this contract were divided into several groups based on geometry and load cycle. These tests consisted principally of cells of the 1/8 ft^2 size with limited testing of the 0.325 ft^2 and the 1.3 ft^2 size cells. The 1/8 ft^2 cells were broken down in the following manner:

35 cells - 200 ASF - 15 psia
5 cells - 200 ASF - 30 psia
4 cells - NASA cycle - 15 psia
1 cell - NASA cycle - 30 psia
3 cells - 100 ASF - 15 psia
2 cells - 300 ASF - 15 psia
4 cells - 200 ASF - 15 psia with T-3N Cathode

Five tests of 1.3 ft^2 (Flight Size) cells were conducted at 200 ASF (three at 30 psia and two at 15 psia) and three tests of the 0.325 ft^2 cells were conducted at 200 ASF and 15 psia. Lastly, a group of five special tests were conducted under a variety of conditions. Summaries for each of these groups of cells will follow.

Tables have been prepared which summarize construction features, operating conditions, and cell performance. In each case the table of cell construction shows the type of electrode and, when appropriate, type of backing materials used on the LAB-40 electrode. The separator material used in the anode, cathode, and electrolyte compartments of each cell was explained in Table XVIII. The method of attaching the current collector to the electrode and the type of epoxy used for potting of the cell are tabulated. When the "UCC" epoxy is stipulated, it refers to the 2774/2793 mixtures. This table also shows the time to the first leak and the average leakage. The average leakage is obtained by integrating all electrolyte leakage from time zero until time the voltage level falls to 0.780 volt and dividing by the time increment.

The table of operating conditions shows the cell type, operating pressure, initial voltage, voltage at time when cell was removed from test, life to 0.780 v and total time that the cell remained on test. Initial and final pressure drops of the electrolyte and gases and the initial and final internal resistance values are included. The failure modes shown here are associated with a nine-point failure mode index developed by Union Carbide for analysis of cell tests.

Finally, the table of cell performance shows the activation date, the peak voltage output, the cell voltage at 100 hr and 200 hr increments thereafter until voltage level reached 0. 780 v or the voltage level at termination if in excess of 0. 780 v. The lifetime to 0. 780 v is the actual number of hours that cell voltage level exceeds 0. 780 v. The lifetime degradation of 8 mv/200 hr and 40 mv/1000 hr are based on smoothed data curves. Task I Milestone 1 answers the question of whether the initial or peak voltage was equal to or exceeded 0. 900 v, and milestone 2 answers the question of whether the actual voltage degradation rate was equal to or less than 20 mv during the first 500 hrs. These data are based on the actual daily measurements and not on smoothed data. The cause of failure shown here is the actual explanation of the cell failure and is more definitive than the failure modes shown on tables delineating operating conditions.

Envelopes for the initial and 500 hr IRI (internal resistance included) polarization curves were generated from the first fifteen life test cells* operated at 200 ASF, 15 psia during Task I of this contract. These curve envelopes are used as a guideline of acceptable cell performance. Polarization data falling outside of these envelopes are shown. If all curves are within the envelope, no graph is presented.

3. 2. 4. 1 1/8 ft^2 Cells Operated at 200 ASF and 15 psia

Approximately half of cells tested under this contract were of the 1/8 ft^2 design operated at 200 ASF and at atmospheric pressure. Thirty-six cell tests of this type were conducted, of which 15 were all LAB-40 type and 21 were hybrids using the Union Carbide T-2 anode coupled to the LAB-40 cathode. Cell construction, operating conditions, and performance can be seen in Tables XX, XXI, and XXII.

Of these 36 cells, nine (3 of LAB-40 and 6 hybrids) met Task I Milestone 1 (initial voltage level equal to or exceeding 0. 900 v) and fifteen (3 LAB-40 and 12 hybrids) met Milestone 2 (less than 20 mv degradation per 500 hours). Four cells met both criteria (A-043, A-055, A-066, and A-077). Mile-

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* i. e. , Cells A-015 to A-087

TABLE XX

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 1/8 ft^2 CELLS TESTED AT 200 ASF, 15 psia

			Separator					Leakage			
								Cathode		Anode	
Cell No.	Anode Type	Cathode Type	Cathode	Anode	Electrolyte	Current Collector	Epoxy	Time to First Leak (Hrs)	Average(1) (ml/hr • ft^2)	Time to First Leak (Hrs)	Average(1) (ml/hr • ft^2)
A015	LAB-40nd*	LAB-40nd	S-1	S-1	S-2	Silver Epoxy	UCC	1	22.0	1	2.00
A021	LAB-40nd	LAB-40nd	S-1	S-1	S-2	Silver Epoxy	UCC	1	6.8	1	1.90
A022	LAB-40nd	LAB-40nd	S-1	S-1	S-2	Silver Epoxy	UCC	1	10.0	1	1.20
A043	UCC-T-2	LAB-40nd	S-1	S-1	S-2	Silver Epoxy	UCC	1	37.0	1	.30
A055	LAB-40(A-II)	LAB-40 (A-II)	S-6	S-1	S-2	Silver Epoxy	UCC	1	5.6	1	.40
A059	UCC-T-2	LAB-40 (A-II)	S-6	S-1	S-2	Silver Epoxy	UCC	480	6.5	1	1.10
A060	UCC-T-2	LAB-40 (A-II)	S-6	S-1	S-2	Silver Epoxy	UCC	1	36.0	200	.05
A066	UCC-T-2	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC	1	1.2	1	.70
A068	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC	570	1.0		.02
A071	UCC-T-2	LAB-40 (A-II-1)	S-7	S-1	S-7	Silver Epoxy	UCC	240	.08	1	.12
A075	LAB-40(A-II-1)	LAB-40 (A-II-1)	S-7	S-1	S-7	Silver Epoxy	UCC	240	.20		0
A077	UCC-T-2	LAB-40(B-II-4)	S-7	S-1	S-7	Silver Epoxy	UCC	140	3.00	1	.05
A079	LAB-40(B-II-4)	LAB-40(B-II-4)	S-7	S-1	S-7	Silver Epoxy	UCC	240	3.10	1	.01
A081	UCC-T-2	LAB-40 (143-A)	S-7	S-1	S-7	Silver Epoxy	UCC		.02		.02
A087	UCC-T-2	LAB-40 (143-B)	S-7	S-1	S-7	Silver Epoxy	UCC	1	6.40	1	.14
A100	UCC-T-2	LAB-40 (B-II-4)	S-7	S-1	S-7	Silver Epoxy	UCC	1	3.2	1	1.60
A101	UCC-T-2	LAB-40 (79-1)	S-6	S-1	2(S-6)	Seam Welded Ni	UCC	1	94.0(2)		0
A102	UCC-T-2	LAB-40(79-2)	S-6	S-1	S-6	Seam Welded Ni	UCC	1	69.0(2)	1	.04
A103	UCC-T-2	LAB-40 (143B)	S-7	S-1	S-7	Silver Epoxy	UCC				0
A104	UCC-T-2	LAB-40 (143A)	S-7	S-1	S-7	Silver Epoxy	UCC	1	13.5	1	.26
A105	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-8	S-8	S-8	Seam Welded Ni	UCC	1	.09	1	.17
A107	UCC-T-2	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Hysol	1	.06	240	.01
A109	UCC-T-2	LAB-40 (B-II-4)	S-9	S-9	S-9	Seam Welded Ag	Ring 101-F	250	.002	550	0
A110	UCC-T-2	LAB-40 (B-II-4)	S-9	S-9	S-9	Seam Welded Ag	Ring 101-F	225	18.0	625	2.70
A112	LAB-40(B-II-4)	LAB-40 (B-II-4)	S-6	S-6	2(S-6)	Seam Welded Ag	Ring 101-F	No test			
A113	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Rezolin	100	4.6	1	5.70
A114	UCC-T-2	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Rezolin	1	7.3		0
A116	UCC-T-2	LAB-40 (B-II)	S-9	S-9	S-9	Seam Welded Ag	Rezolin	450	17.4	1	.004
A117	LAB-40(B-II-4)	LAB-40 (B-II-4)	S-9	S-9	S-9	Seam Welded Ag	Rezolin	70	0	1	0
A118	UCC-T-2	LAB-40 (B-II)	S-9	S-9	S-9	Seam Welded Ag	Bjorksten/ Rezolin	1	.16	1	High
A119	LAB-40 (C-II)	LAB-40 (C-II)	S-6	S-6	S-6	Seam Welded Ag	Rezolin	425	0	70	0
A120	LAB-40 (C-II)	LAB-40 (C-II)	S-6	S-6	S-6	Seam Welded Ag	Rezolin	625	0	1	.16
A123	UCC-T-2	LAB-40 (B-II)	S-6	S-6	S-6	Seam Welded Ag	Bjorksten/ Rezolin	130	.4	72	.50
A125	LAB-40(C-II)	LAB-40 (C-II)	S-11	S-11	S-10	Seam Welded Ag	Bjorksten/ Rezolin	1	.03	48	.02
A126	LAB-40(C-II)	LAB-40 (C-II)	S-11	S-11	S-10	Spot Welded Ag	Ring 101-F Rezolin	No data			
A127	UCC-T-2	LAB-40 (C-II)	S-11	S-11	S-10	Spot Welded Ag	Ring 101-F Rezolin	1		1	

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*nd No designation beyond LAB-40 is available for the earliest lots of American Cyanamid electrodes

(1) Average leakage is obtained by integrating all KOH leakage from time zero until time when voltage falls to 0.78 volt and dividing by that time increment.

(2) High leakage due to improper preparation of current collector tab area during fabrication.

TABLE XXI

OPERATING CONDITIONS OF 1/8 ft^2 CELLS AT 200 ASF, 15 psia

Cell No.	Type	Pressure psia	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop KOH at 125 ml/min (inch KOH)		H_2 inches H_2O		O_2		Internal Resistance (m Ω · ft^2)		Failure Mode
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A015	101	15	.880	<.500	1620	1794			.10	.30	1.20	4.2	.30	.30	Test stand equipment
A021	101	15	.880	.695	1910	2030	.98		.05	.15	1.50	>10.0	.26	.50	Test stand equipment
A022	101	15	.885	.500	1394	1394	2.00		.10	.90	.60	5.1	.25	.30	Cell construction, test stand equipment
A043	101	15	.910	.875	918	918	2.40		.05	.10	.90	2.4	.31	.31	Cell construction, test stand equipment
A055	101	15	.905	.765	1230	2219	2.60		.05	1.00	.40	7.0	.29	.30	Anode
A059	101	15	.870	.680	2160	2284	.65	.86	.10	.10	.20	>10.0	.45	.70	High internal resistance, Anode
A060	101	15	.900	.750	1370	1514	1.40	1.10	.10	.10	.15	>10.0	.33	.43	Both anode & cathode
A066	101	15	.910	.400	1386	1522	2.60		.10	.90	.60	4.7	.32	.60	Both anode & cathode, test stand equipment, High IR
A068	101	15	.910	.680	930	1054		16.00	.10	.15	.20		.22	.26	Anode. Cell construction
A071	101	15	.890	.700	945	969	3.50		.10	.30	1.20	4.2	.37	.50	Both anode & cathode
A075	101	15	.910	.740	950	980			.30	.40	.20	2.8	.31	.37	Anode. test stand equipment
A077	101	15	.900	<.600	1030	1078	3.00		.05	.10	4.00	4.0	.33	.35	Both anode & cathode. cell construction
A079	101	15	.895	.720	1260	1683			.10		.20		.25	.33	Anode
A081	101	15	.905	.650	690	812	2.00		.10	.60	.80	3.0	.55	.70	High internal resistance. both anode & cathode
A087	101	15	.900	<.500	710	912	2.80	5.20	.15	.15	.50	1.8	.47	.60	Anode. Cell construction, High IR
A100	101	15	.895	.752	1530	1580	3.50	55.00	.10	.10	.30	1.2	.30	.40	Cell construction
A101	101	15	.890	.857	888	888	1.10	1.60	.10	.10	.10	.1	.40	.40	Excessive leakage
A102	101	15	.850	.840	1024	1024	1.20	2.00	.10	.10	.10	.2	.50	.55	Excessive leakage
A103	101	15	.880	.869	42	42	12.90		.90	.40	.40	.5	No Data		Cell construction
A104	101	15	.860	.833	114	114	9.60		.10	.10	.10	.3	.30		Excessive leakage
A105	101	15	.860	.712	813	1034	1.80	11.00	.10	.40	.15	.2	.35	.38	Both anode and cathode
A107	101	15	.859	<.100	55	507	15.00		.25	.45	.80	1.9	.35	.80	Anode. High IR
A109	101	15	.857	.340	270	1648	7.00		.20	.05	.60	1.65	.30	.65	Both anode and cathode, High IR
A110	101	15	.880	.490	1226	1445	15.00	14.00	.15	.30	1.50	1.25	.35	.45	Both anode and cathode
A112	101	15		No Test											Cell construction
A113	101	15	.885	.815	120	120	8.40		.25	.20	.45	.65	.30		Cell construction
A114	101	15	.885	.865	658	912	5.00	5.60	.20	.15	.50	1.90	.30	.45	Anode, excessive leakage
A116	101	15	.877	.865	1224	1224	5.00		.05	.05	.10	.35	.65	.40	Excessive leakage
A117	101	15	.830	.703	24	238	1.35	2.40	.10	.25	.15	.40	.35	.60	Anode, High IR
A118	101	15	.837	<.100	60	72	3.70		.05	.20	.45	.45	.30		Cell construction
A119	101	15	.775	.650*	0	1024	3.90	4.40	.50	3.50	.20	.40	.49	.49	Anode
A120	101	15	.800	.650	22	1351	3.70	3.70	.05	.35	.15	.25	.42	.46	Both anode and cathode
A123	101	15	.865	.625	930	1292	42.00		.45	.45	.22	.75	.40	.50	Both anode and cathode
A125	101	15	.856	.610	71	386	8.60	9.20	.05	.05	.10	.30	.30	.32	Anode
A126	101	15	.805	.438*	24	40	37.50		.20	.20	.50	1.10			Cell concentration
A127	101	15	.890				18.20		.20		.45		.30		

* at 100 ASF

TABLE XXII

PERFORMANCE SUMMARY OF 1/8 ft^2 CELLS AT 200 ASF, 15 psia

			Cell Voltage												Lifetime (Hours)			Task I Milestones		
Stack No.	Activation Date	Peak	100 Hours	300 Hours	500 Hours	700 Hours	900 Hours	1100 Hours	1300 Hours	1500 Hours	1700 Hours	1900 Hours	2100 Hours	End or .780V	to .780V	8 mV/200 Hrs	40 mV/1000 Hrs	1	2	Cause of Failures
A015	12/01/66	.880	.880	.880	.880	.880	.875	.865	.850	.790					1620	1124	1370	No	Yes	Test stand problem
A021	1/09/67	.880	.880	.870	.850	.835	.825	.810	.800	.800	.795	.780			1910	410		No	No	KOH pump failure
A022	1/04/67	.885	.880												199	180		No		Internal short
	2/03/67*	----	----	.880	.870	.860	.850	.840	.840						1394	410	1335		No	
A043	1/19/67	.910	.900	.900	.905	.900	.890							.875	918	715		Yes	Yes	Electrolyte blockage, fire in cell
A055	3/10/67	.905	.905	.890	.890	.870	.870	.825		.835					1230	240	1000	Yes	Yes	Poor anode
A059	2/22/67	.870	.870	.870	.870	.870	.870	.880	.870	.850	.835	.800	.795		2160	1415	1728	No	Yes	Poor anode, high int. res.
A060	2/27/67	.900	.900	.880	.875	.860	.855	.835	.800						1370	240		Yes	No	Poor anode, weak cathode
A066	4/12/67	.910	.910	.910	.905	.885	.870	.860	.840						1386	675	1024	Yes	Yes	House H_2 supply failed
A068	4/18/67	.910	.905	.890	.865	.815	.790								930	180		Yes	No	Electrolyte blockage. Anode
A071	4/14/67	.890	.890	.890	.890	.870	.840								945	525		No	Yes	Poor electrodes
A075	5/09/67	.910	.890	.890	.870	.825	.780								950	450		Yes	No	House H_2 supply failed
A077	4/14/67	.900	.900	.915	.905	.885	.885								1030	509	1030	Yes	Yes	Cracked KOH manifold
A079	4/17/67	.895	.895	.890	.875	.865	.845	.825		.810					1260	360		No	Yes	Poor anode
A081	4/28/67	.905	.900	.885	.780										690	120		Yes	No	Poor electrodes, high int. res.
A087	4/27/67	.900	.895	.880	.840	.820									710	120		Yes	No	Electrolyte blockage. Poor anode
A100	7/19/67	.895	.880	.873	.890	.886	.891	.882	.891	.840					1530	1320	1488	No	Yes	Electrolyte flow blockage
A101	8/14/67	.890	.882	.882	.881	.875								.857	888	768		No	Yes	Cathode seal leak
A102	8/09/67	.850	.840	.838	.846	.833	.830							.840	1024	1024	1024	No	Yes	Cathode seal leak
A103		.880												.869	42			No	No	Electrolyte flow blockage
A104	7/23/67	.860	.833											.833	114	114		No	No	High cathode leakage. Loose backing
A105	8/23/67	.860	.848	.850	.825	.807									813	430		No	No	Poor electrodes
A107	12/05/67	.859													55			No	No	Poor anode
A109	11/21/67	.857	.838												270	120		No	No	Poor electrodes
A110	11/22/68	.880	.868	.867	.864	.844	.824	.799							1226	672		No	Yes	Poor electrodes
A112	Shorted																			
A113	12/8/67	.885	.866											.815	120	70		No	No	Loose cathode backing. Liquid in H_2 space
A114	1/04/68	.885	.878	.865	.877									.865	658	658		No	Yes	Poor anode. High cath. leakage
A116	2/07/68	.877	.860	.870	.858	.847	.872	.870						.865	1224	1224	1224	No	Yes	Cathode seal leak
A117	2/08/68	.830													24			No	No	Poor anode
A118	1/25/68	.837													60			No	No	Short near H_2 inlet
A119	2/19/68	.775													0			No	No	Poor anode
A120	2/26/68	.800													22			No	No	Poor electrodes
A123	3/01/68	.865	.820	.800	.813	.808	.801								930	930		No	No	Poor electrodes
A125	4/09/68	.856													71			No	No	Poor anode
A126	4/26/68	.805													24			No	No	Pump failure. Fire in stack
A127	5/02/68	.890	.887	.883	.880	.870	.831	.856	.851	.848	.844							No	Yes	Still on Test (7/15/68)

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

* Stack potential reversed at 221 hrs. Stack washed, dried and returned to test. Time runs continuously

stone 1 was not met by any cell tested under Task III, and Milestone 2 was not met by any of the all-LAB-40 cells tested under Task III. Seven of the hybrid cells tested under Task III met this second milestone.

The performance of cells tested under Task III can be readily seen from Fig. 18 which shows the initial polarization curve envelopes of Task I and Task III cells. Figure 19 also shows clearly the rapid deterioration after 500 hours.

Cell test terminations were due to a variety of reasons, and thirteen cell terminations had more than one contributing factor. Eleven of the cells were terminated because of poor anodes (7 with LAB-40 anodes, and 4 with T-2 anodes), ten because of both poor anodes and cathodes (2 with LAB-40 anodes and 8 with T-2 anodes), eleven had cell construction problems, six suffered from test stand equipment problems, four experienced excessive leakage, and seven had high internal resistance. It is interesting to note that not one cell failure was attributable exclusively to low cathode potential.

Electrolyte leakage was one of the major problems which plagued these tests. Various construction changes were made in an attempt to solve this problem, but at termination of the contract the problem had not been completely solved, although some improvement was made. The leakage data in Table XX are reported only during the time the cell voltage exceeded 0.780 volt. In certain cases, as A-119, the cell never operated above this voltage, so no leakage value is reported.

In several cells operated during Task III, an interesting phenomenon involving bubbling of H_2 into the electrolyte was observed. Bubbling-through occurred even when the hydrogen pressure was less than the KOH pressure.

Initial H_2 blow-through in the KOH at 0.4 SCFH air (normal operating condition) was observed on all cells from A-116 to A-125. This blow-through could be reduced by reducing the H_2 flow. However, when cells were initially activated, H_2 blow-through would occur with no H_2 purging across the anode (i.e., only use rate being supplied to cell). This high initial blow-through continued for at least several days, and occasionally would stop some time between the fifth and tenth day. Both Union Carbide and Cyanamid anodes displayed this phenomenon. On one cell (A-119) a humidifier was added on the H_2 inlet, and the blow-through was greatly reduced. This phenomenon, which appears to be a function of the wet-

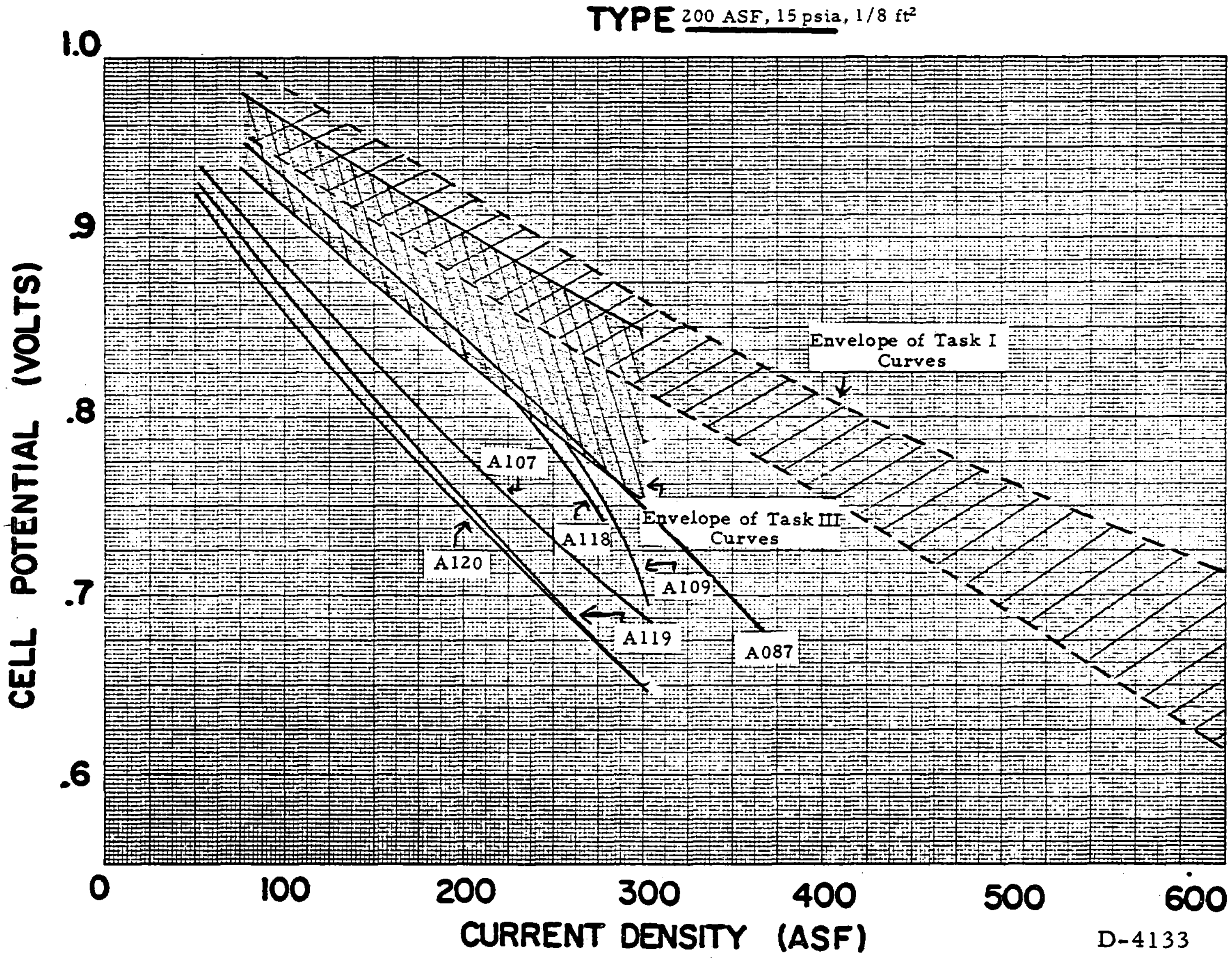

Fig. 18 - Envelope of Initial IRI Polarization Curves at 200 ASF.

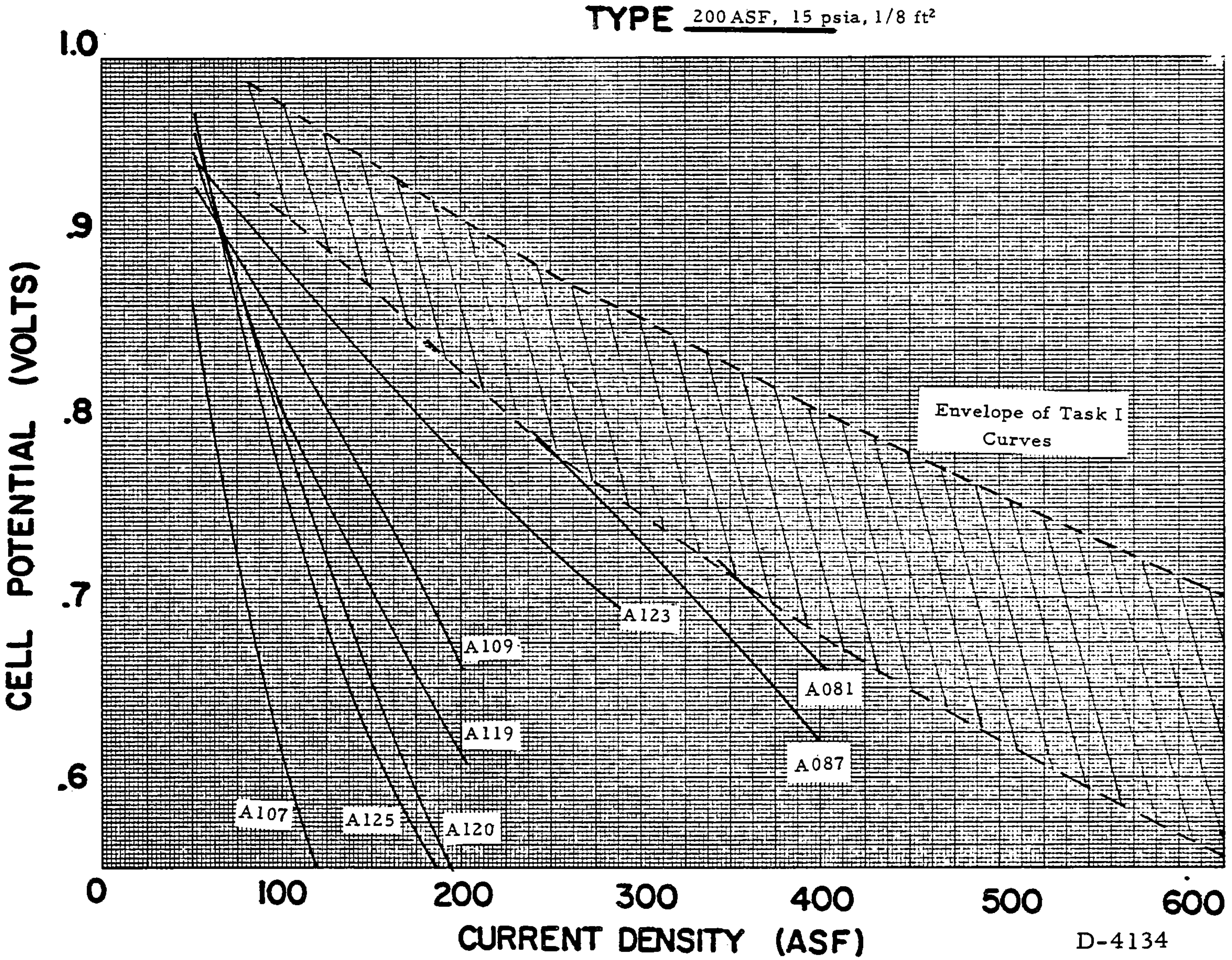

Fig. 19 - Envelope of IRI Polarization Curves After 500 Hours at 200 ASF.

proofing, has been previously observed, studied, and reported by J. H. Fishman, R. H. Kislow, and N. I. Palmer in "Nature," Vol. 211, No. 5056, p. 1401 (1966).

In an attempt to determine if the porting material or the actual washing of the cell could be the cause of this blow-through, 12 quick cell tests were conducted. Six cells were constructed using LAB-40 cathodes and T-2 anodes; and six, using all-LAB-40 electrodes. Cells were assembled and separated into three equal groups, each group containing two of the T-2 paired anodes. Small pieces of the water soluble plastic were placed into the electrolyte and gas chambers of Group I. This group, plus Group II, was washed with hot water for a period of 15 hours and drained dry. As desired, all the soluble plastic was washed away.

These tests showed significant hydrogen blow-through occurring only on cells incorporating LAB-40 anodes. This is not true of the standard NASA cell tests, as both LAB-40 and T-2 anodes have shown comparable blow-through phenomenon. The quick cell tests which exhibited blow-through were the following: the two cells in which the water-soluble ports had been placed and washed away, the one cell which had been only washed, and the one untreated cell. These four cells also displayed rapid voltage degradation of the anode. LAB-40 anodes were initially poor in all instances.

In the quick cell tests, no separator was used, so neither the blow-through nor the low voltage can be related to any type of separator bubble-trapping phenomenon.

These data were not well defined, and no definite conclusions regarding electrode treatment could be drawn. A further experiment was conducted on a 1/8-ft^2 special half-cell with a transparent window to view the KOH channel. (See Special Cells Section, page 102). This test proved that this blow-through phenomenon could in no way be related to the electrochemical performance of the cell, nor to the dissolvable port material, but could be related to the washing of the cell. Cells A-126 and A-127 were constructed with no washing of the electrodes, and no blow-through was observed on either of these cells.

This finding confirmed the suspicion that the washing of the electrodes caused the blow-through.

The normal internal resistance of the 1/8-ft^2 cells operated at 200 ASF and 15 psia was ~0.30 to 0.35 $m\Omega \cdot ft^2$. Nine cells indicated initial high internal resistance in excess of 0.40 $m\Omega \cdot ft^2$, and seven cells had final internal resistance in excess of 0.60 $m\Omega \cdot ft^2$. In four cells the internal resistance increased by a factor of two.

Backing delamination was another problem that occurred on many of the more recently tested cells. A limited number of cells also suffered from inadequate electrolyte flow caused by a flow blockage probably related to this delamination problem or to disintegration of the separator material. Cells A-125 and A-127 utilize a horizontal type separator stick, and have displayed a high electrolyte-pressure drop.

Nine cells tested in this group exceeded 1000 hours with a voltage degradation of less than 40 mv/1000 hours, but none of these maintained this degradation rate for 2000 hours. The longest test was Cell A-059 which operated for 2,160 hours to a final potential of 0.78 volt, and maintained a degradation rate of less than 40 mv/1000 hours for 1.728 hours.

3.2.4.2 1/8-ft^2 Cells Operated at 200 ASF and 30 psia.

Since there is no reason why a device intended for use elsewhere than on the surface of the earth should operate at a pressure of 15 psia, a group of eight cells was built for operation at 30 psia. Considerations such as the change in chemical activity with pressure led us to expect higher potentials from fuel cells operating at higher pressures; this proved to be the case. Of the eight cells, only five (three of the LAB-40 type, and 2 hybrids) were actually operated at 30 psia. The remaining three suffered from mechanical difficulties. Tables XXIII, XXIV, and XXV, respectively, show cell construction, operating conditions, and performance of these cells.

Of the five cells that were operated at 30 psia, all had an initial potential of 0.920 volt. Cells A-074 and A-078 (the two hybrid cells) were the best performers of this group, with lifetimes to 0.780 volt of 2,720 hours and 2,368 hours, respectively. It should be noted, however, that because of excessive leakage at 1,152 hours it was necessary to remove Cell A-074 from the pressure stand and to operate it at 15 psia. Despite this change in operating pressure, the cell operated for a longer period of time than any other cell tested under this contract.

TABLE XXIII

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 1/8 ft^2 CELLS OPERATED AT 200 ASF and 30 psia

								Leakage			
			Separators					Cathode		Anode	
Cell No.	Anode Type	Cathode Type	Cathode	Anode	Electrolyte	Current Collector	Epoxy	Time to First Leak (Hrs)	Average (ml/hr·ft^2)	Time to First Leak (Hrs)	Average (ml/hr·ft^2)
A057	LAB-40(B-II)	LAB-40 (B-II)	S-6	S-1	S-2	Silver Epoxy	UCC		No test		
A067	UCC-T-2	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC		No test		
A070	UCC-T-2	LAB-40 (A-II-1)	S-7	S-1	S-7	Silver Epoxy	UCC		No test		
A074	UCC-T-2	LAB-40 (A-II-1)	S-7	S-1	S-7	Silver Epoxy	UCC	1	4.9	1	0.06
A078	UCC-T-2	LAB-40 (B-II-4)	S-7	S-1	S-7	Silver Epoxy	UCC	70	0.5	1	0.20
A083	LAB-40(143-A)	LAB-40 (143-A)	S-7	S-1	S-7	Silver Epoxy	UCC	1	.6	180	1.00
A089	LAB-40(143-B)	LAB-40 (143-B)	S-7	S-1	S-7	Silver Epoxy	UCC	-	0	70	0.20
A111	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-6	S-1	S-6	Seam Welded Ag	Ring 101-F	1	36.0	1	5.20

TABLE XXIV

OPERATING CONDITIONS OF 1/8 ft^2 CELLS AT 200 ASF AND 30 psia

							Pressure Drop								
							KOH at 125 ml/min inches KOH		H_2 inches H_2O		O_2 inches H_2O		Internal Resistance (m Ω · ft^2)		
Stack	Type	Pressure psia	Voltage Initial	Voltage Final	Life to .780 V (Hours)	Total Time on Test (Hours)	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Failure Mode
A057	101	30			28	28		No Test							Cell construction
A067	101	30		No Test											Excessive leakage
A070	101	30		No Test											Cell construction
A074	101	30 & 15	0.925	0.731	2720	3037	8.00	78.0	0.40	0.10	2.80	3.3	0.33	0.53	Cell construction & both anode and cathode
A078	101	30	0.920	0.540	2368	2442		2.0	0.10	0.20	2.20	>5.0	0.36	0.50	Both anode and cathode.
A083	101	30	0.920	Low	180	285									Test stand equipment
A089	101	30	0.920	0.760*	142	190							0.23	0.23	Test stand equipment
A111	101	30	0.921	0.764	700	752	5.80	5.6	1.80	>2.00	0.40	1.5	0.30	0.30	Anode, Excessive leakage

---

* 100 ASF

TABLE XXV

PERFORMANCE OF 1/8 ft^2 CELLS AT 200 ASF and 30 psia

Cell No.	Activation Date 1967	Time of Pressurization (Hrs)	Cell Voltage Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	1100 Hrs	1300 Hrs	1500 Hrs	1700 Hrs	1900 Hrs	2100 Hrs
A-057	4/05/67	28												
A-067	4/19/67													
A-070														
A-074*	4/19/67	29	.920	.920	.910	.900	.890	.890	.885	.830*	.840	.840	.830	.800
A-078	4/21/67	76	.920	.920	.905	.910	.905	.895	.885	.880	.895	.880	.865	.865
A-083	6/07/67	50	.920	.920										
A-089	6/23/67	74	.920	.920										
A-111	12/8/67	73	.921	.915	.894	.846	.780							

Stack No.	2300 Hrs	2500 Hrs	2700 Hrs	End or .780V	Lifetime (Hrs) to .780 V	8 mV/200 hrs	40 mV/1000	Task I Milestones 1	2	Cause of Failure
A-057										Failed mechanically on pressurization.
A-067										Excessive KOH leakage through electrodes
A-070										Construction difficulties
A-074*	.800	.795	.786		2720	312 at 30 psia 2100 at 15 psia	1052 at 30 psia 2450 at 15 psia	Yes	Yes	Anode and Cathode poor. KOH blockage.
A-078	.845				2368	1000	2210	Yes	Yes	Cathode, & Anode Poor
A-083				.900	180			Yes	No	System Problems.
A-089					142	120		Yes	No	KOH leakage System problems.
A-111					700	260		Yes	No	Weak Anode. Electrodes delaminated.

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hours

* At 1152 hours, stack was removed from 30 psia test stand and put on test 15 psia because of excessive leakage at the higher pressure. 55 mV decrease in voltage because of depressurizing

The internal resistance of both these long-lived cells increased by ~50 per cent during test. Electrolyte leakage occurred through the electrodes of all cells except through the cathode of A-089. This cell, however, was on test only 142 hours when test stand problems led to its early failure. Two cells in this five-cell group failed because of test stand equipment; one because of a poor cathode, one because of a poor anode and excessive leakage, and one because of disintegrating electrolyte separator which caused a KOH flow blockage.

Figures 20 and 21 show the initial and the 500-hour IRI polarization curves for the cells operating at 30 psia. No polarization data were obtained on Cell A-083. The rapid degradation of Cell A-111 is very evident. It is not feasible to generate a curve envelope for these pressure cells because of the limited number tested.

Task I, Milestone 1, was met by all five of the cells which were operated at 30 psia, but Milestone 2 was met by only two cells (i. e., A-074 and A-078).

3.2.4.3 Tests under a Simulated System Load Cycle

Four hybrid stacks (operated at 15 psia) and one LAB-40 (operated at 30 psia) were tested under the load described in Table XXVI.

TABLE XXVI

SIMULATED NASA LOAD CYCLE

Time From	Time To	Current (amps)	Current Density (ASF)	Time (Hrs)
0830 hrs	0930 hrs	7.5	63	1.00
0930	1015	14.8	124	0.75
1015	1030	24.0	200	0.25
1030	1130	14.8	124	1.00
1130	1330	7.5	63	2.00
1330	1430	14.8	124	1.00
1430	1630	24.0	200	2.00
1630	0830	14.8	124	16.00

The actual load cycle used in the tests differs somewhat in detail from the NASA load cycle specified for the 5-kW system design. (NASA CR-72305.) It has the same percentage of the operating time under the same loads as the

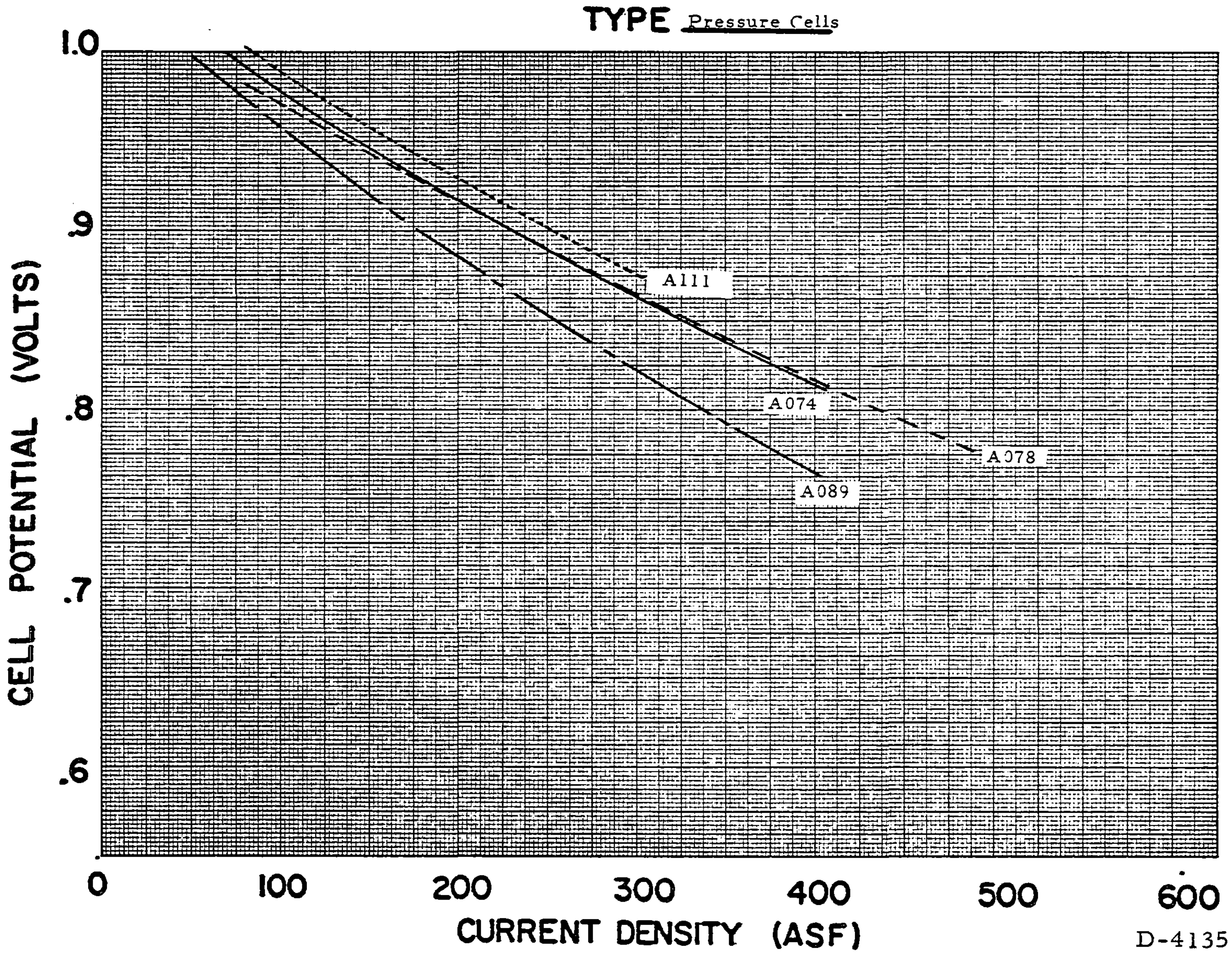

Fig. 20 - Initial IRI Polarization Curves of 1/8 ft^2, 30 psia Cells

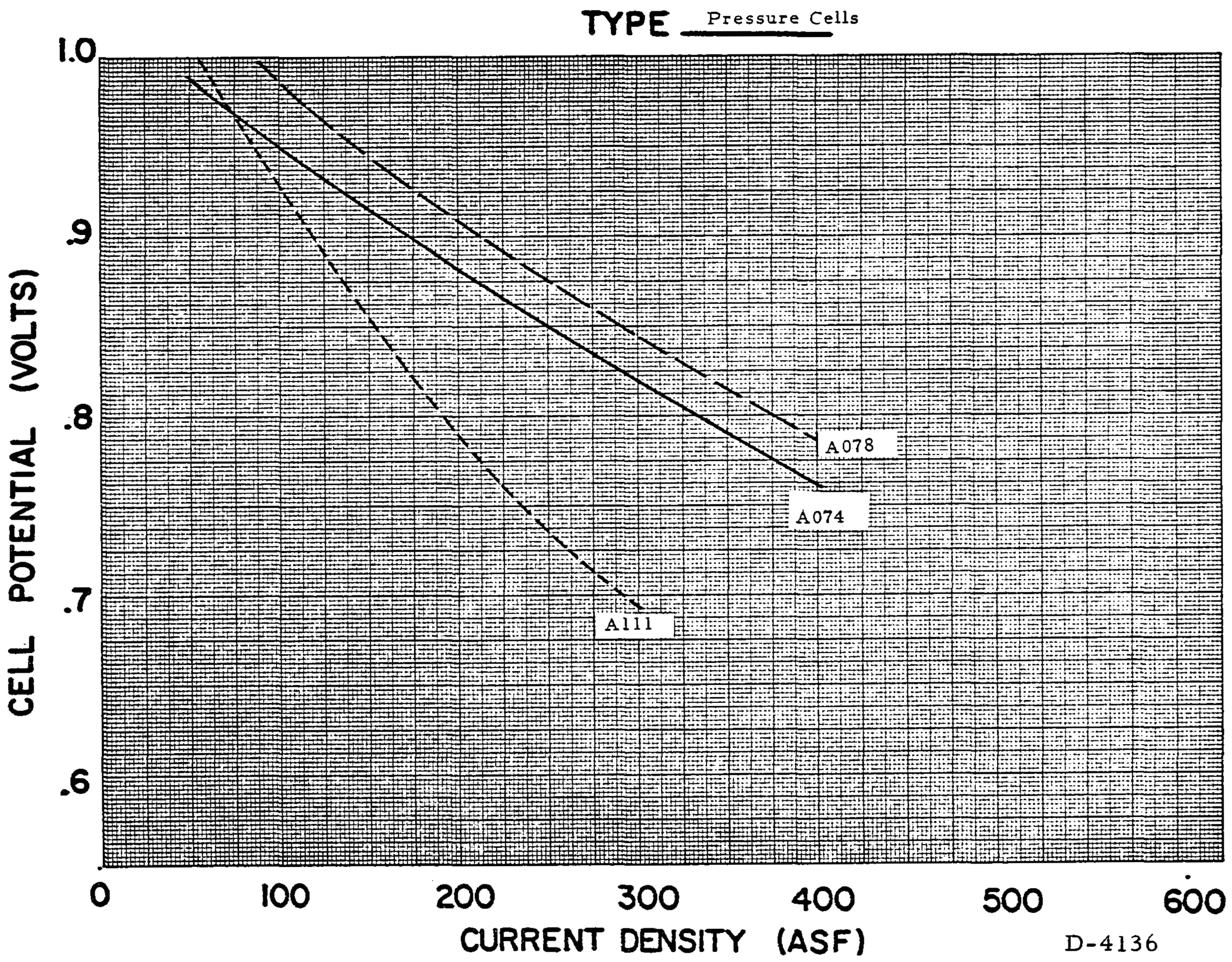

Fig. 21 - After 500 Hours IRI Polarization Curves of 1/8 ft^2, 30 psia Cells

system specification requires, but the exact timing was rearranged for convenience of single-shift operation of the test stands. This cycle was followed for five days each week, with operation on the weekend at 124 ASF. Tables XXVII, XXIII, and XXIX show construction features, operating conditions, and performance of these cells. One cell test (A-115) was conducted at 30 psia as opposed to 15 psia for the other load cycle tests. This pressure test was the only cell in this group which met Task I, Milestone I. Milestone 2 was met by all except A-069 which suffered internal damage caused by a pump failure at 356 hours.

All but cell A-069 behaved quite uniformly with life times to 0.780 v of ~2000 hours: initial voltage of ~0.88 v at 15 psia; voltage degradation during major portions of test less than 8 mv per 200 hours; high leakage through both electrodes (except anode of pressure cell), and internal resistances of ~0.3 $m\Omega \cdot ft^2$ throughout life test. Cell A-090 maintained this resistance level for 1700 hours and then increased drastically. Further indications of acceptable cell performance and low voltage degradation can be seen in Fig. 22. This figure shows that the initial polarization curves of all cells (except A-084) fall within the envelope of the initial, normal polarization curves. Cell A-084 deviates only slightly from the envelope at high current densities.

The T-2 anodes in the cyclic-operated cells appeared to leak much more than those tested at 200 ASF continuously. Leakage through all the cathodes was high and sporadic.

3.2.4.4 1/8-ft^2 Cells at 100 and 300 ASF

The majority of cells under this contract were tested at a current density of 200 ASF. Except for the NASA duty cycle tests described in the preceding section, only five other tests were intentionally conducted at current densities different from the normal 200 ASF. These tests included three at 100 ASF (one LAB-40, one hybrid, and one with a ChemCell cathode and a Union Carbide T-2 anode) and two at 300 ASF (one LAB-40, and one hybrid). Construction features, operating conditions and cell performance can be found in Tables XXX, XXXI, and XXXII.

The number of cells running at these various current densities was too limited to permit any conclusions to be drawn about electrode quality, except in the case of the ChemCell cathode, which was clearly unacceptable. Its initial

TABLE XXVII

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 1/8 ft^2 CELLS OPERATING ON SIMULATED NASA LOAD CYCLE

Cell No.	Anode Type	Cathode Type	Separators			Current Collector	Epoxy	Leakage to Cathode		Leakage to Anode	
			Cathode	Anode	Electrolyte			Time to First Leak (Hrs)	Average (ml/hr · ft^2)	Time to First Leak (Hrs)	Average (ml/hr · ft^2)
A069	UCC-T-2	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC	48	1.0	1	18.0
A073	UCC-T-2	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC	24	2.1	1	1.0
A084	UCC-T-2	LAB-40 (143-A)	S-7	S-1	S-7	Silver Epoxy	UCC	90	2.4	1	4.8
A090	UCC-T-2	LAB-40 (143-B)	S-7	S-1	S-7	Silver Epoxy	UCC	330	7.5	90	2.9
A115	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Rezolin	470	14.8	800	.1

TABLE XXVIII

OPERATING CONDITIONS OF 1/8 ft^2 CELLS ON SIMULATED NASA LOAD CYCLES

Cell No.	Type	Pressure psia	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop KOH at 125 ml/min inches KOH		Pressure Drop H_2 inches H_2O		Pressure Drop O_2 inches H_2O		Internal Resistance (m Ω · ft^2)		Failure Mode
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A069	101	15	.880	<.100	356	356							.30	.33	Test stand equipment
A073	101	15	.880	.650*	2500	2600							.30	.35	Both anode & cathode
A084	101	15	.880	.832	2200	2200		8.4					.30	.35	Excessive leakage
A090	101	15	.885	.515*	1990	2042							.30	1.00	Anode High int. res.
A115	101	30	.920	.786	1840	1846	5.50		.28	.40	.25	.30	.30	.30	Anode

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* at 100 ASF

TABLE XXIX

PERFORMANCE SUMMARY OF 1/8 ft^2 CELLS OPERATING ON SIMULATED NASA LOAD CYCLES

Cell No.	Pressure psia	Current Density ASF	Activation Date	Peak	Cell Voltage(volts) 100 Hours	300 Hours	500 Hours	700 Hours	900 Hours	1100 Hours	1300 Hours	1500 Hours	1700 Hours
A069	15	124 200	4/25/67	.920 .880	.920 .880	.920 .870	---- ----	---- ----	---- ----				
A073	15	124 200	4/25/67	.925 .880	.925 .880	.925 .880	.920 .880	.925 .880	.920 .870	.905 .850	.900 .840	.920 .860	.910 .860
A084	15	124 200	5/4/67	.940 .880	.925 .875	.920 .860	.920 .865	.915 .860	.915 .855	.915 .850	.910 .845	.905 .845	.905 .840
A090	15	124 200	5/4/67	.930 .885	.930 .885	.925 .880	.920 .875	.920 .875	.915 .865	.905 .855	.905 .855	.905 .855	.900 .840
A115	30	124 200	1/4/68	.965 .920	.960 .920	.956 .913	.953 .910	.947 .908	.946 .903	.943 .892	.922 .868	.891 .839	.849 .785

Stack No.	1900 Hours	2100 Hours	2300 Hours	End or .780V	Lifetime (Hours) to .780V	8 mV/200hrs	40 mV/1000hrs	Task I Milestones 1	2	Cause of Failure
A069				.920	356			No	No	Internal damage caused by pump failure
A073	.900 .835	.900 .830	.900 .825	.880 .780	2500	1800	1960	No	Yes	Anode & cathode both poor
A084	.915 .850	.905 .840	---- ----	.905 .832	2200	2200	2200	No	Yes	Excessive KOH leakage thru anode
A090	.895 .825	---- ----			1990	1990	1990	No	Yes	Cell shut down due to low electrolyte level
A115	---- ----			.850 .786	1840	1320	1320	Yes	Yes	High cathode leakage. Poor anode

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

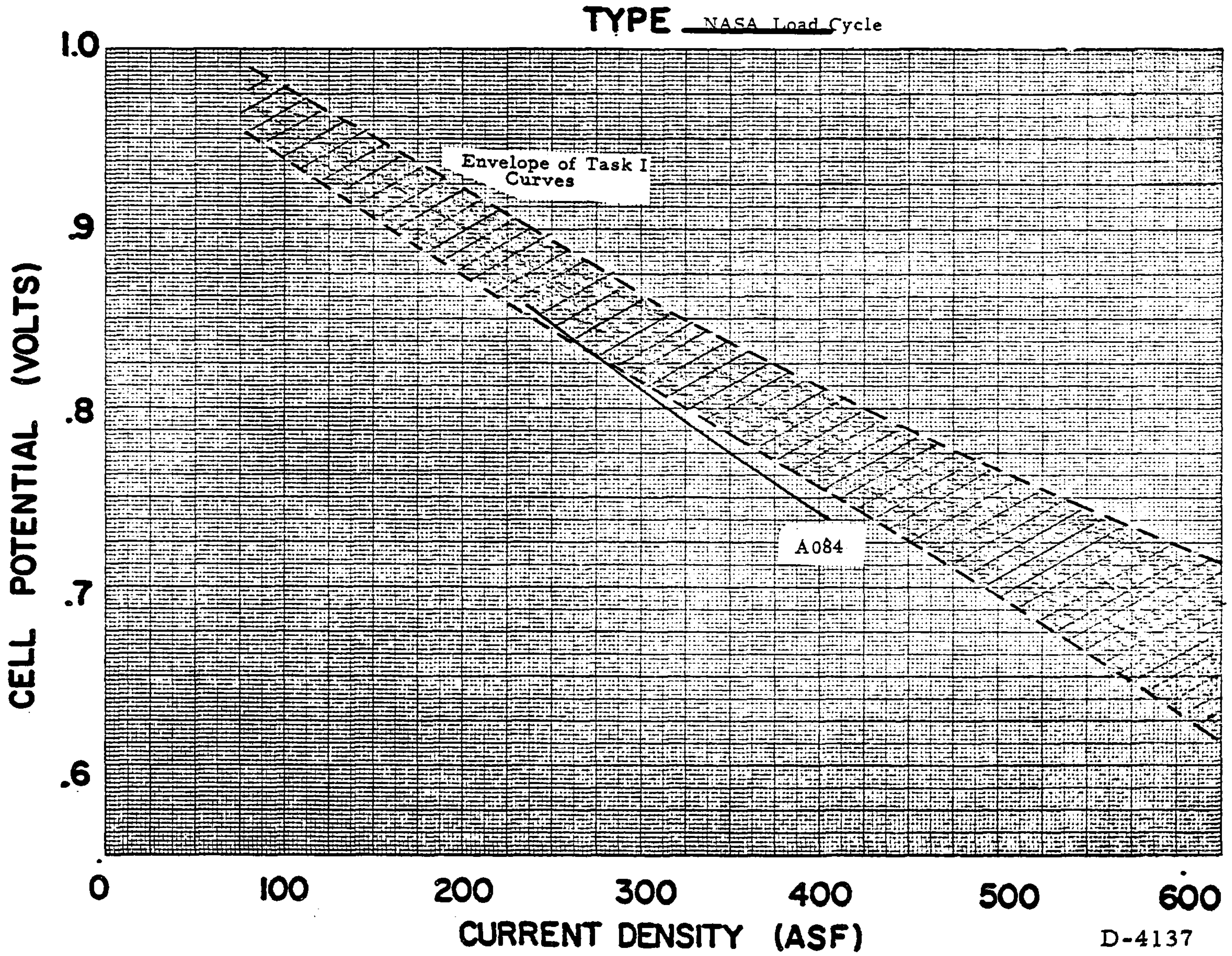

Fig. 22 - Envelope of Initial IRI Polarization Curves; NASA Load Cycle.

TABLE XXX

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 1/8 ft^2 CELLS OPERATING AT 300 ASF

									Leakage			
									Cathode		Anode	
				Separators								
Cell No.	Current Density ASF	Anode Type	Cathode Type	Cathode	Anode	Electrolyte	Current Collector	Epoxy	Time to First Leak (Hrs)	Average (ml/hr · ft^2)	Time to First Leak (Hrs)	Average (ml/hr · ft^2)
A018	100	LAB-40nd*	LAB-40nd	S-1	S-1	S-2	Silver Epoxy	UCC	1	40	1	2.0
A035	100	UCC-T-2	ChemCell H 9454N	S-1	S-1	S-2	Silver Epoxy	UCC	1	73	1	2.8
A061	100	UCC-T-2	LAB-40(A-II)	S-6	S-1	S-2	Silver Epoxy	UCC	1	23	1	.1
A053	300	LAB-40(A-II)	LAB-40(A-II)	S-6	S-1	S-2	Silver Epoxy	UCC	170	0.4	1	2.0
A072	300	UCC-T-2	LAB-40(A-II-1)	S-7	S-1	S-7	Silver Epoxy	UCC	-	0	1	4.0

*nd No designation beyond LAB-40 is available for the earliest lots of American Cyanamid electrodes

TABLE XXXI

OPERATING CONDITIONS OF 1/8 ft^2 CELLS AT 100 and 300 ASF

Cell No.	Type	Current Density	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop KOH at 125 ml/min inches KOH		Pressure Drop H_2 inches H_2O		Pressure Drop O_2 inches H_2O		Internal Resistance (m Ω · ft^2)		Failure Mode
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A018	101	100	.950	<.500	770	828			.05	.25	.10	1.30	.30	.35	Excessive leakage, Test stand equipment
A035	101	100	.910	.730	905	929	1.00		.05	.15	.25	1.70	.43	.40	Excessive leakage, Test stand equipment
A061	101	100	.970	.765	1160	1174	2.20	2.4	.10	.05	.20	>9.00	.35	.33	Cell construction, Test stand equipment
A053	101	300	.860	.680	406	602	2.40		.20	.30	.10	1.00	.27	.32	Test stand equipment
A072	101	300	.870	.670*	220	284			.10	.40	.50	2.00	.30		No diagnostic tests

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* 200 ASF

TABLE XXXII

PERFORMANCE SUMMARY OF 1/8 ft^2 CELLS OPERATED AT 100 and 300 ASF

Cell No.	Current Density ASF	Activation Date	Cell Voltage Peak	100 Hours	300 Hours	500 Hours	700 Hours	900 Hours	1100 Hours	End or .780V	Lifetime to .780V
A018	100	11-30-66	.950	.940	.950	.948	.890				770
A035	100	1-13-67	.910	.910	.865	.831	.810	.780			905
A061	100	3-2-67	.970	.968	.962	.960	.960	.955	.945		1160
A053	300	3-10-67	.860	.860	.815						406
A072	300	4-21-67	.870	.825							220

Cell No.	Lifetime 8m V/200	40m V/1000	Task I Milestones 1	2	Cause of Failure
A018	528	-	Yes	Yes	Several Accidental Shut Downs
A035	230	-	No	No	Excessive Leakage, Accidental Shut Down
A061	1160	1160	Yes	No	Cell Overheated and Melted Separator
A053	144	-	Yes	No	KOH Pump Failure
A072	168	-	No	No	No Diagnostic Test

1 Initial voltage equal to or exceeding 0.900 v at 200 ASF

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

voltage level was low, degradation was rapid, internal resistance was high and cathode leakage was exorbitant. The cathode leakage rate of all three of the cells operating at 100 ASF was very high. Voltage degradation of the cells operating at 300 ASF was rapid.

The poor performance of the Chemcell and the cells operating at 300 ASF is very evident from Figs. 23 and 24. All cell failures (except A-072) were in some manner related to test stand equipment. Diagnostic tests were not conducted, so it is not possible to determine which electrode was more effected by the equipment failure.

Of the cells operating at 100 ASF Task I, Milestone 1, were met by the LAB-40 and the hybrid cells while Milestone 2 was met by only the LAB-40 cell. Task I, Milestone 1, was met by the LAB-40 cell operating at 300 ASF. Neither of the 300 ASF cells met Milestone 2.

3. 2. 4. 5 Small Cells Using T-3N Cathodes

Four cells using Union Carbide type T-3N cathodes and T-2 anodes were tested during the program. All four cells fell below acceptable NASA standards (peak voltage: 0. 840 v, max. lifetime: 195 hours to 0. 780 v). Tables XXXIII, XXXIV, and XXXV show construction features, operating conditions and performance of these cells. Initial low voltage was not unexpected as these cells used a new type of experimental electrode. Diagnostic tests at termination showed that two cells had poor cathodes, one a poor anode and one with both electrodes poor. Increasing internal resistance of the cells was also noted as the tests progressed. This increase in internal resistance was probably due to one of the following reasons:

1. The active materials separating from the Ni facing, thus causing poor electrical contact;
2. Oxidation of the porous nickel or;
3. Loss of contact of the welded tabs.

Electrolyte leakage through the electrodes was low, but the cells were not on test a sufficient length of time to form any definite conclusions regarding the leakage problem.

A break-in period was required for the T-3N cathodes, so a special

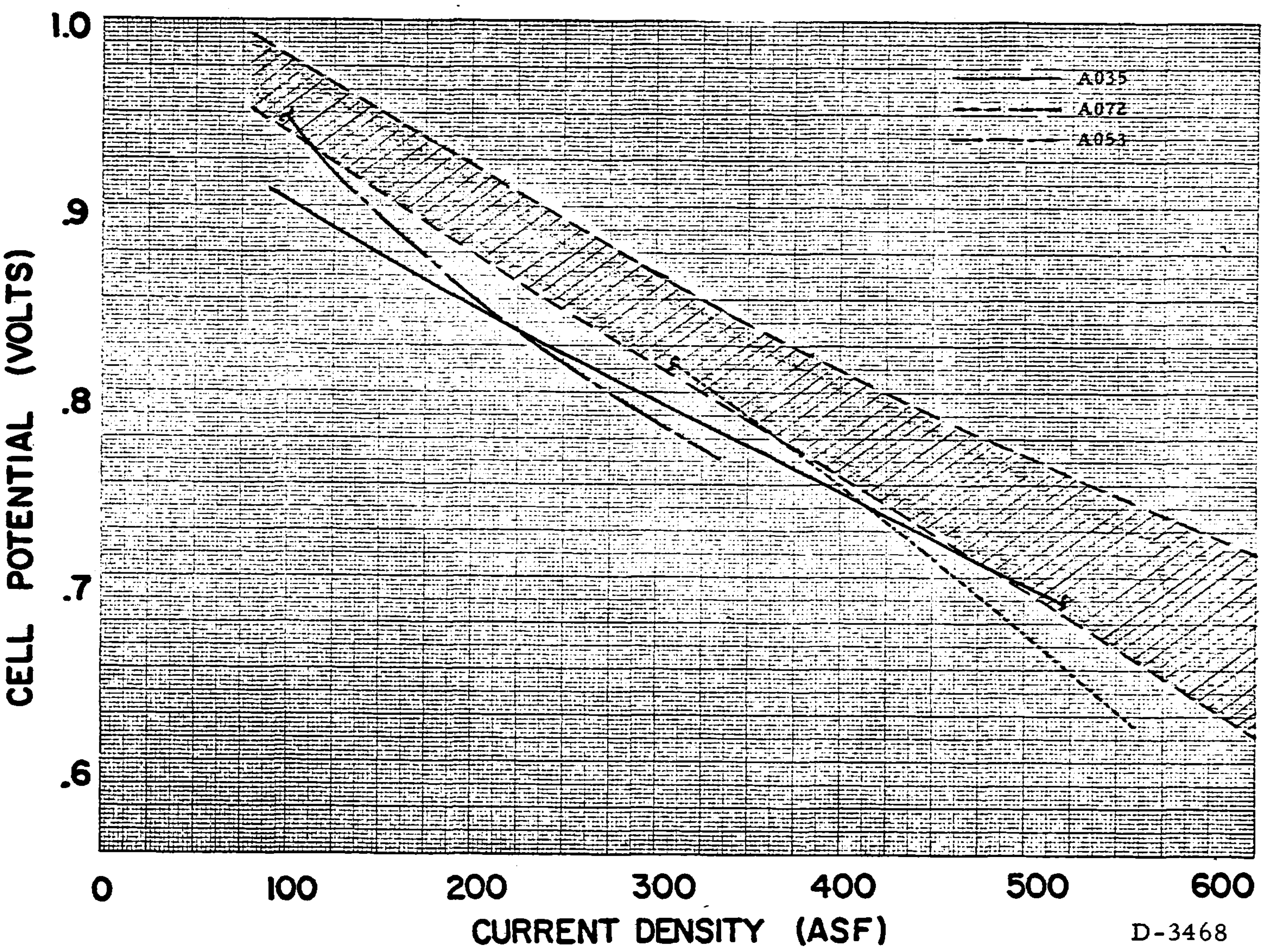

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Fig. 23 - Envelope of Initial IRI Polarization Curves
1/8 ft^2 Cells at 100 and 300 ASF.

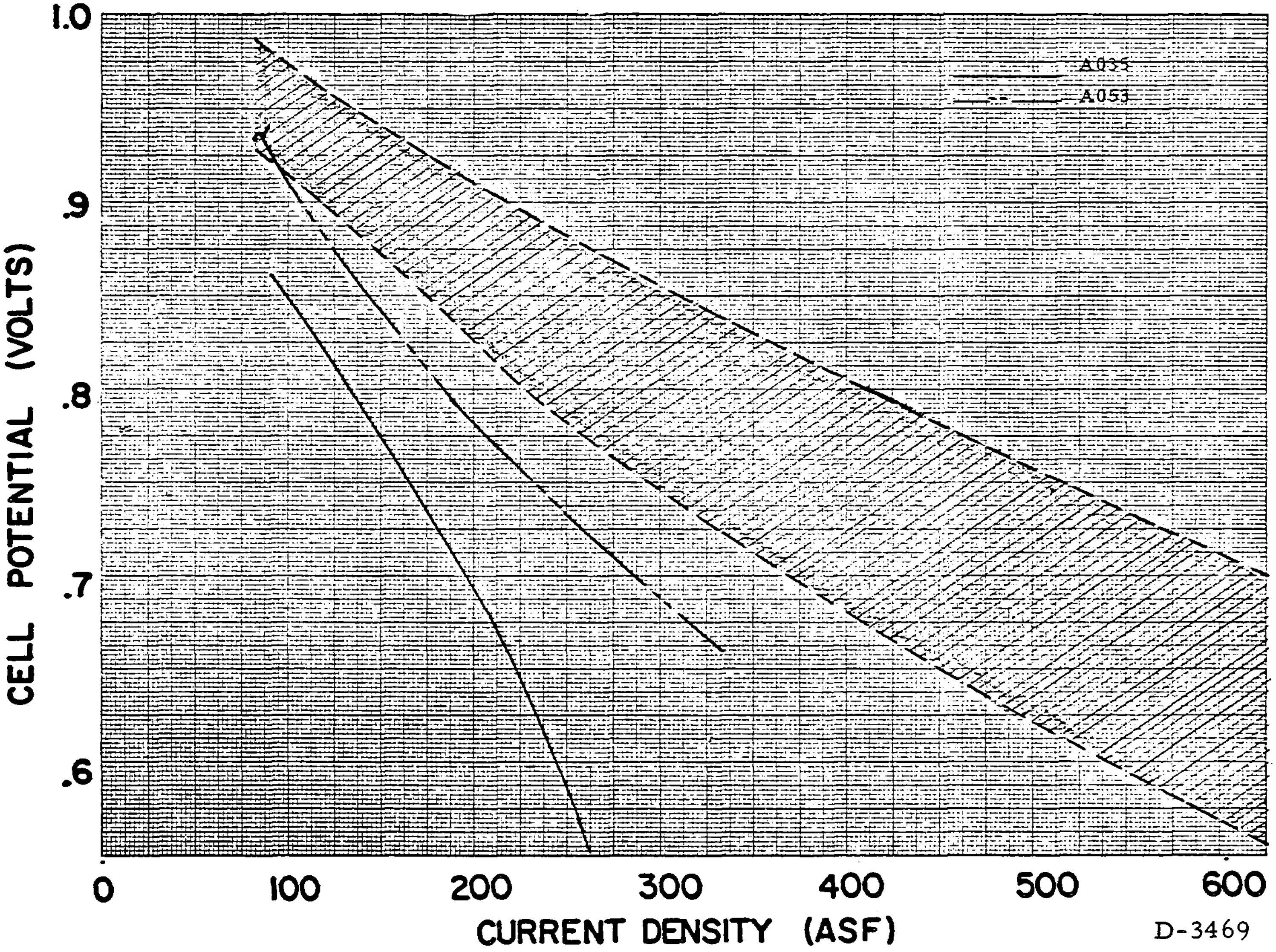

Fig. 24 - Envelope of IRI Polarization Curves~500 Hours After Initial
1/8 ft^2 Cells at 100 and 300 ASF

TABLE XXXIII

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 1/8 ft^2 CELLS WITH T-3N CATHODES

Cell No.	Anode Type	Cathode Type	Separators			Current Collector	Epoxy	Leakage			
								Cathode		Anode	
			Cathode	Anode	Electrolyte			Time to First Leak (Hrs)	Average (ml/hr· ft^2)	Time to First Leak (Hrs)	Average (ml/hr· ft^2)
A-095	UCC-T-2	UCC-T-3N	S-7	S-1	S-7	Silver Epoxy	UCC	1	0.1	-	0
A-096	UCC-T-2	UCC-T-3N	S-7	S-1	S-7	Silver Epoxy	UCC	-	0	-	0
A-097	UCC-T-2	UCC-T-3N	S-7	S-1	S-7	Silver Epoxy	UCC	-	0	1	0.26
A-098	UCC-T-2	UCC-T-3N	S-7	S-1	S-7	Silver Epoxy	UCC	175	0.02	175	0.02

TABLE XXXIV

OPERATING CONDITIONS AT 200 ASF FOR 1/8 ft^2 CELLS WITH T-3N CATHODES

Cell No.	Type	Pressure psia	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop						Internal Resistance (m Ω · ft^2)		Failure Mode
							KOH at 125 ml/min inches KOH		H_2 inches H_2O		O_2				
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A095*	101	15	.895	.860	240	240							.35	.45	Cathode, High int. resistance
A096	101	15	.835	.735	130	278							.33	.55	Cathode. High int. resistance
A097	101	15	.825	.725	64	140		3.5	.40	.20	.15	.20	.37	.60	Anode, High internal resistance
A098	101	15	.840	.780	195	290			.20	.70	.30	.90	.55	.58	Both anode and cathode, High internal resistance

---

* 100 ASF

TABLE XXXV

PERFORMANCE SUMMARY OF 1/8 ft^2 CELLS WITH T-3N CATHODES

Cell No.	Activation Date	Peak	Cell Voltage 100 Hrs.	End or .780V	Lifetime (Hours) to .780V	8mV/200 Hrs.	40mV/1000 Hrs.	Task I Milestone 1	2	Cause of Failure
A095*	6-15-67	.920	.920	.860	-	-	-	No	No	Poor Cathode, High int. res.
A096	6-28-67	.835	.810	-	130	130	-	No	No	Poor Cathode, High int. res.
A097	7-25-67	.810	-	-	64	64	-	No	No	Poor Cathode, High int. res.
A098	6-30-67	.840	.800	-	195	150	-	No	No	Electrodes Poor High int. res.

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

* 100 ASF

start-up procedure was adopted. On start-up, the entire group was placed on open circuit for 12 hours followed by another 24 hours at whatever current density all could easily sustain, and then the cells loaded to 200 ASF at an O_2 inlet pressure of 10 to 15" w.c. for 48 hours. O_2-inlet pressure was then periodically increased in 24 or 48 hour increments until 65" w.c. of O_2-inlet pressure was attained. Cells were then operated continuously at the oxygen-inlet pressure giving the best performance.

After the conclusion of these four tests, NASA directed that the development of the T-3N cathode be changed from the Ni substrate to use only Ag as a plaque material. This change was reflected in the work previously described under "High Platinum Loaded Cathode." However, development was not carried to the point where further 1/8 ft^2 cell tests were warranted.

As can be seen from Fig. 25, polarization data on these cells(both IR-free and IR-included) were well below the average of other cells tested under the contract.

3.2.4.6 Flight-Size Cells

Five of the larger flight-size units (two hybrid and three LAB-40) were tested under this contract. Flight-size units utilize four pairs of 0.325 ft^2 electrodes connected in parallel giving a total active area of 1.3 ft^2 per stack. Three of the tests were conducted at 30 psia and two at 15 psia. The three pressure cells all had initial voltage levels of ~0.900 v and two had degradation rates of less than 20 mv during the first 500 hours. Figures 26 and 27 show the IR-included polarization curves initially, and after 500 hours on test for all "Flight-Size" Cells. The initial polarization data on Cells A-201 and A-203 were not taken until cells had been running for over 330 hours, so this could explain why the initial polarization level of these cells is lower than that of A-205.

The performance of this entire group was uniform with cells maintaining a fairly stable voltage level followed by a steep decline. Construction, operating conditions, and performance of these cells can be seen in Tables XXXVI, XXXVII, and XXXVIII. An oxygen circulation problem as can be seen from the excessively high final oxygen pressure drop data (Table XXXVII), occurred in each cell tested in this group. This problem was probably caused

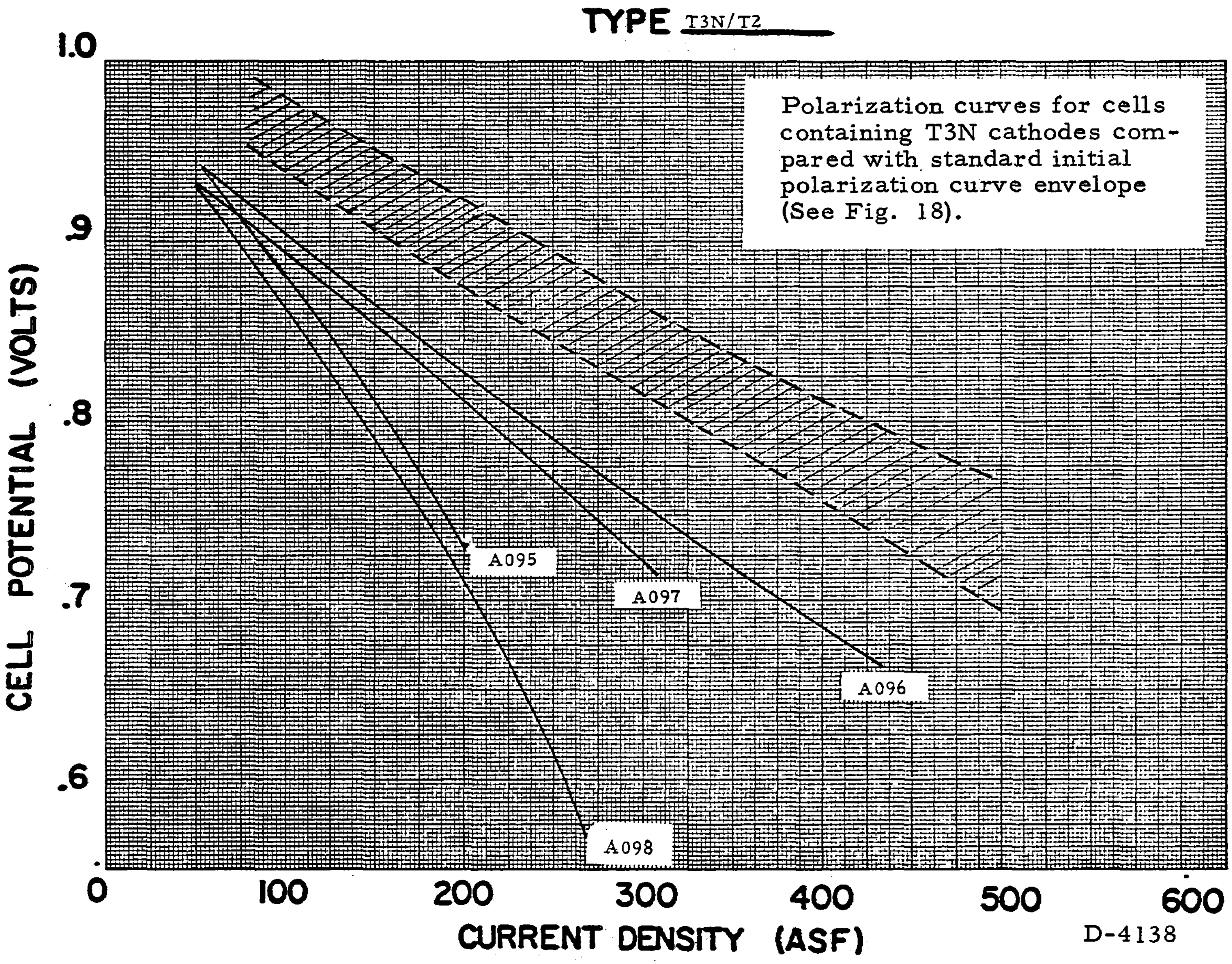

Fig. 25 - Envelope of Initial IRI Polarization Curves for T3N Cathodes.

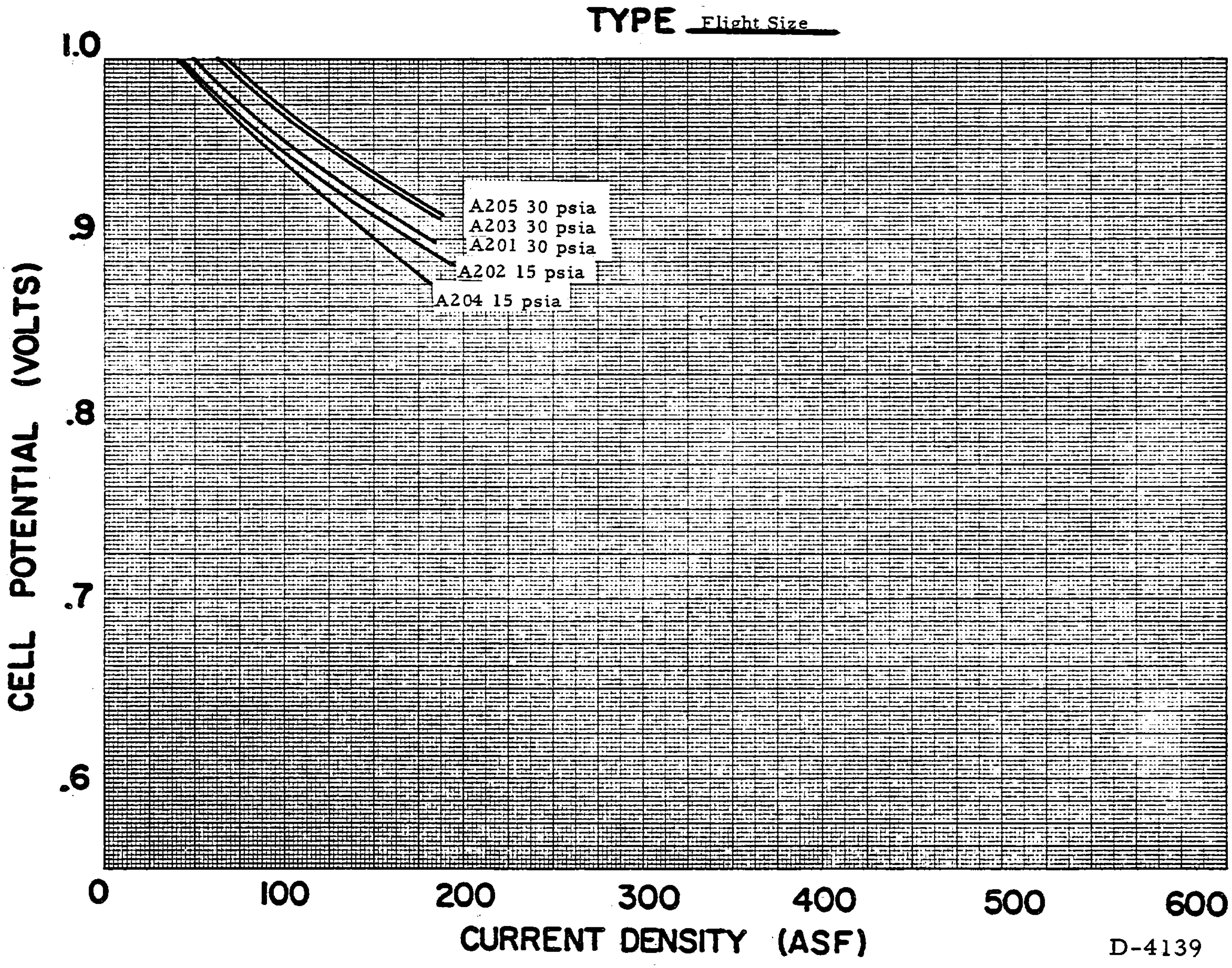

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Fig. 26 - Initial IRI Polarization Curves for All Flight-Size Cells

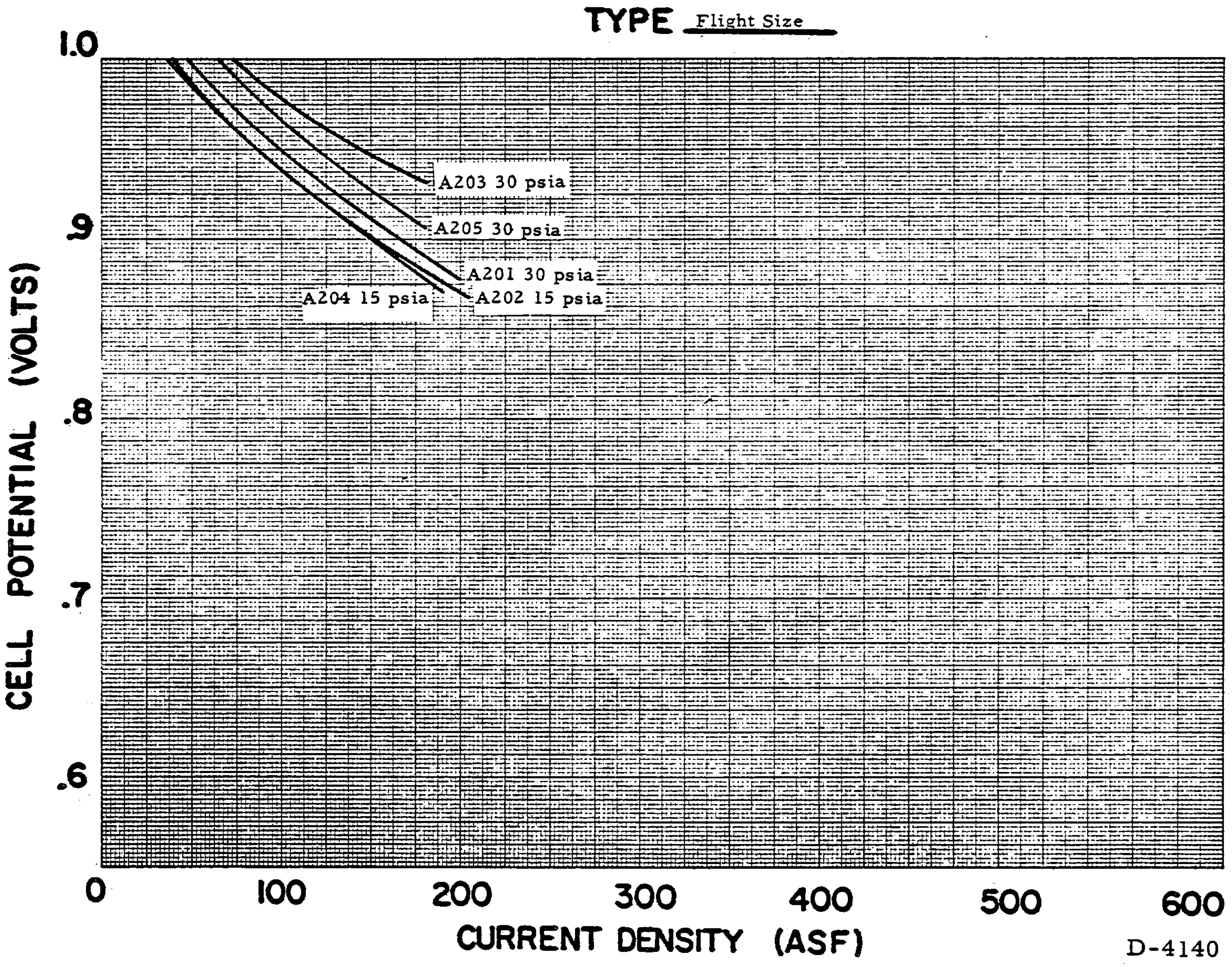

Fig. 27 - IRI Polarization Curves for All Flight-Size Cells After ~500 Hours on Test

TABLE XXXVI.

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR FLIGHT-SIZE CELLS

Cell No.	Anode Type	Cathode Type	Separators			Current Collector	Epoxy	Leakage			
								Cathode		Anode	
			Cathode	Anode	Electrolyte			Time to First Leak (Hrs)	Average (ml/hr · ft²)	Time to First Leak (Hrs)	Average (ml/hr · ft²)
A201	LAB-40 (B-II)	LAB-40 (B-II)	S-7	S-6	S-7	Silver Epoxy	UCC	325	2.5	470	.14
A202	UCC-T-2	LAB-40 (A-II-2)	S-7	S-6	S-7	Silver Epoxy	UCC	1	.73	690	.18
A203	UCC-T-2	LAB-40 (A-II-2)	S-7	S-6	S-7	Silver Epoxy	UCC	1	.33	1	.07
A204	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-7	S-6	S-7	Silver Epoxy	UCC	1	.07	1	.01
A205	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-7	S-1	S-7	Silver Epoxy	UCC	600	.25	1	.35

TABLE XXXVII.

OPERATING CONDITIONS OF FLIGHT-SIZE CELLS

Cell No.	Type	Pressure psia	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop						Internal Resistance (m Ω · ft²)		Failure Mode
							KOH at 500 ml/min inches KOH		H_2 inches H_2O		O_2				
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A201	404	30	.900	.795	974	974			.80	1.20	1.00	8.4		.55	Cell construction
A202	404		.885	.582	1000	1079	2.80		.70	1.80	1.00	>9.0	.40	.60	Cell construction
A203	404	30	.910	.620	1340	1360	3.30		.62	1.90	.80	5.7	.40	.50	Cell construction
A204	404		.870	.765	825	859	0.75		.65	.65	1.20	2.3	.40	.40	Cell construction
A205	404	30	.900	<.400	1043	1132		3.4	.60	1.50	1.50	6.6	.40	.50	Cell construction

TABLE XXXVIII

PERFORMANCE SUMMARY OF FLIGHT-SIZE CELLS

Cell No.	Pressure psia	Time of Pressurization Hrs	Starting Date	Voltage at							
				Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	1100 Hrs	1300 Hrs
A-201	30	70	4/11	.900	.900	.900	.885	.880	.825		
A-202	15		6/13	.880	.880	.880	.880	.875	.800		
A-203	30	65	6/06	.910	.910	.905	.915*	.910	.900	.895	.855
A-204	15		6/15	.865	.865	.865	.860	.845			
A-205	30	100	8/16	.900	.900	.892	.878	.866	.895**		

Stack No.	End or .780 V	Lifetime (Hrs.)			Task I Milestones		Cause of Failure
		to .780 V	8 mV/200 hrs	40 mV/1000 hrs	1	2	
A-201		974	910	---	Yes	Yes	O_2 Circulation Problems
A-202		1000	745	---	No	Yes	O_2 Circulation Problems
A-203		1350	1250	1270	Yes	Yes	O_2 Circulation Problems
A-204		820	670	----	No	Yes	O_2 Circulation Problems
A-205		1042	650	1042	Yes	Yes	O_2 Circulation Problems

* Potential rose 0.020 volt after shutdown at 369 hours to make changes in house O_2 supply.

** Potential rose 0.03 volt when changing from house O_2 to bottled O_2 and an additional 0.10 V when changing back to house O_2 supply.

Milestones
1 Initial voltage equal to or exceeding 0.900 v.
2 Voltage degradation equal to or less than 20 mv during first 500 hrs.

by delamination of the cathode backing into the separator space. This argument is supported by the fact that upon disassembly of the cell a definite "quilting" pattern associated with the separator could be seen on the backing of the LAB-40 electrodes.

Electrolyte leakage occurred through all the electrodes, with A-204 displaying the least amount. The internal resistance of all cells, except A-204, increased 25 to 50 percent during life test.

Task I, Milestone 1, was met by the three pressure cells and Milestone 2 was met by all cells.

3.2.4.7 0.325 ft^2 Cells

Three cells (two hybrid and one LAB-40)of the 0.325 ft^2 size were constructed and tested. This special cell size was intended primarily to check certain cell construction features intended for flight-size cells rather than to life test electrodes. These cells use electrodes of the same size as the flight-size cells but only one anode-cathode pair is included instead of the four in the flight-size cells. After only three hours, the one LAB-40 test (A-214) had to be terminated because of excessive electrolyte leakage through a delaminated backing on the anode. Another test (A-215) lasted approximately 330 hours when a weak cathode and high internal resistance caused its removal. The third test (A-212) in this group had low voltage degradation for over 1900 hours and then experienced rapid decay. This cell did, however, have a low initial voltage level. This fact can be seen in Fig. 28 which shows its initial IRI performance to fall outside of the established performance envelope. Its 500 hour performance was within the envelope. Cell A-214 also appears very weak, but this is probably due to excessive leakage causing flooding of the active sites. Cell failure of A-212 was caused by a poor anode and high internal resistance. Construction features, operating conditions and cell performance can be found in Tables XXXIX, XL, and XLI.

Electrolyte leakage through all the electrodes was very high. Pressure adjustments of the inlet gases on Cell A-212 did appear to have some beneficial effects on reducing the leakage rate. The internal resistance had more than doubled at the end of test. Task I, Milestone 1, was not met by any of these cells, while Milestone 2 was met by Cell A-212.

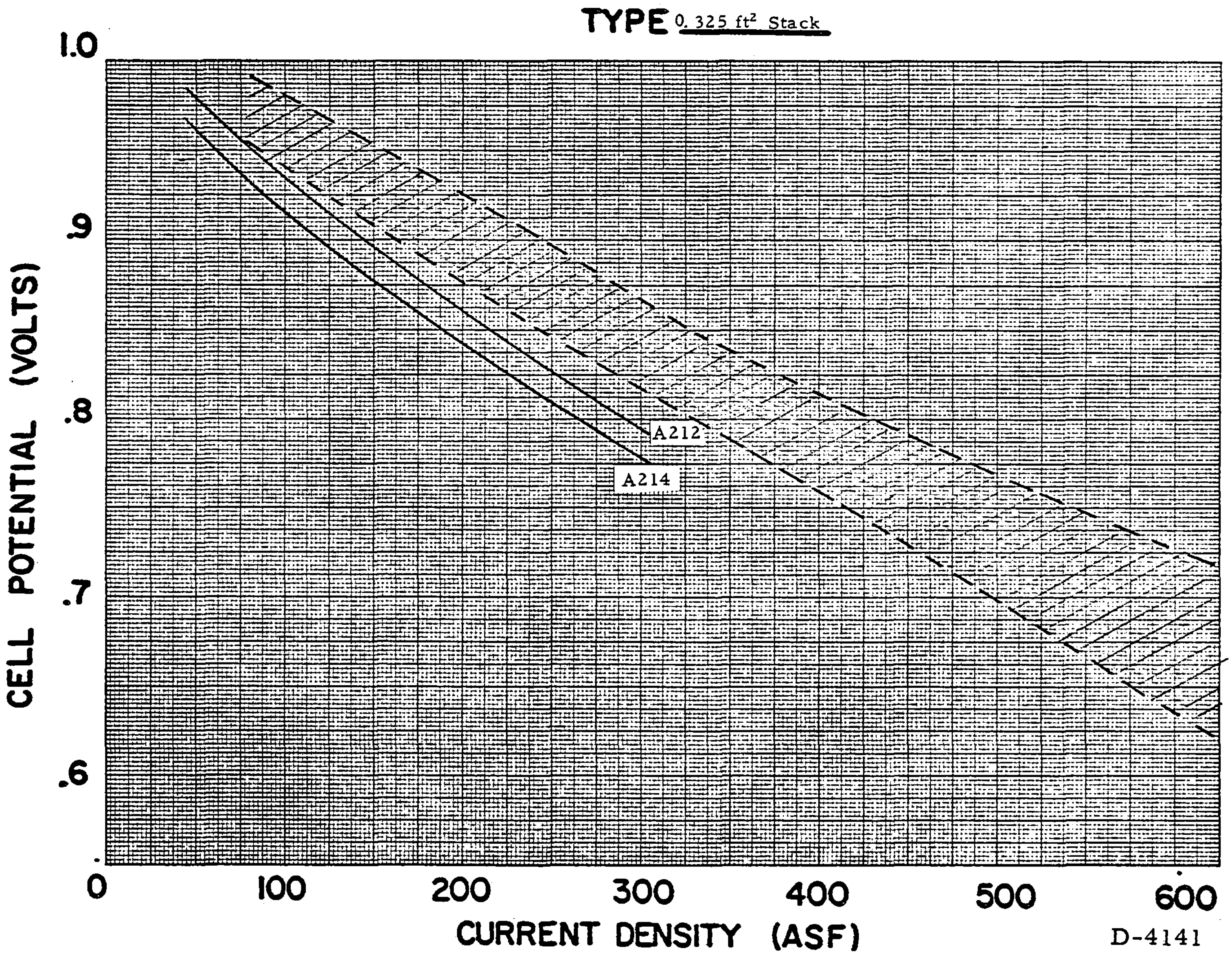

Fig. 28 - Envelope of Initial IRI Polarization Curves for 0.325 ft^2 Cells

TABLE XXXIX

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR 0.325 ft^2 CELLS

Cell No.	Anode Type	Cathode Type	Separators			Current Collector	Epoxy	Leakage			
								Cathode		Anode	
			Cathode	Anode	Electrolyte			Time to First Leak (Hrs)	Average (ml/hr · ft^2)	Time to First Leak (Hrs)	Average (ml/hr · ft^2)
A212	UCC-T-2	LAB-40 (B-II)	S-8	S-8	S-8	Seam Welded Ni	UCC	1	15.0	1	1.8
A214	LAB-40(A-II-2)	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Ring 101-F	1	13.0	1	218.0
A215	UCC-T-2	LAB-40 (A-II-2)	S-9	S-9	S-9	Seam Welded Ag	Ring 101-F	1	7.5	1	4.5

TABLE XL

OPERATING CONDITIONS FOR 0.325 ft^2 CELLS AT 200 ASF

Cell No.	Type	Pressure psia	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop						Internal Resistance (m Ω · ft^2)		Failure Mode
							KOH at 125 ml/min Inches KOH		H_2 Inches H_2O		O_2				
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A212	101	15	.865	.144	1930	1985	1.80	1.5	.50	.40	1.20	1.1	.40	.90	Anode. High int. res.
A214	101	15	.877		3	3	2.00		.75		1.10		.40		Excessive leakage
A215	101	15	.885	.725	330	436	9.20	9.0	.75	.80	1.80	2.3	.35	.75	High internal resistance Cathode

TABLE XLI

PERFORMANCE SUMMARY OF 0.325 ft^2 CELLS

			Cell Voltage									
Cell No.	Activation Date	Peak	100 Hours	300 Hours	500 Hours	700 Hours	900 Hours	1100 Hours	1300 Hours	1500 Hours	1700 Hours	1900 Hours
A212	9-8-67	.865	.860	.860	.850	.848	.842	.855	.850	.840	.822	.799
A214	10-17	.877										
A215	10-26	.885	.875	.822								

	Lifetime (Hours)			Task I Milestones		
Stack No.	to .780V	8m V/200	40m V/1000	1	2	Cause of Failure
A212	1930	1920	1900	No	Yes	Poor Anode. High internal resistance
A214	3	3	-	No	No	Loose Anode Backing Excessive Leakage
A215	330	192	-	No	No	High Internal Resistance Weak Cathode

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

3.2.4.8 Special Cells

In addition to the various types of standard cell tests discussed previously, five special tests were conducted and each is treated separately here. Construction, operating conditions and performance of these cells can be seen in Tables XLII, XLIII, and XLIV. From Fig. 29 and 30 showing the envelopes of initial and 500 hour IRI polarization curves, it is easy to see that certain cells were very weak.

As noted previously, some of the early cells tested under this contract showed the wet-proofed backing was separating from the active portion of the LAB-40 electrodes. Because of the numerous accidental test stand shutdowns which had occurred in the early stages of this program, there was a question as to whether this separation might be due to age or to temperature cycling. A second and more important question arose as to whether this thermal cycling might have a deleterious effect upon cell performance. Cell A-051 was run to evaluate these problem areas.

Typically, this cell was operated for about 16 hours (overnight) at 200 ASF. Each morning it was placed on open circuit, the heater shut off, and water circulated through a Ni cooling coil immersed in the electrolyte tank, so that the unit was brought to between room temperature and 35°C by about noon. Then the cooling water was turned off, the heater turned on again, and the system brought back to 90°C by late afternoon at which time the load was reapplied for the next load cycle.

Initially, the cell showed 0.895 v; but from 60 to 133 hours on load (163 hours on test), the potential was 0.90 v and then it began to drop slowly. After 32 thermal cycles (1,123 hours on test, 944 hours at 200 ASF), the cell still showed 0.825 v. At that time cycling was discontinued and the stack performance was followed at 200 ASF continuous duty. The potential continued to drop at about the same rate as it had done while on the cycling test, falling below 0.78 v after some 1,370 hours under load; 1,527 hours on test. At 1,452 hours (0.73 v) dilute gas tests showed the anode performance had dropped off much more than the cathode. This dilute gas test resulted in rejuvenation of the cell to 0.88 v. However, this improvement did not last, and at 1,478 hours (0,80 v), the stack was removed for washing and drying. Upon return to test, it showed only 0.69 v, so the load was reduced to 100 ASF, giving 0.855 v.

TABLE XLII

CONSTRUCTION FEATURES AND LEAKAGE DATA FOR "SPECIAL CELLS"

Cell No.	Anode Type	Cathode Type	Separators			Current Collector	Epoxy	Leakage			
			Cathode	Anode	Electrolyte			Cathode		Anode	
								Time to First Leak (Hrs)	Average (ml/hr · ft²)	Time to First Leak (Hrs)	Average (ml/hr · ft²)
A051	LAB-40(A-II)	LAB-40 (A-II)	S-6	S-6	S-2	Silver Epoxy	UCC	400	3.5	1	.03
A099	UCC-T-2	LAB-40 (B-II-9)	S-7	S-1	S-7	Silver Epoxy	UCC		.004	403	.02
A122	LAB-40(C-II)	LAB-40 (C-II)	**S-6**	**S-6**	S-6	Seam Weld Ag	Rezolin	500	0	525	.02
Gulton	UCC-T-2	LAB-40 (B-II-4)			7(S-6)	-	Gulton Elastomer	1	1.0	1	.157

TABLE XLIII

OPERATING CONDITIONS OF SPECIAL CELLS

Cell No.	Type	Pressure (psia)	Voltage		Life to .780 V (Hours)	Total Time on Test (Hours)	Pressure Drop						Internal Resistance (m Ω · ft²)		Failure Mode
							KOH at 125 ml/min (Inches KOH)		H₂ (Inches H₂O)		O₂				
			Initial	Final			Initial	Final	Initial	Final	Initial	Final	Initial	Final	
A051	101	15	.900	.690	1370	1522	0.30	2.1	.40	2.00	.35	6.90	.31	.29	Anode
A099	101	15	.900	.591	668	842	4.80	17.6	.10	.40	.70	.60	.30	.50	Cell construction anode
A122	101	15	.861	.720	180	1392	2.50	5.8	.05	.60	.15	1.10	.33	.43	Anode
Gulton	101	15	.813	.653	280	915	12.50						.90	1.30	Cathode

TABLE XLIV

PERFORMANCE SUMMARY OF SPECIAL CELLS

				Cell Voltage							Lifetime (Hours)			Task I		
Cell No.	Type of Test	Activation Date	Peak	100 Hrs	300 Hrs	500 Hrs	700 Hrs	900 Hrs	1100 Hrs	1300 Hrs	to .780V	8mV/200	40mV/1000	Milestones 1	2	Cause of Failure
A051	Thermal Cycle	2-16-67	.900	.900	.879	.878	.865	.850	.835	.815	1370	264		Yes	No	Poor Anode
A099	Weekly Dilute Gas	8-1-67	.900	.899	.880	.861					668	264		Yes	No	Poor Anode
A122	Horizontal Test	2-27-68	.861	.820							180	160		No	No	Poor Anode
Gulton*	Leakage Test 2 1/8" Dia.Cell @122 ASF	2-16-68	.813	.800							280	190		No	No	Poor Cathode, leakage controlled by matching gas & electrolyte inlet pressures

1 Initial voltage equal to or exceeding .900V

2 Voltage degradation equal to or less than 20 mV during first 500 hrs

* Gulton - Stack potential reversed at 221 hrs. Stack washed, dried and returned to test. Time runs continuously

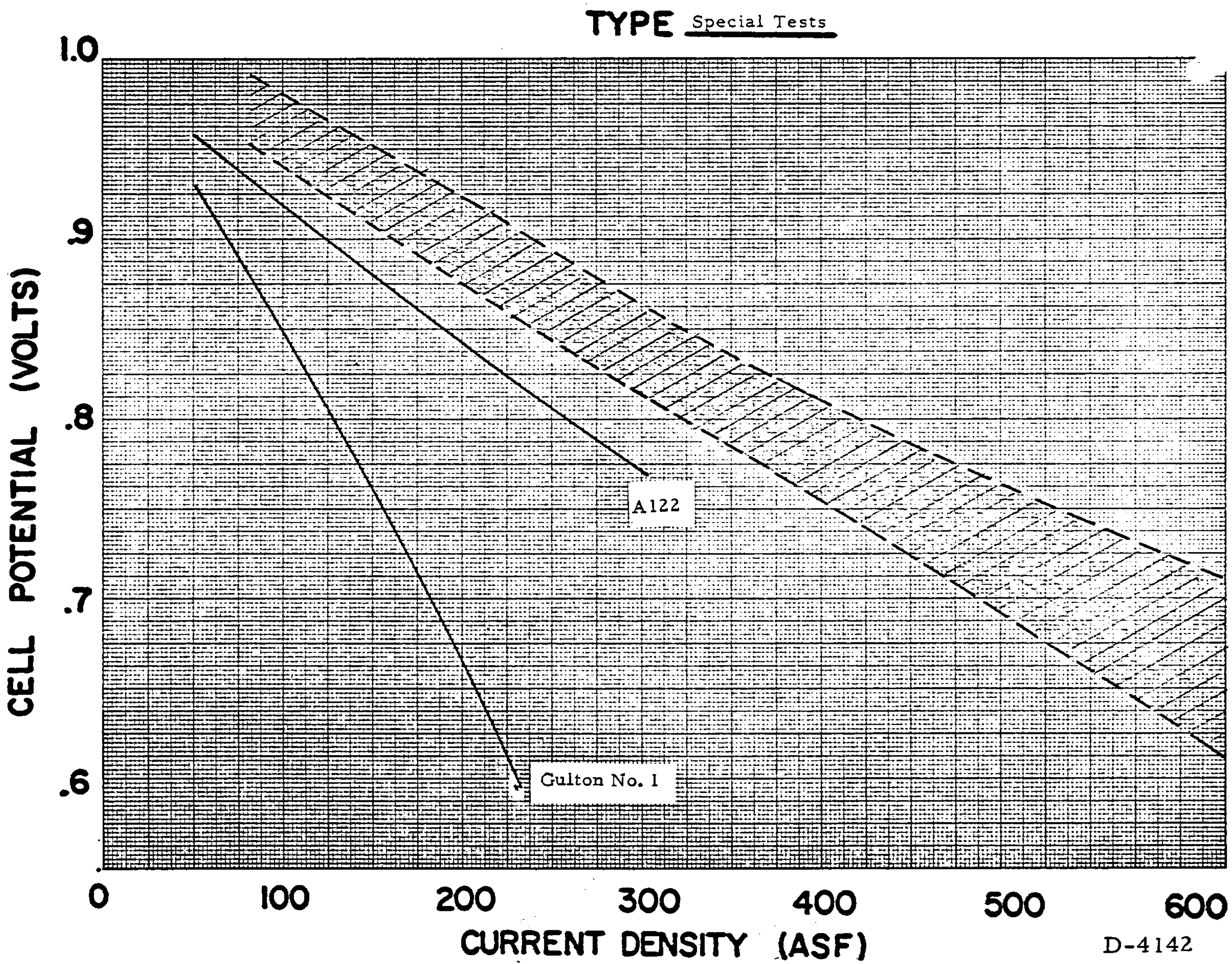

Fig. 29 - Envelope of Initial IRI Polarization Curves for Special Tests.

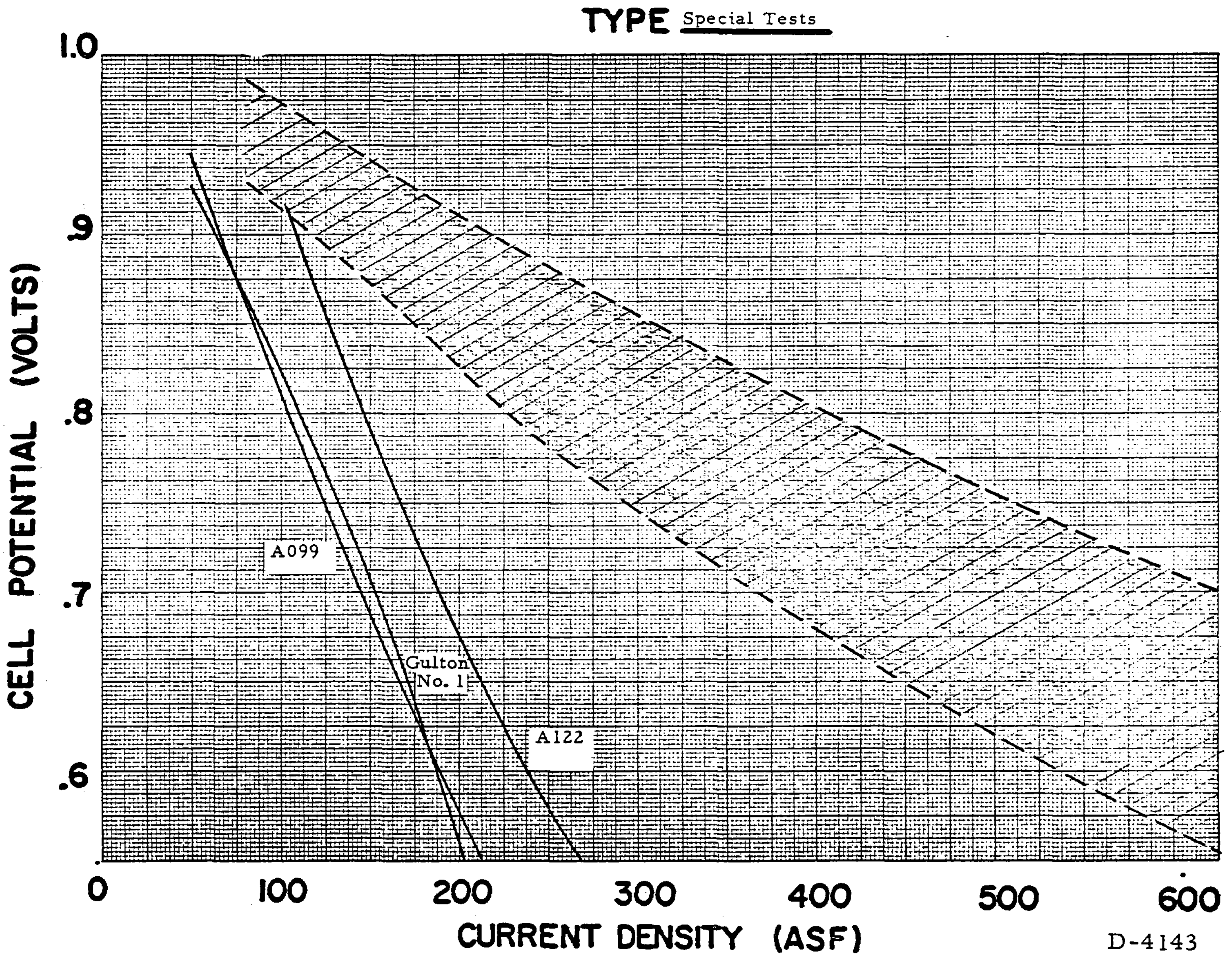

Fig. 30 - Envelope of IRI Polarization Curves After ~500 Hours on Test for Special Tests.

By 1,498 hours, the potential was down to 0.78 v, and after 1,522 hours under load (1,678 hours on test), the unit was removed at 0.70 v. Just before removal, another set of dilute gas tests showed the anode to be very weak; when the dilute H_2 was substituted for the 100 percent H_2, the stack potential dropped to zero in about 70 seconds.

While the stack was still functioning well (at 805 hours under load), it was used to obtain interrupter readings at different temperatures and thus allow estimating the temperature dependence of both the voltage and electrical resistivity. The curves are shown in Fig. 31. These are the values obtained for the resistivity as follows:

Temperature (°C):	36	45	65	90
Resistivity (milliohms · ft^2):	0.84	0.60	0.47	0.34

The temperature dependence of the resistivity is not linear, nor even simple, and was therefore not evaluated analytically. The temperature dependence of the potential is also not simple, but it is most marked at the lower temperatures; less so at the higher end of the scale of interest. To a first approximation, one may take $\Delta V/\Delta T$ = 3 mv/°C between about 50° and 90°C.

In this particular test, thermal cycling had no effect on backing delamination. However, many subsequent tests operated at constant temperature showed delamination of the LAB-40 backing. The relationship between thermal cycling and delamination remains clouded since the fabrication history of the LAB-40 electrodes varied from lot to lot. The question of whether this cycling has an adverse effect upon cell performance is also not easily answered. This cell did degrade somewhat more rapidly than the majority of early cell tests, but degradation was fairly uniform and did not accelerate until cycling had ceased and cell was operating under constant conditions. Anode leakage was low; cathode leakage, high. Very little change was noted in internal resistance. Task I, Milestone 1, was met but Milestone 2 was not.

As previously mentioned (e.g., Cell A-051) after performing a dilute gas test, cell voltage frequently increases. (A dilute gas test consists of separately and sequentially substituting air for oxygen and a 15 percent hydrogen - 85 percent argon mixture for hydrogen.) Cell A-099 was operated

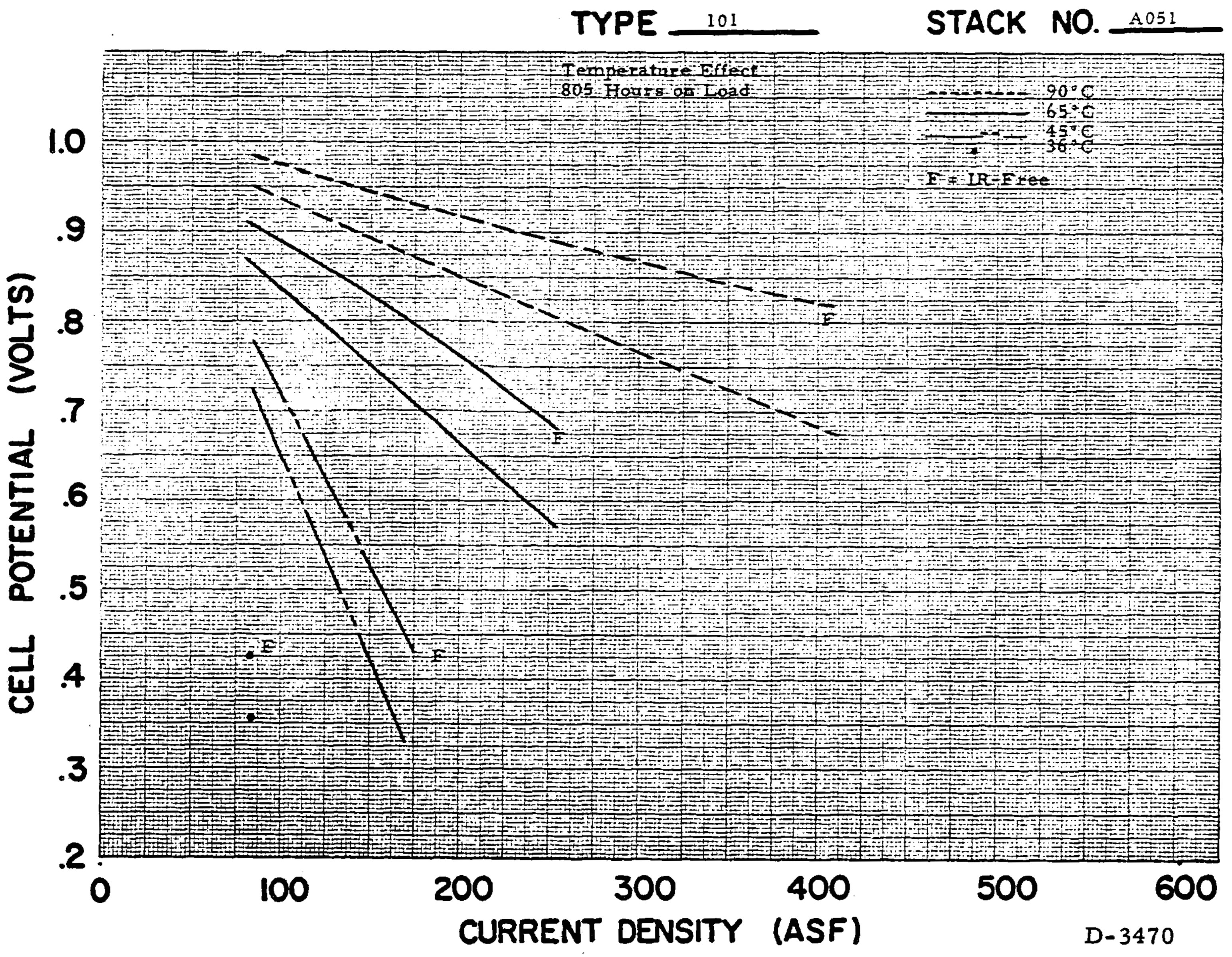

Fig. 31 - Effect of Thermal Cycling at 200 ASF on Polarization Curves.

in a routine manner at 200 ASF but subjected to a weekly dilute gas test. This test proved that although a slight temporary voltage improvement may be observed, a much more rapid decline will follow. This cell suffered extremely rapid decay immediately following the fourth dilute gas test. Periodic dilute gas tests were very detrimental to the T-2 anode. During life test this cell also suffered from poor electrolyte flow. Electrolyte leakage on this cell was extremely low. Internal resistance increased by ~70 percent. Task I, Milestone 1, was met but not Milestone 2. It may well be that the LAB-40 anode would behave differently than the T-2 anode on a similar cycle, but this possibility was not examined.

Another special cell was A-122 which was tested in a horizontal position to reduce the hydrostatic head and thereby improve the pressure balance between gases and electrolyte, thus reducing leakage. We were able to control cathode leakage but the anode leakage was excessive after ~525 hours. This was a poor performing cell both from the standpoint of low initial voltage and rapid decay. Neither Task I Milestone was met. The very poor performance of this cell continued until at 715 hours an accidental admittance of argon gas into the hydrogen manifold caused a marked improvement in the cathode performance. Cell voltage increased dramatically from 0.582 v to 0.883 v (the highest voltage level the cell had ever attained) and did not suffer from another rapid decline until ~1250 hours. At this time, cell failure was because of a poor anode. Internal resistance increased ~30 percent.

Another special cell test designed to study the leakage problem was the "Gulton" cell. This cell was a small 2-1/8 inch diameter cell which was not potted but rather was held together by mechanical means. The electrode edge seals were made by Gulton Inudstries using a proprietary elastomer which is molded directly to the electrode. We were able to control the electrolyte leakage through the electrodes by adjusting the gas pressures to equal the electrolyte inlet pressure. From an electrochemical standpoint this cell was poor, but not surprising. Because the cell was built using existing Gulton molds a very large KOH gap (~0.125") resulted, and, consequently, the cell had high internal resistance. The maximum current density at which it could operate was only 122 ASF. Internal resistance was initially three times higher than average and increased by approximately 40 percent. Cell testing was ultimately terminated due to a very weak cathode.

One special half-cell was constructed to study the hydrogen blow-through phenomenon which occurred on all cells constructed from A-116 to A-125. All these cells utilized water soluble ports in cell construction, and it was suspected that the dissolving of port material or the water washing of the cell rather than any electrochemical properties contributed to the anomalous blow-through. To explore this, a cell was constructed consisting of a H_2 gas space with separator, anode, electrolyte space with horizontal serpentine stick separator and end plates. The original intent was to use no water on these electrodes, to study the blow-through, then to wash and restudy. However, excessive electrolyte leakage necessitated washing of the cell initially so repairs could be made. This half-cell was then put on test and blow-through was detected immediately. The test proved that this phenomenon could in no way be related to the electrochemical performance of the cell nor to the dissolvable port material, but it could be related to the washing of the cell. Two later cell tests, A-126 and A-127, appeared to confirm this finding. Neither of these cells were washed and no anomalous blow-through was experienced.

3.3 Materials Compatibility

Materials of potential use in cell fabrication were tested for compatibility with electrolyte in Ni cylinders containing 200 ml of 12 N KOH. These cylinders were heated at 100°C for one week. Tested materials were removed from the electrolyte, carefully washed and dried, and investigated for any changes in physical properties. The electrolyte was examined for changes in surface tension, equilibrating molarity and chemical contamination. Other Union Carbide supported work subsequently, indicated that the surface tension of the KOH had no deleterious effect upon cell life or performance, and this test was deleted on some later samples.

From the entire group of 65 samples tested, only the following twelve samples were clearly unacceptable because of physical deterioration:

1) Phenoxy A; 2) polypropylene woven-screen Lamports No. 7700; 3) Panelyte No. 164; 4) Epoxy No. 19 (Jones-Darney 5101 and Shell 871 with Z hardener); 5) Epoxy Flex Bar X7050; 6) polyurethane diaphragm; 7) polyisoprene diaphragm; 8) Hypalon diaphragm; 9) Silastic RTV 732; 10) Delrin; 11) Dynel cloth and 12) Dynel wool.

It may be noted that a large number of epoxy samples were tested in the hope of finding a resin which would contribute to the solution of the cell leakage problem. The majority of epoxies tested, however, showed no appreciable deterioration and as far as this test was concerned appeared quite comparable.

Results of the compatibility tests are summarized in Table XLV.

TABLE XLV

MATERIALS COMPATIBILITY TESTS

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Ni Crucible	114.3	13.8	-----	
Unannealed Polysulfone	114.0	15.5	-----	No obvious physical changes, KOH clear.
Annealed Polysulfone	114.2	15.4	-----	Ibid
Kel-F	116.1	14.9	-0.05	Ibid

(Continued)

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
G. E. PPO Hi Frequency Insulated 681-111 (Polyphenylene Oxide)	114.0	13.6	-0.02	Ibid
PPO-Buff Color-No. 53401	113.8	14.0	-0.04	Ibid
Penton	114.4	14.3	+0.003	Ibid
UCC Epoxy (15 g No. 2774, 5 g No. 2793)	114.6	14.0	+0.35	Ibid
UCC Epoxy (50 g No. 2774, 14.5 g No. 0822)	102.4	13.9	+0.49	Physical appearance was changed from translucent whitish to translucent yellowish. No detectable physical changes. KOH clear
UCC Epoxy (25 g No. 2795, 7.25 g No. 0822)	107.3	13.8	+0.53	Ibid, except accompanied by very slight swelling.
50% UCC 2774 and 0822 (10:3) with 50% UCC 2774 and 2793 (4:1)			+0.33	Slight yellowing of material, mottled whitish coating formed around outside diameter, some surface roughening.
UCC Epoxy 2774 and ZZLB-0325 (7:3)			+0.38	Slight darkening of color, slight roughening and pitting of surface.
UCC Epoxy 2774 and ZZLB-0340 (7:3)			+0.32	Slight darkening of color, no other obvious physical changes.
UCC Epoxy 2774 and 2793 Hardener (3:1)			-0.01	Clear sample yellowed slightly in color. No other obvious physical changes.
Rezolin Epoxy Paul Lecher Kunststoff Chem. Co. Inc. 12.5 grams Blend "A" Epoxy 2.4 grams 916 AD Hardener			-0.17	Yellow samples darkened slightly in color. No other obvious physical changes.

(Continued)

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Rezolin Epoxy Paul Lecher Kunststoff Chem. Co. Inc. 10 grams No. L930 Epoxy 6 grams No. L930 Hardener			+30.0	Color change from metallic gray to a dull gray. No other obvious physical changes.
D. Ring Chemical Co. Epoxy 10 grams No. 101 Epoxy 5 grams No. E Hardener			-0.55	Color change from yellow to orange. No other obvious physical changes.
D. Ring Chemical Co. Epoxy 10 grams No. 101 Epoxy 5 grams No. F Hardener			+0.27	Ibid
D. Ring Chemical Co. Epoxy 10 grams No. 100 Epoxy 5 grams No. E Hardener			-0.05	Ibid
D. Ring Chemical Co. Epoxy 10 grams No. 100 Epoxy 5 grams No. F Hardener			+0.62	Color change from yellow to dark yellow. No other obvious physical changes.
D. Ring Chemical Co. Epoxy 13 grams No. 100 Epoxy 11 grams No. A Hardener			-19.1	Submitted sample was a clear tacky flowable slurry. It was spread on a polysulfone sheet and put on test. When removed from test sample had become firm and rubbery. Weight loss could be due to curing or a portion of the sample could actually have flowed off the polysulfone carrier.
D. Ring Chemical Co. Epoxy 10 grams No. 100 Epoxy 5 grams No. A Hardener			+1.33	Sample changed from a transparent yellow to a translucent yellow. No other obvious physical changes.
All Bond Epoxy (1:1) Allaso Products, Braintree, Massachusetts			+0.84	Ibid

Material Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
All Bond Epoxy (2:1) Allaso Products Braintree, Massachusetts			+0.71	Color change from yellow to orange, no other obvious physical changes.
Shell Epoxy 815 (92%) with Armstrong Activator A (8%)			+0.26	Slight yellowing of material, whitish coating formed around outside diameter, no other obvious physical changes.
Carboline XA51 Mix, 15 g Adhesive, 15 g Catalyst	107.7	13.4	+1.33	Physical appearance from a shiny dark blue to dull gray. Some slight swelling. No other obvious physical changes. KOH clear.
Baked Carboline Epoxy on Polysulfone	100.8	14.8	-0.05	Physical appearance from a shiny black to a dull gray with dark patches. KOH clear.
Teflon Rods	114.5	13.8	-0.05	Physical appearance came slightly more white as though slight clearing occurred. No other obvious physical changes. KOH clear.
Nylon Rods	116.0	14.5	-0.12	Material showed a slight yellowing. No other obvious changes. KOH clear.
Natural Polypropylene Tubing; Mfgr: Allied Resin	97.6	14.2	-0.18	Physical appearance was changed from translucent white to translucent yellow. No other obvious changes. KOH clear.

(Continued)

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Polypropylene Electrolyte Spacer	111.6	14.3	-0.23	Physical appearance from clear transparent grid to slightly yellowish, more opaque grid. No obvious strength or dimensional changes. KOH clear.
Rulon A	117.1	15.5	+0.21	KOH did not readily wash off when handled in normal manner. Crystals adhered to surface upon drying. Diam. increase of 0.77%. Color change: dark red to mottled red. KOH clear.
Type 304 Stainless Steel	115.1	14.6	-0.02	No obvious physical change. Gray coating on surface easily wiped off. Yellow color imparted to KOH.
Handy and Harmon No. 630; Braze on Ni-200 Sheet	116.6	14.9	-----	No obvious physical change. Milky color imparted to KOH.
Ni Screen-Chore Girl	113.6	----	+0.17	Material discolored (as oxidation) and embrittled KOH clear.
Fluoroloy	73.8	13.8	+0.03	No obvious physical change. KOH clear.
ABS	68.4	14.1	+0.03	Physical appearance from shiny reflective black to dull mat black. Slight swelling (~1.5%) in thickness. No other obvious change. KOH clear.
Impolene(Polypropylene) Tubing-Natural; Mfgr: Imperial-Eastman	74.6	13.9	-0.28	No obvious physical change. Slight yellowish stain on material. KOH clear.

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Impolene (Polypropylene) Tubing-White; Mfgr: Imperial-Eastman	66.8	14.4	-0.40	No obvious physical change. Slight yellowish stain on material. KOH clear.
Air-Dried Carbonline Epoxy on Polysulfone	81.4	15.0	-0.17	Physical appearance from shiny black to dull gray with black patches. KOH clear.
Air-Dried Neoprene Cement(B. F. Goodrich) on Polysulfone Sheet	114.6	12.9	-0.21	No obvious physical changes. KOH clear.
Epoxy No. 8-A; Maraset 124-C with No. 75 Hardener	115.5	14.2	-0.09	Ibid
Ethylene Propylene Diaphragm AiResearch Division Garrett Corp.		13.1	-0.49	No obvious physical changes of material. KOH has tiny white particles suspended in it and had a rubber-like odor.
Ni Stranded Wire Welded to Ni Tab	114.0	13.9	0.00	Weld showed no sign of deterioration. Ni stranded wire corroded to a brownish color as of nickel oxide.
Styrene with 40% Fiber Glass Liquid Nitrogen Processing Corp., Malvern, Penna.	108.7	12.9	+4.41	Color changed from tan to white. Surface became rough. Swelling of ~7%. KOH clear.
Expanded Nylon, Code 10, 65-1 Stabilized Exmet Corp., Bridgeport, Conn.	105.6	12.8	-1.61	Postmortem of two NASA cells incorporating this material as KOH separator revealed its disintegration. These cells were on test 1100 and 1800 hours. No indication of disintegration is evident from this one week test (168 hours). Material is stiff before test and no appreciable

(Continued)

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Expanded Nylon Code 10, 65-1 Stabalized Exmet Corp., Bridgeport, Conn. (Continued)	105.6	12.8	-1.61	change in physical properties is detectable. Material did shrink ~12% across width and stretch ~4% along length, but it is possible to stretch material back to approximate original size. KOH clear. An extended time test is probably necessary to show effect of KOH upon this material.
Expanded Teflon Exmet Corp. Bridgeport, Conn.	75.9	12.4	-0.28	Material more pliable than nylon. Material did shrink ~30% across width and stretch ~4% along length, but it is possible to stretch back to approximate original size. No obvious physical changes. KOH clear. No disintegration of this material has as yet been observed in any cell post-mortems.
EPT Sheet Compound EP-47, Sulfur-free Philpott Rubber Co.	69.5	12.6	+0.55	Original whitish appearance Thyaran no longer evident after test. Sample became slightly sticky. No other obvious physical changes. KOH yellow.
EPT Sheet Compound EP-47. Retested after washing, drying, and degreasing	109.8	14.0	-1.62	This sample of previously tested material was washed with Alconox, dried, washed with Freon, dried and retested. Only notable change was that the surface tension of the KOH was not lowered as in the previous test.

(Continued)

Materials Tested	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
EPT Sheet Compound EP-47. Retested after washing, drying, and degreasing. (Continued)	109.8	14.0	-1.62	Physical properties of material remained unchanged. Slight stickiness still evident. KOH clear.
Sulfur-free EPT "O" Rings	60.9	13.6	-1.79	No obvious physical changes. KOH clear. Presoaking in KOH would probably eliminate this effect on surface tension.
U. S. I. Chemicals Co., No. 6115 Cross Link Polyethylene Molded by "Scott Molders" Kent, Ohio			-0.55	This material originally is a rubbery black open structure containing staggered 1/2 in. and 3/16 in. holes with ~1/16 in. material between holes. Material became slightly gray in color and stiffened slightly. No other obvious physical changes.
U. S. I. Chemicals Co. No. 6312 Cross Link Polyethylene Molded by "Kent Molded Plastics, Inc. "Kent, Ohio			-0.35	This material is originally a rubbery black solid structure with 1/16" ribs placed at 3/8" spacing across sheet. The only physical change noted here was a very slight embrittlement of sample.
Zytel 101 Nylon			1 week -0.15	Slight yellowing
DuPont			9 weeks -1.37	Yellow spots on non-submerged end. Slight etching of surface.
Zytel			1 week -0.15	Slight yellowing
DuPont			9 weeks -3.39	Yellow spots. Heavily etched. Soft.

(Continued)

Materials	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change %	Comments
Phenoxy A	97.3	13.9	+2.10	Physical appearance from clear transparent to translucent whitish sheet. Lost strength, became brittle, tore easily. Thickness increase ∼25%. KOH clear. Unacceptable.
Polypropylene Woven Screen (Lamports-7700)	104.2	13.7	-0.41	Appearance from a white opaque to yellow opaque. Lost strength, became brittle, broke with slight pressure. Less than 1/2 sample below KOH surface. KOH clear. Unacceptable
Panelyte No. 164	104.0	14.4	+0.68	Lost smooth green laquer coating leaving rough tan woven structure. Swelling: ∼20% (edges) - ∼8% (center). Edges powdery; KOH clear. Unacceptable.
Polyisoprene Diaphragm AiResearch Division, Garrett Corp.	71.0	12.9	-4.4	Material became soft and tacky. Lost physical strength. Negligible thickness change. KOH clear. Unacceptable.
Polypropylene Wood			4 weeks -1.9	Slight yellowing of color. Shrinkage ∼5%. Material maintained its strength and stability. No other obvious physical changes.
Hypalon Diaphragm AiResearch Division Garrett Corp.	83.0	12.7	-0.53	Material became stiff and lost much of its elasticity. Color changed from light blue to light green. No thickness change. KOH clear. Unacceptable.

(Continued)

Materials	KOH Surface Tension (dynes/cm)	KOH Concentration (M)	Weight Change (%)	Comments
Polyurethane Diaphragm AiResearch Division Garrett Corp.	41.6	13.1	-40.4	Lost physical strength and could be easily pulled apart. Thickness decreased from 30 to 80%. Note low KOH surface tension. KOH slightly yellow. Unacceptable.
Premixed Epoxy (Short Glass Fill) Flex Bar X7050 U. S. Polymeric	55.3	13.5	+0.89	Physical appearance changed from a black to gray color. KOH adhered to surface and was not washed off during normal washing procedure. Material became soft and flaked off easily. KOH clear. Unacceptable
Epoxy No. 19: Jones-Darney 5101 and Shell 871 with Z Hardener	99.7	14.0	+15.34	Material became very brittle and broke easily. Some internal cracking occurred during test. KOH clear. Unacceptable
Silastic RTV 732 (Silicone Rubber) Dow Corning Corp.	104.0		-46.07	Tiny pinholes were formed in material and one edge became very thin. Very easily torn apart. KOH clear. Unacceptable
Delrin (Acetal Resin Plastic) Mfgr: E. I. duPont Five different types of material tested.				Material disintegrated. KOH yellow. Unacceptable.
DYNEL Cloth			4 week test	Originally a cream color canvas-like fabric. Turned dark brown, disintegrated in part, and remaining fabric became very weak. Unacceptable.
Kendall DYNEL Wool No. H479-A			4 week test	Original white wool material changed to a brown compact ball. Some disintegrated and remainder easily torn apart. Unacceptable.

SECTION IV

4.0 CONCLUSIONS AND RECOMMENDATIONS

Based upon the factual data presented in Section 3.0 of this report, the following conclusions are warranted:

a) Initial performance levels of 0.88 to 0.90 volt are obtainable at 200 ASF in cells operating in circulating 14 N KOH at 90 to 100°C and 15 psia using either Union Carbide T-2 anodes or American Cyanamid LAB-40 anodes coupled with LAB-40 cathodes. Voltage degradation rates of less than 20 mv/500 hours were experienced in many of these same cells.

b) Increasing the operating pressure from 15 to 30 psia results in about a 30 mv increase in cell potential. Although the data is inconclusive, there appears to be an effective increase in lifetime at the higher operating pressure.

c) Based upon a cut-off potential of 0.78 volt, the maximum lifetime achieved during continuous operation at 200 ASF was 2720 hours.

d) Delamination of the wet-proofed backing layer from the LAB-40 electrodes was a recurrent problem throughout the course of the contract. The C-II backing modification (material and fabrication) tested near the end of the program appeared to solve this problem.

e) The optimum cell construction evolved included the following features:

(1) Polysulfone structural materials;
(2) Silver current collection tabs welded to the electrodes;
(3) Horizontal serpentine KOH separator;
(4) Expanded polysulfone mesh gas separators;
(5) Use of pre-machined rather than water soluble ports and orifices.

The optimum adhesive or potting epoxy for cell fabrication was not established.

f) In cells which were water-washed as an essential part of the fabrication operation, anomalous blow-through of hydrogen into the electrolyte was generally observed. Manifestation of this anomalous blow-through was bubbling of hydrogen gas into the electrolyte even though the electrolyte pressure was higher than the H_2 pressure. Other investigators have observed a similar transport phenomenon with highly wet-proofed porous structures.

g) The major unsolved problem during this program was leakage of electrolyte into the gas spaces. In many cases it was impossible to determine whether the leakage came directly through the active portion of the electrodes or through the electrode edge seals. Leakage was, generally, more prevalent on the cathode side of the cells. Although construction improvements such as welded tabs, alternate epoxy adhesives, and alternate potting procedures minimized the problem, the leakage was not eliminated.

h) Excellent performance was obtained from cells operating on simulated system duty cycle entailing 124 ASF (84%), 63 ASF (9%) and 200 ASF (7%) cyclic loads.

i) Successful operation of "flight-size" cells containing 1.3 ft^2 active electrode area (sub-divided into four anode-cathode pairs) at 15 and 30 psia and 200 ASF established the feasibility of extrapolating the construction and testing work on 1/8 ft^2 cells to multi-cell prototype stacks.

j) Preliminary studies on a T-3N cathode as a means of reducing or eliminating cathode weepage were promising in that 1) half-cell potentials in the same range as the LAB-40 cathodes were obtained in 2 cm^2 cells and 2) weepage in short-duration tests was extremely low. Considerable additional experimental work would be required to bring the T-3N cathode to the same state of development as the other electrodes emphasized in this program.

Since the major unsolved problem encountered during the performance of Contract NAS3-9430 was electrolyte weepage into the gas spaces and since the T-3N electrode concept offers considerable promise of solving this problem, we would recommend that additional work be undertaken to conclusively demonstrate the virtue of this concept in high performance electrodes suitable for NASA missions. Upon successful demonstration, the concept could then logically be extended to T-3N <u>anodes</u> as well. This electrode development work should be supplemented by a companion cell fabrication program designed to select the optimum adhesive or potting resin for leak-tight cells with a 3000 hour operational capability.

APPENDIX

Individual Cell Performance Curves

STACK NO.: A 073
TYPE: 101
CATHODE: Lab-40 A-II-2
ANODE: UCC T-2

CURRENT DENSITY: NASA Load Cycle
AVG. TEMP.: 90°C
AVG. NORMALITY: 11.5 N
RACK POSITION: D-3

PRESSURE LEVEL: 15 psia

——— 200 ASF
- - - - 124 ASF

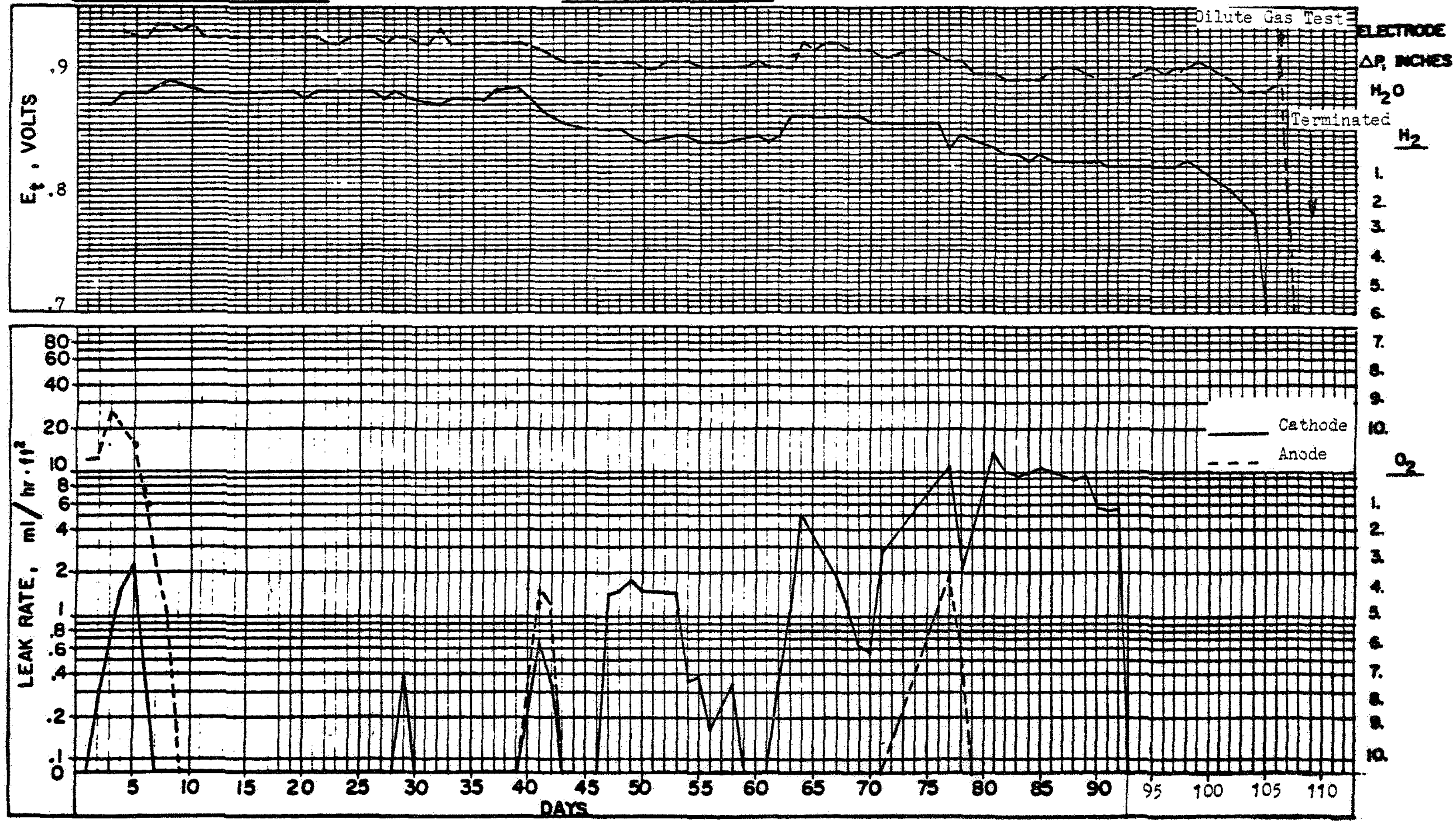

Figure A-1 - Voltage and Leak Rate versus Time on Load - Cell A-073

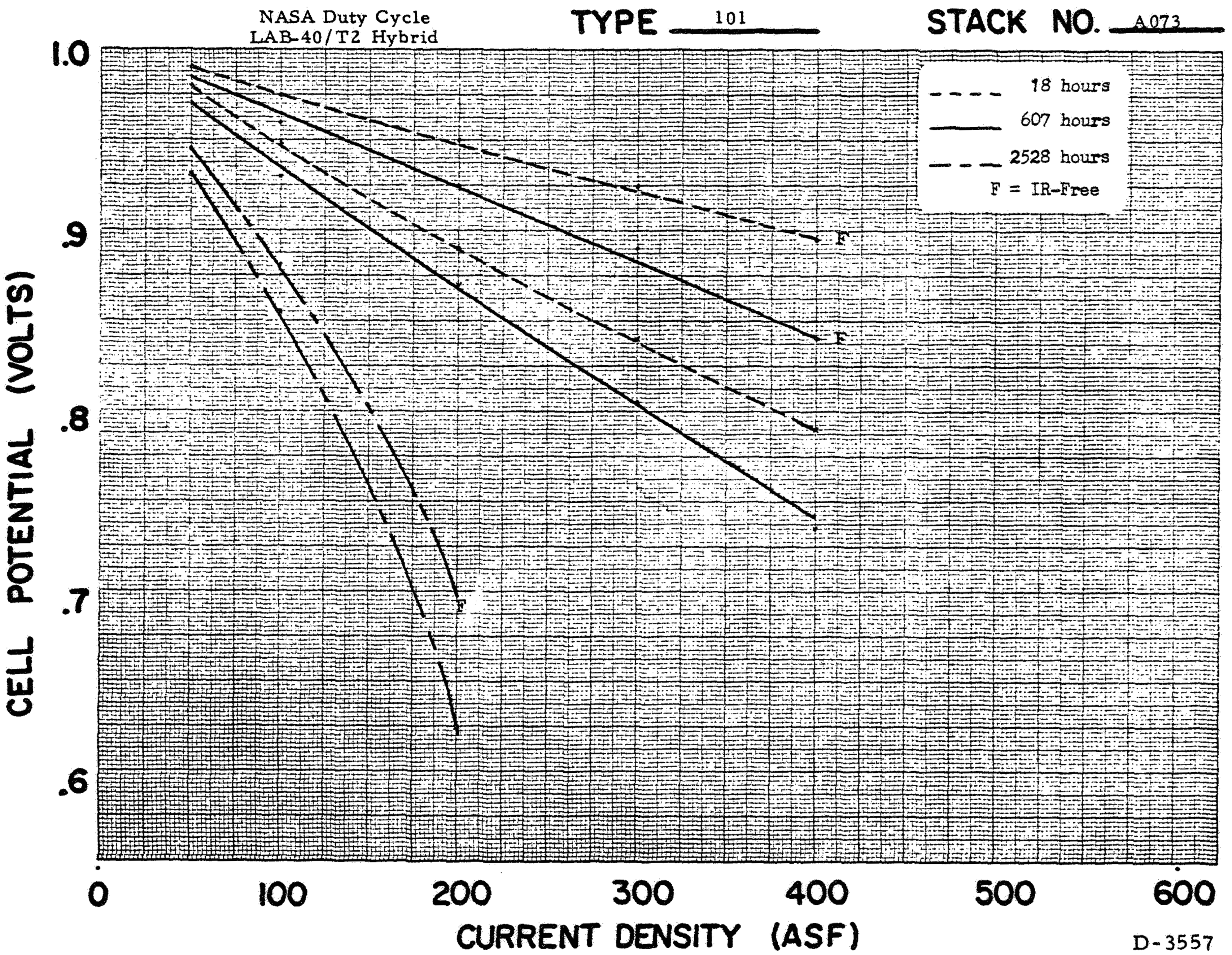

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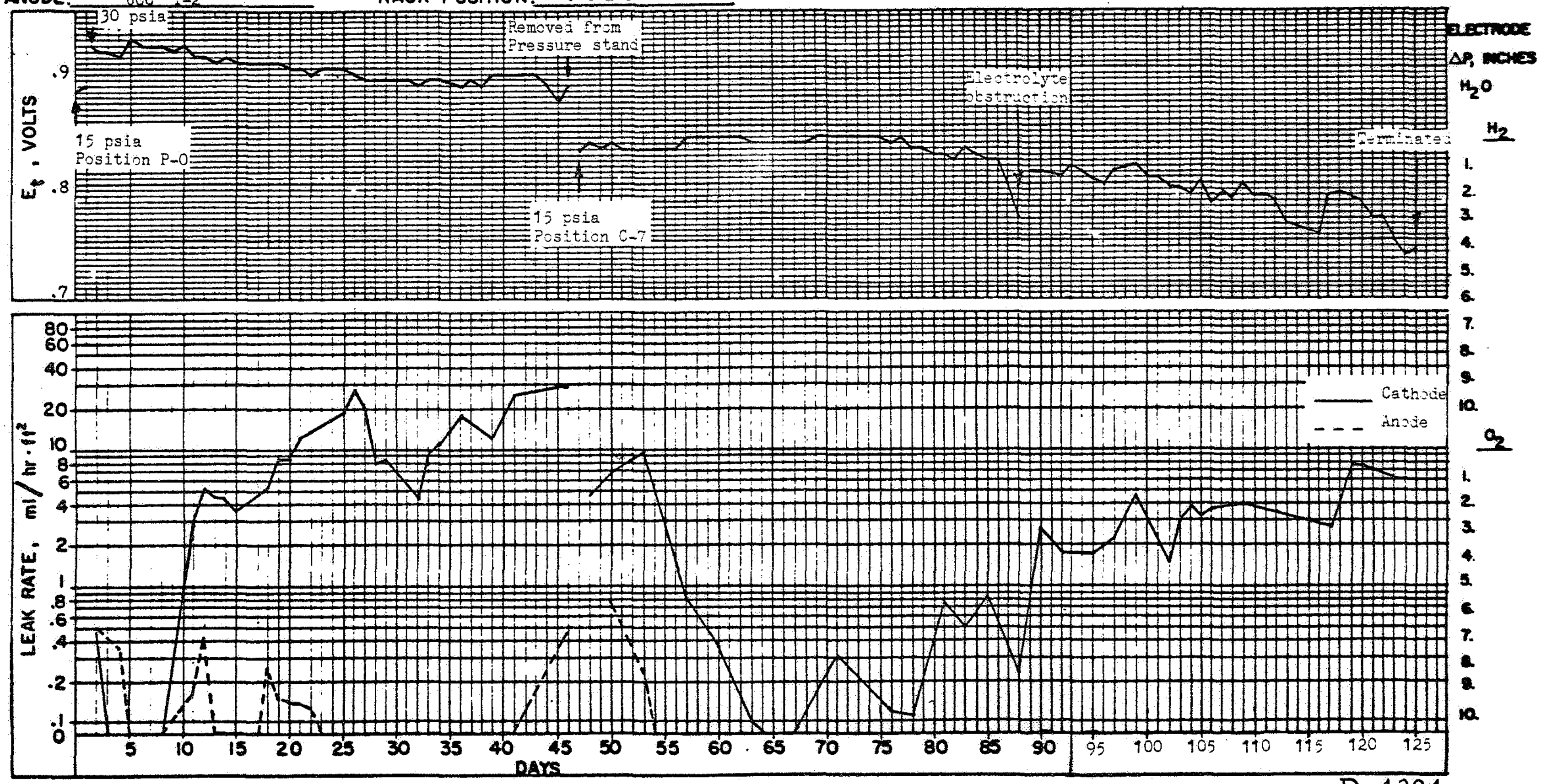
Figure A-2 - Polarization Curves - Cell A-073

STACK NO.: A 074 **CURRENT DENSITY:** 200 ASF **PRESSURE LEVEL:** 30 & 15 psia

TYPE: 101 **AVG. TEMP.:** 90°C

CATHODE: Lab-40 A-II-1 **AVG. NORMALITY:** 14 N

ANODE: UCC T-2 **RACK POSITION:** P-0 & C-7



D-4304

Figure A-3 - Voltage and Leak Rate versus Time on Load - Cell A-074

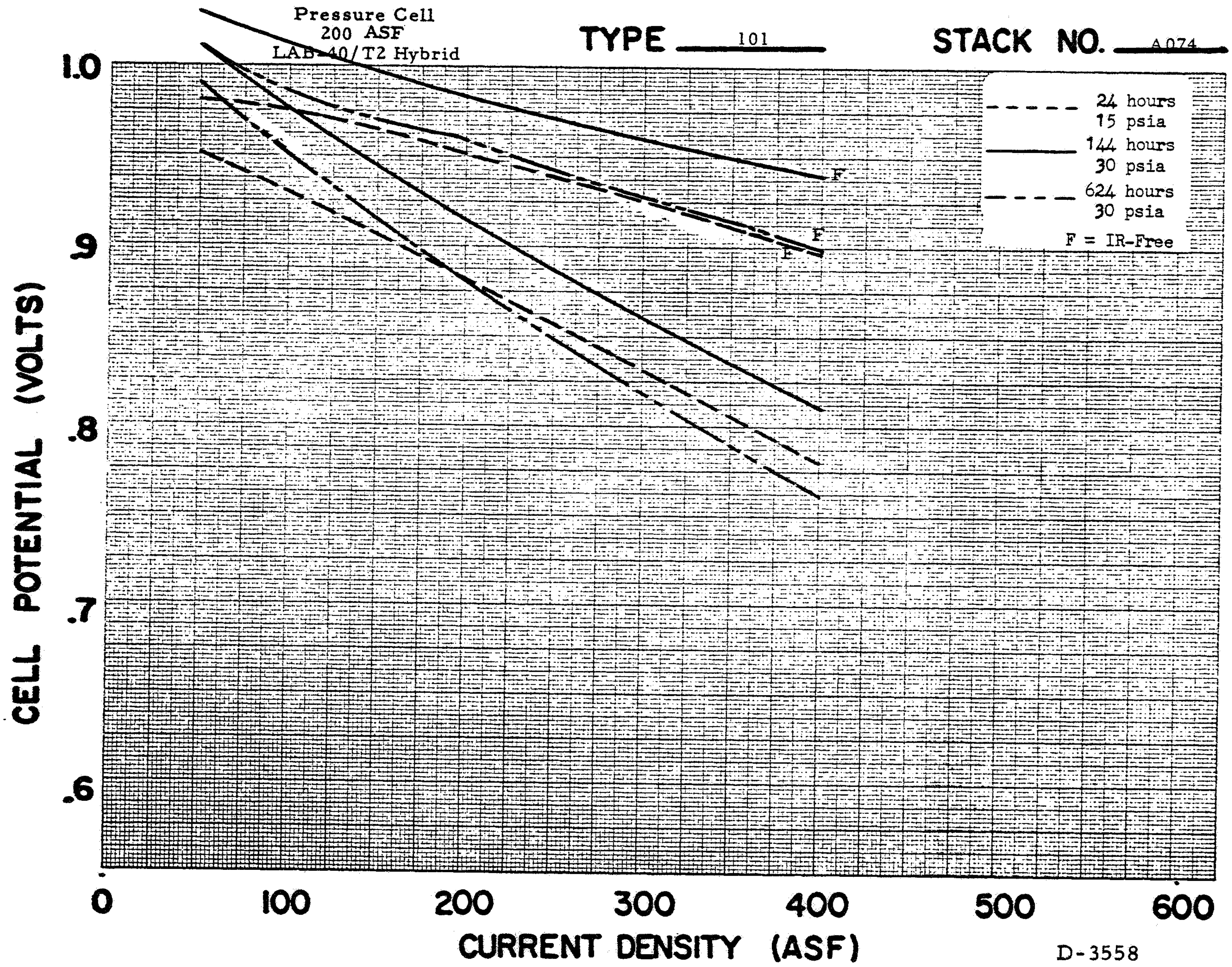


Figure A-4 - Polarization Curves - Cell A-074

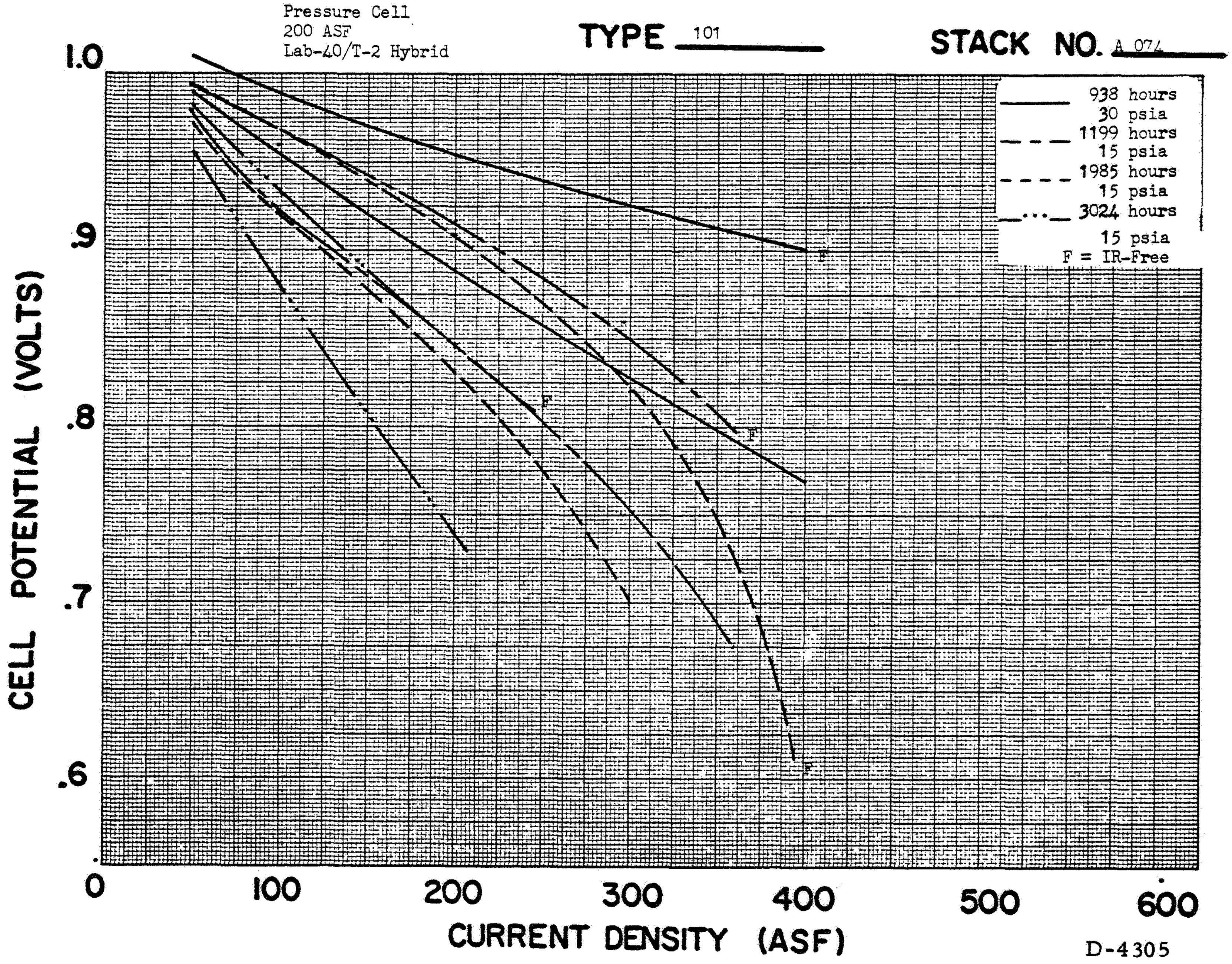
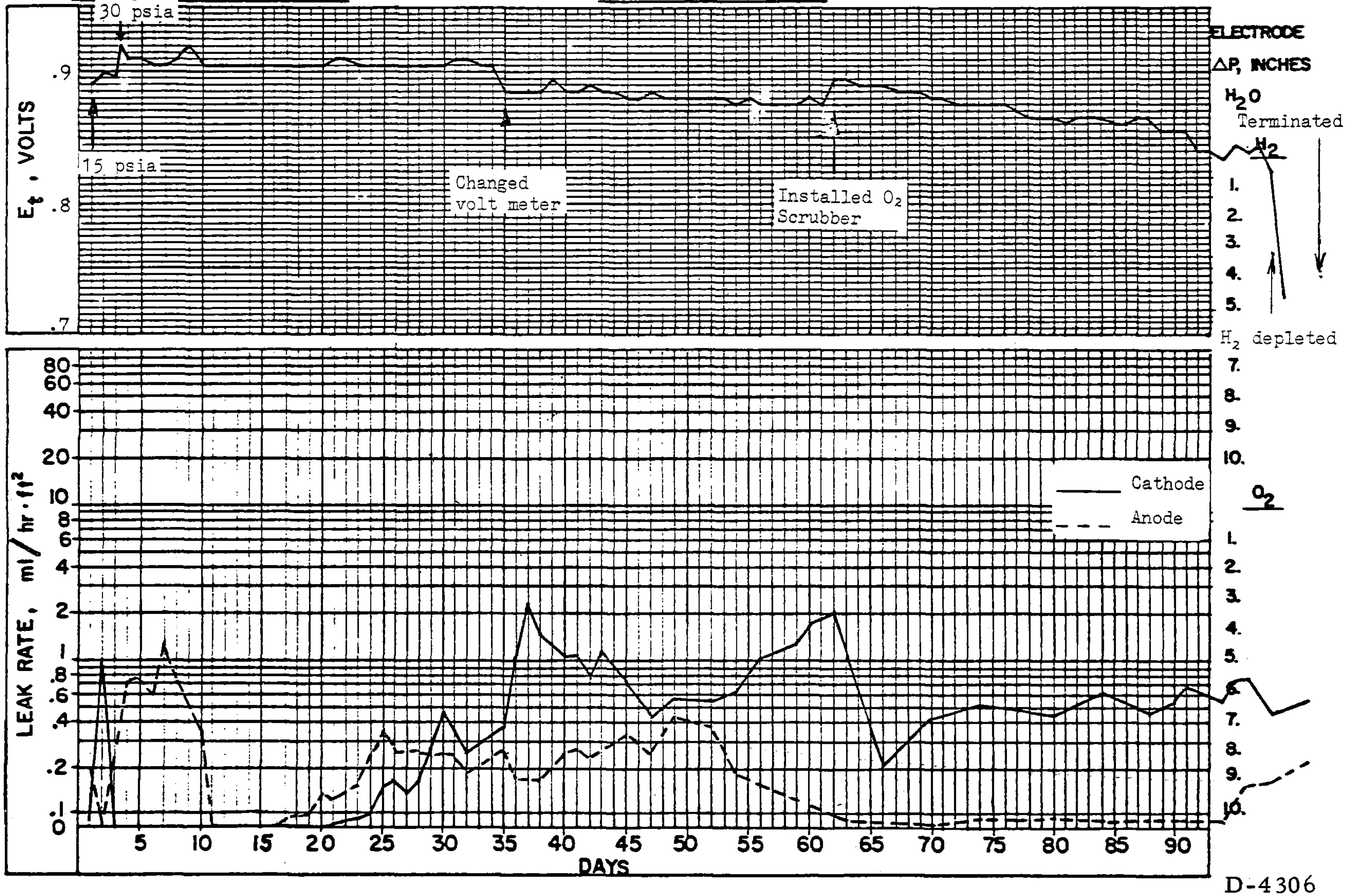


Figure A-5 - Polarization Curves - Cell A-074

STACK NO.: A 078
TYPE: 101
CATHODE: Lab-40 B-II-4
ANODE: UCC T-2

CURRENT DENSITY: 200 ASF
AVG. TEMP.: 90°C
AVG. NORMALITY: 14 N
RACK POSITION: P-2

PRESSURE LEVEL: 30 psia



D-4306

Figure A-6 - Voltage and Leak Rate versus Time on Load - Cell A-078

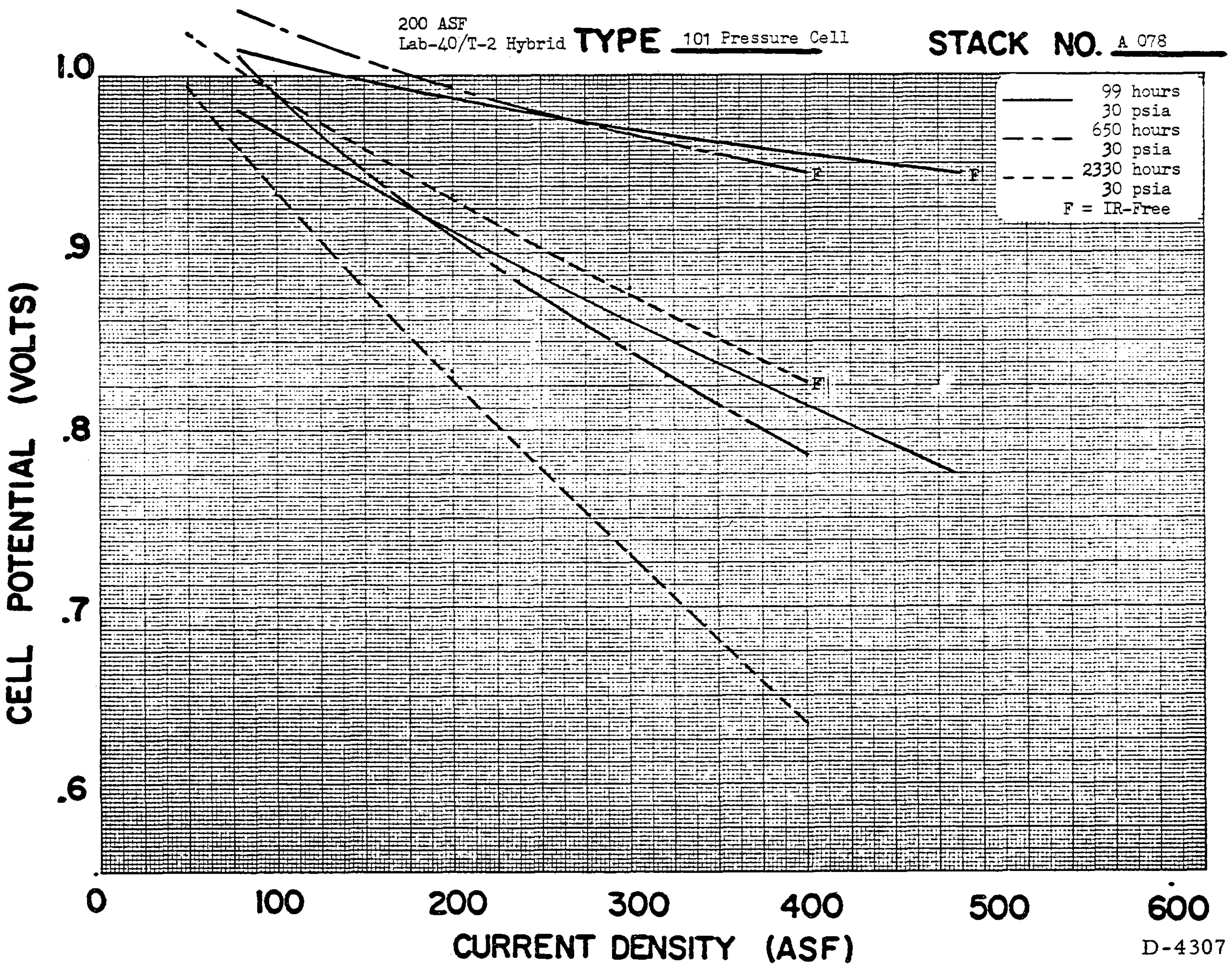
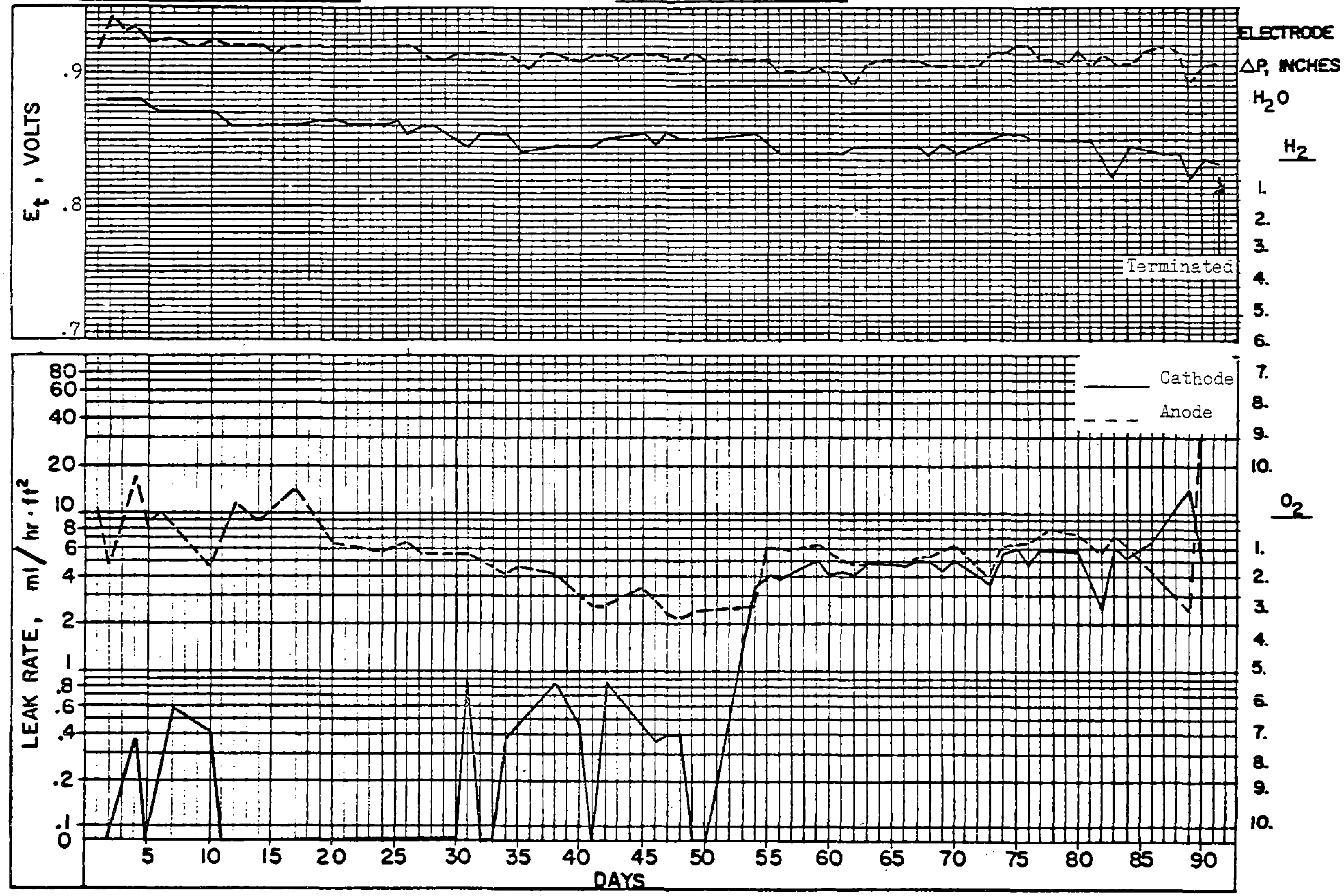


Figure A-7 - Polarization Curves - Cell A-078

STACK NO.: A 084
TYPE: 101
CATHODE: Lab-40 143-A
ANODE: UCC T-2

CURRENT DENSITY: NASA Load Cycle
AVG. TEMP.: 90°C
AVG. NORMALITY: 11.5 N
RACK POSITION: D-2

PRESSURE LEVEL: 15 psia
——— 200 ASF
- - - - 124 ASF



D-4308

Figure A-8 - Voltage and Leak Rate versus Time on Load - Cell A-084

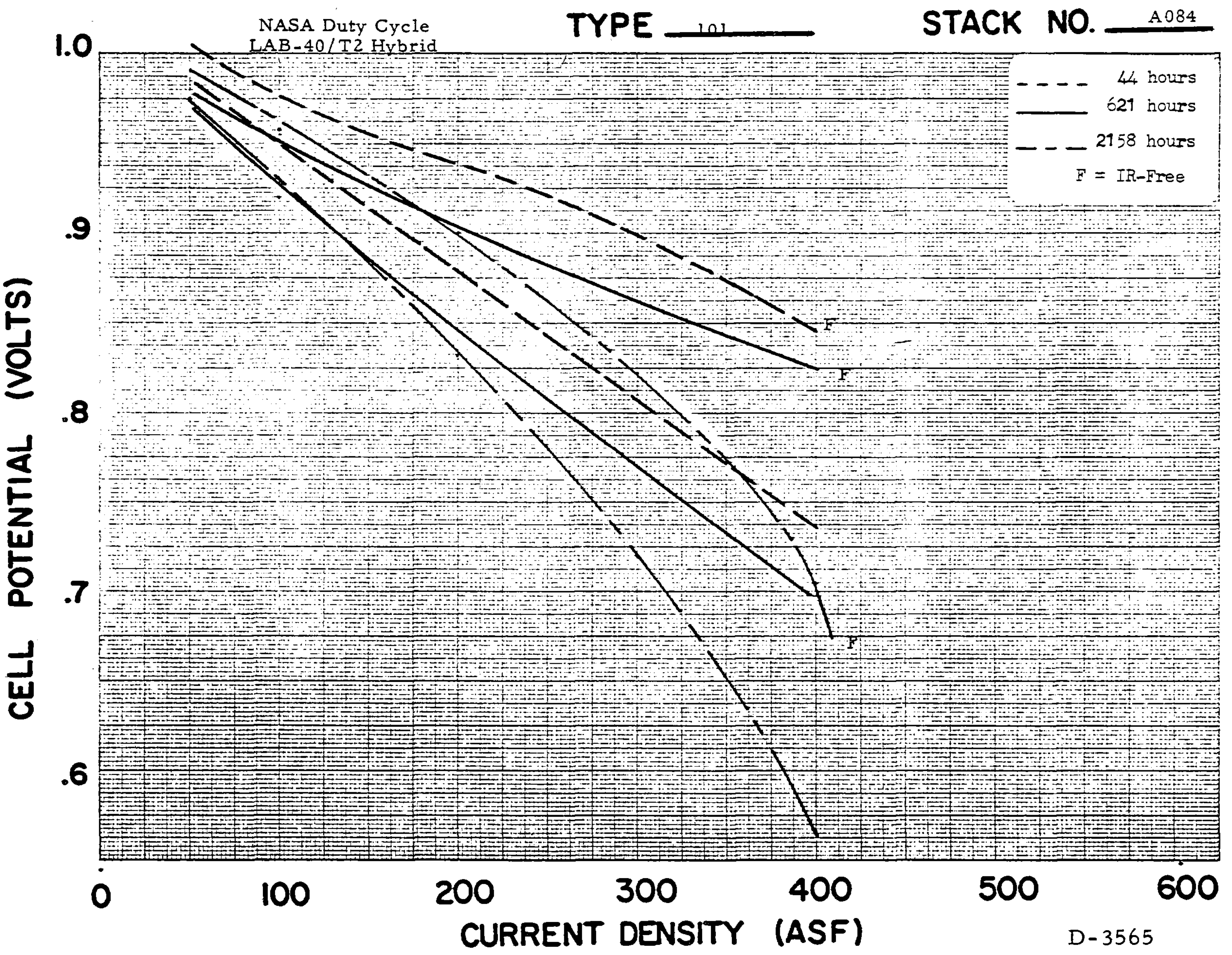


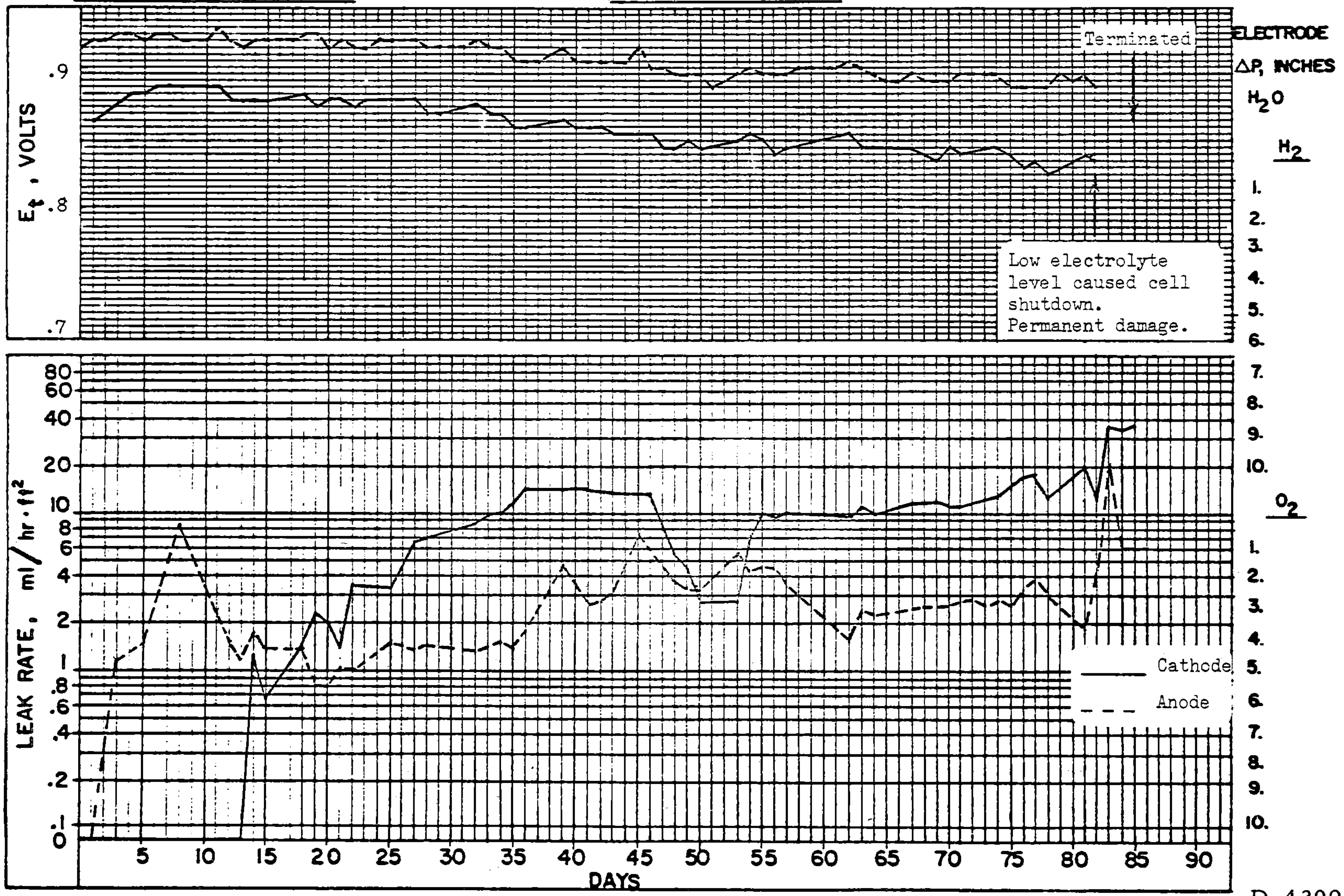
Figure A-9 - Polarization Curves - Cell A-084

STACK NO.: A 090
TYPE: 101
CATHODE: Lab-40 143B
ANODE: UCC T-2

CURRENT DENSITY: NASA Duty Cycle
AVG. TEMP.: 90°C
AVG. NORMALITY: 11.5 N
RACK POSITION: D-5

PRESSURE LEVEL: 15 psia

——— 200 ASF
- - - - 124 ASF



D-4309

Figure A-10 -Voltage and Leak Rate versus Time on Load - Cell A-090

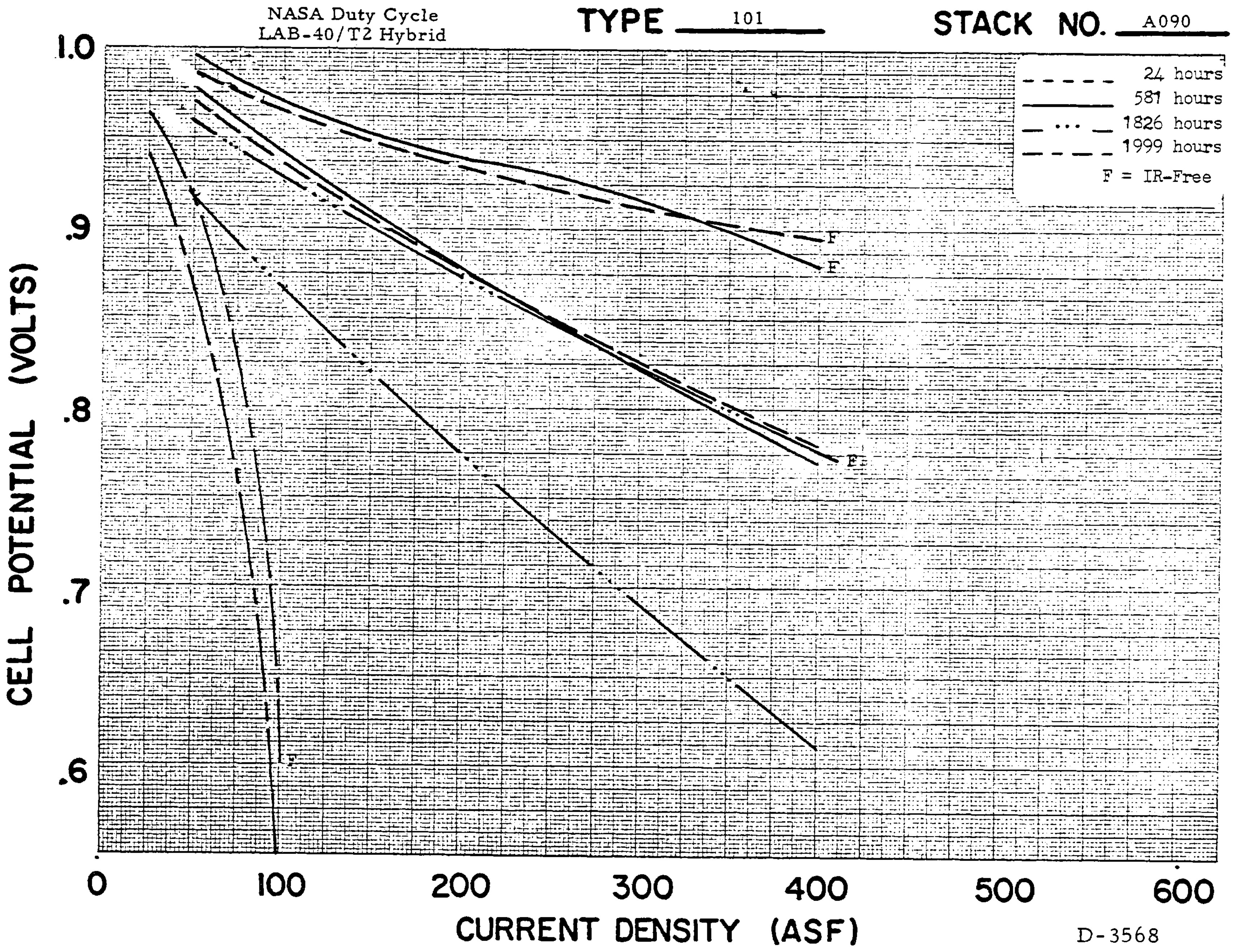


Figure A-11- Polarization Curves - Cell A-090

STACK NO.: A115
TYPE: 101
CATHODE: Lab-40 A-II-2
ANODE: Lab-40 A-II-2

CURRENT DENSITY: NASA Cycle
AVG. TEMP.: 90°
AVG. NORMALITY: 14N
RACK POSITION: P-6

PRESSURE LEVEL: 30 psia
———— 200 ASF
- - - - - - - 124 ASF

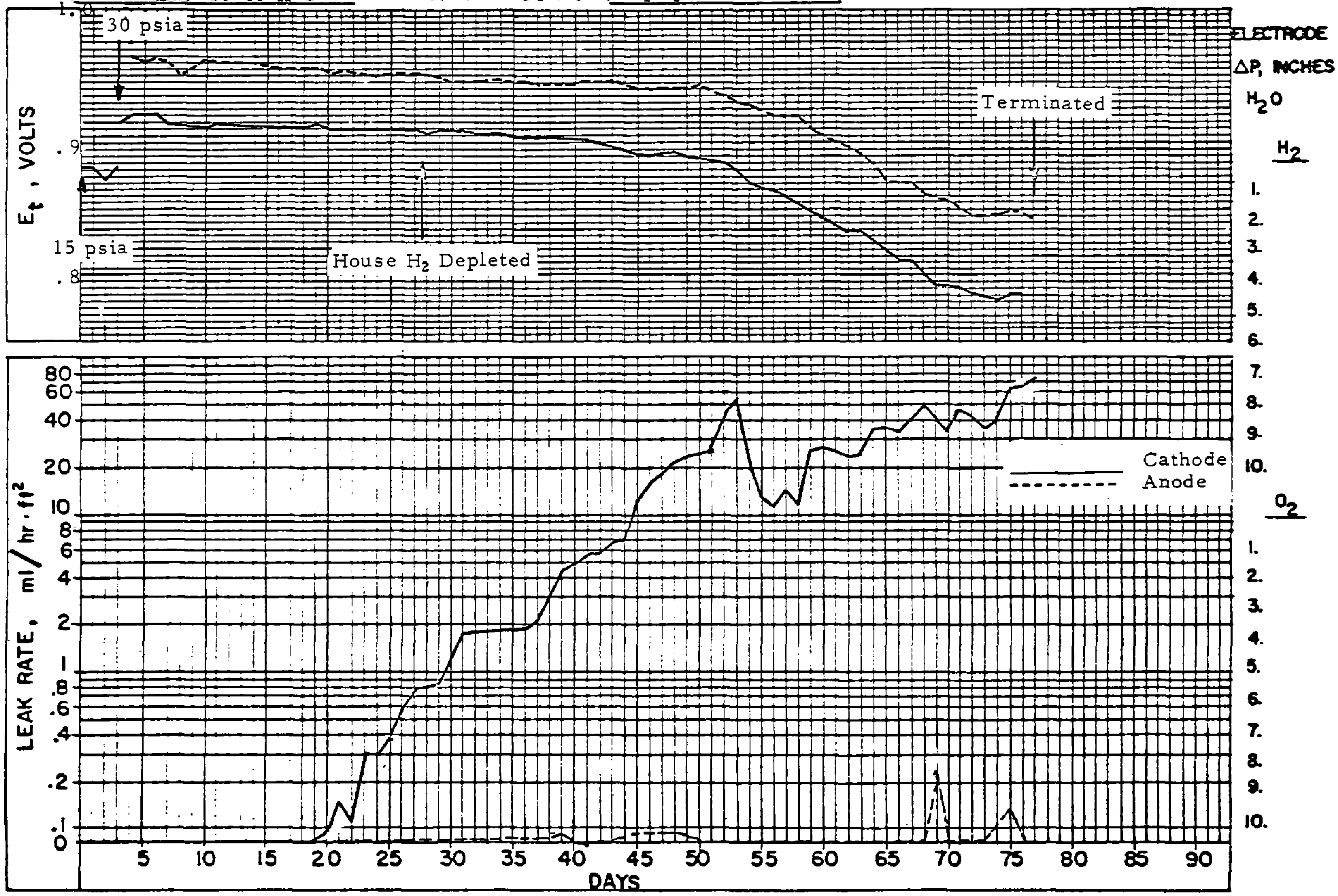


Figure A-12 - Voltage and Leak Rate Versus Time on Load - Cell A-115

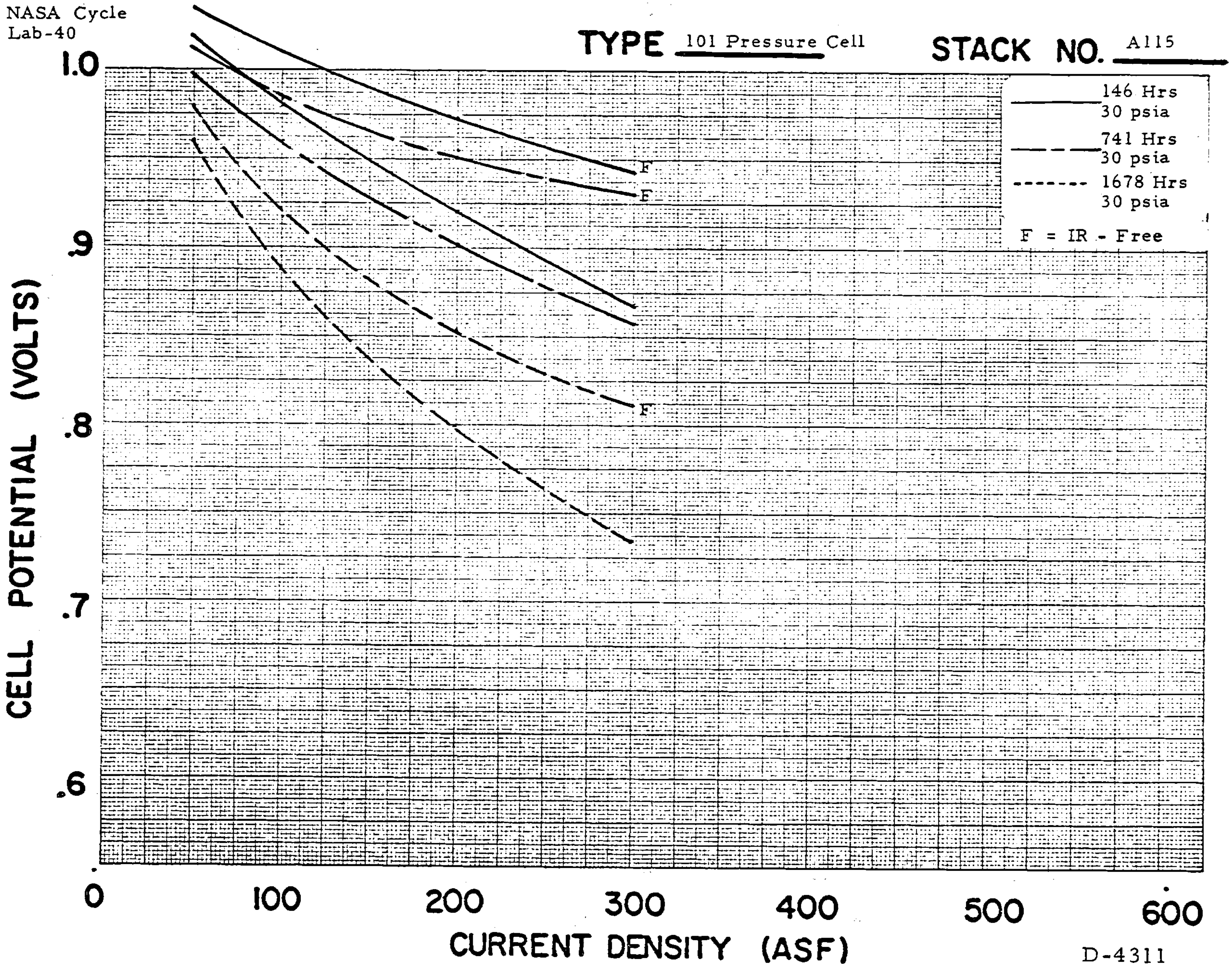
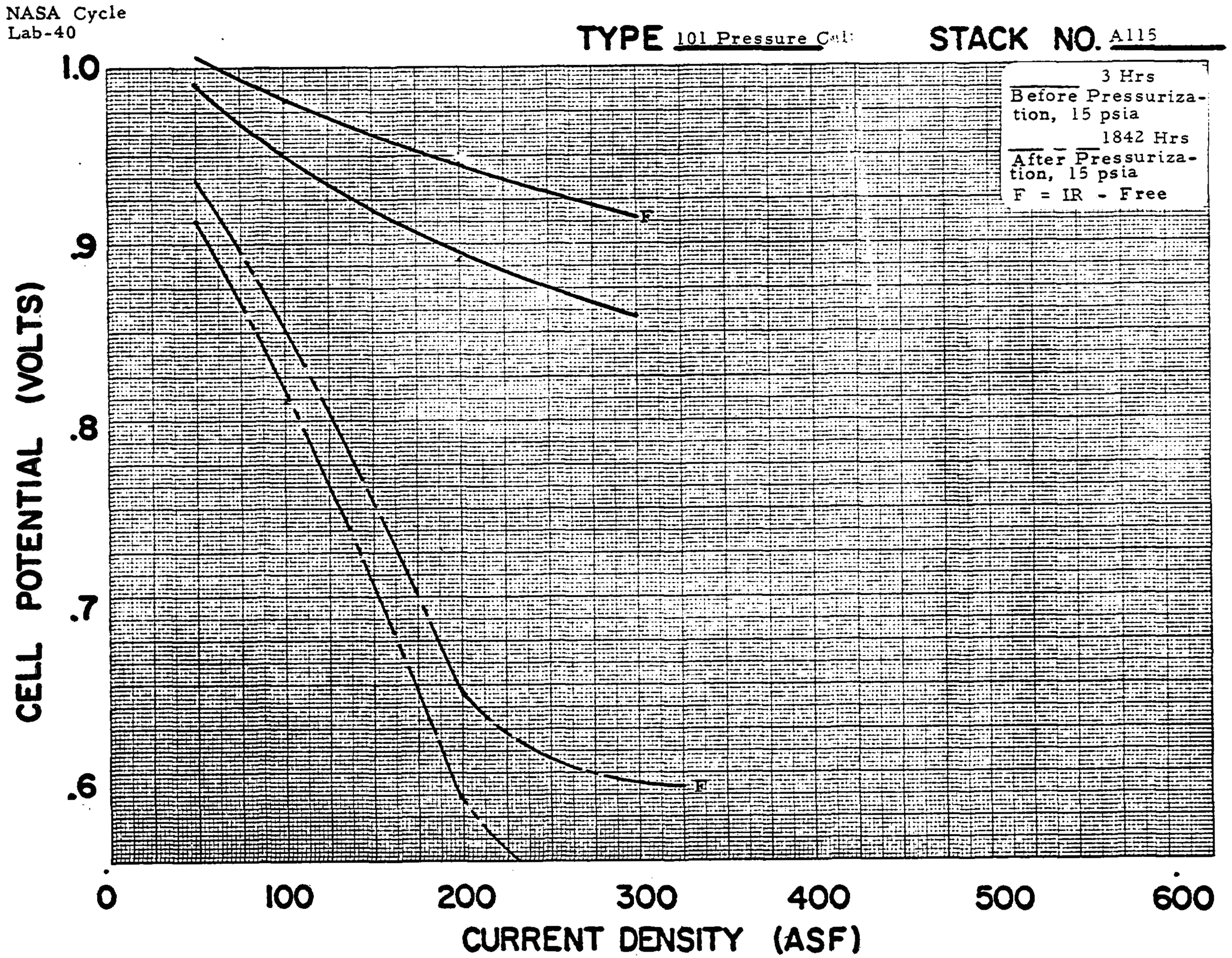


Figure A-13 - Polarization Curves - Cell A-115



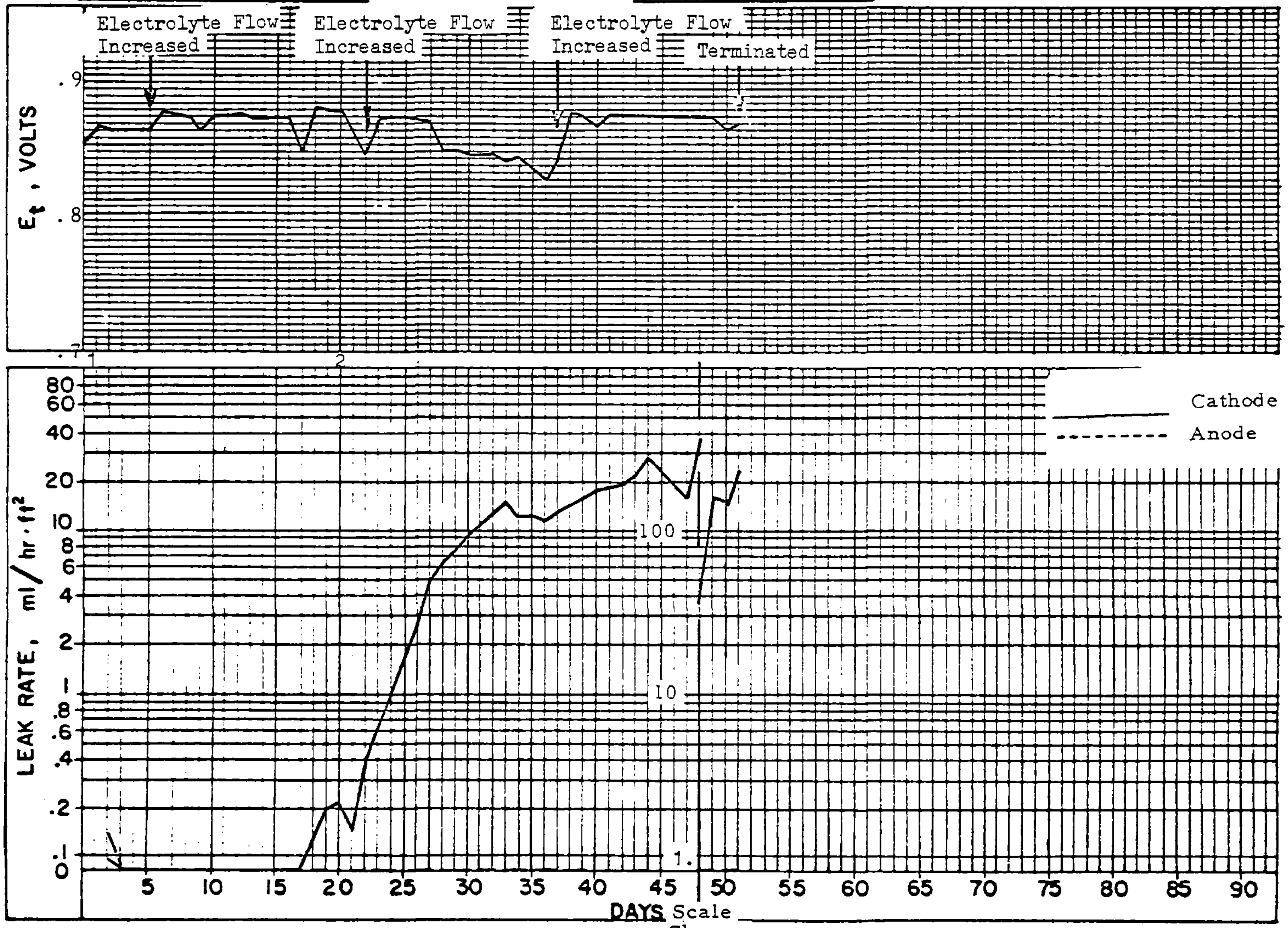
D-4312

Figure A-14 - Polarization Curves - Cell A-115

STACK NO.: A116
TYPE: 101
CATHODE: Lab-40 B-II
ANODE: UCC T-2

CURRENT DENSITY: 200 ASF
AVG. TEMP.: 90°C
AVG. NORMALITY: 14N
RACK POSITION: C-6

PRESSURE LEVEL: 15 psia



Gas Inlet Pressure In. H_2O

H_2
1. 15.0
2. 25.0
3.
4.
5.
6.
7.
8.
9.
10.

O_2
1. 15.7
2. 19.0
3.
4.
5.
6.
7.
8.
9.
10.

D-4313

Figure A-15 - Voltage and Leak Rate Versus Time on Load - Cell A-116

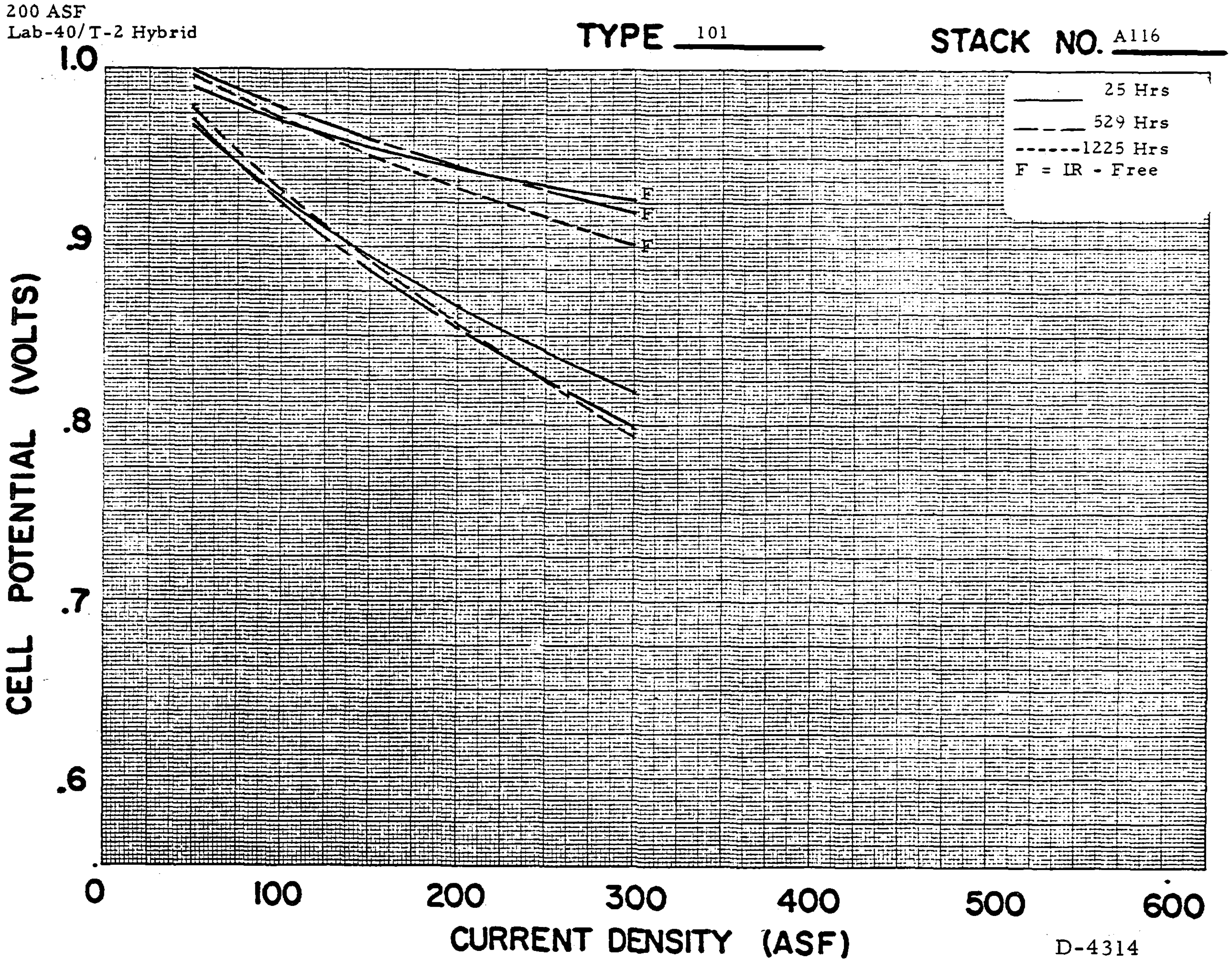
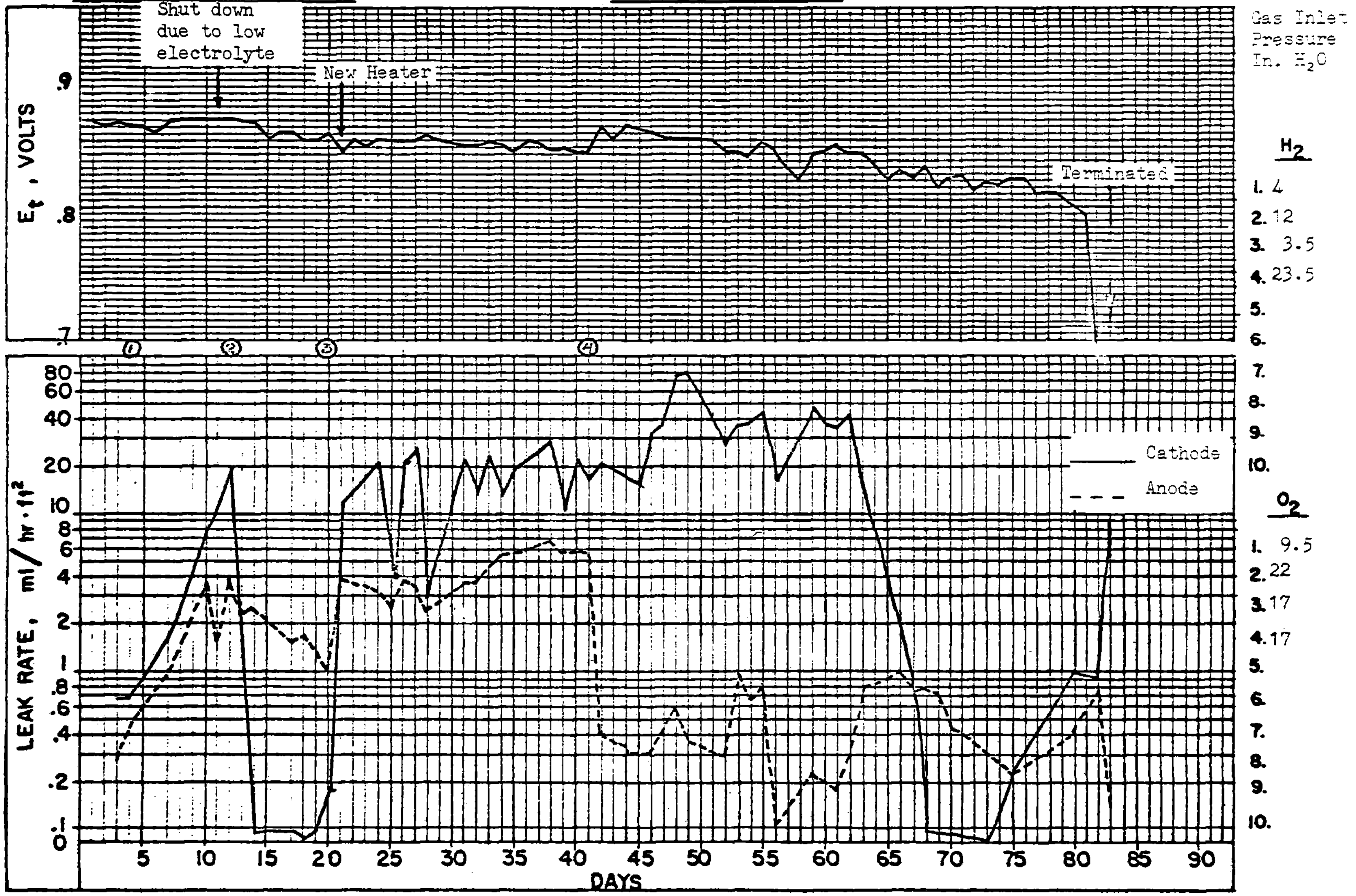


Figure A-16 - Polarization Curves - Cell A-116

STACK NO.: A 212
TYPE: 101
CATHODE: Lab-40-B-II
ANODE: UCC T-2

CURRENT DENSITY: 200 ASF
AVG. TEMP.: 90°C
AVG. NORMALITY: 14N
RACK POSITION: C-1

PRESSURE LEVEL: 15 psia



D-4315

Figure A-17 - Voltage and Leak Rate Versus Time on Load - Cell A-212

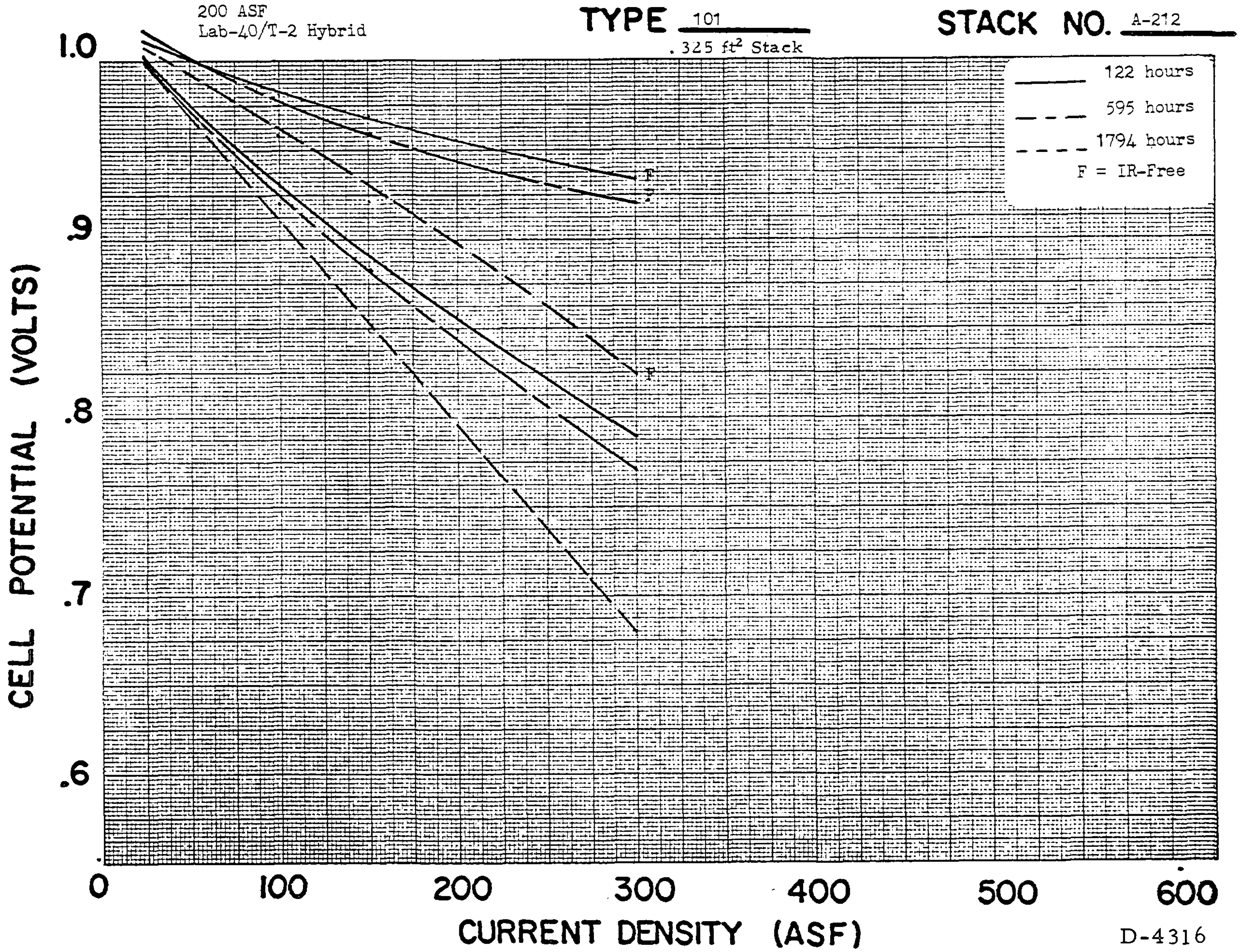


Figure A-18 - Polarization Curves - Cell A-212

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