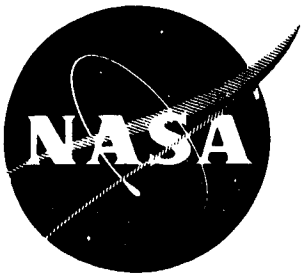

NASA CR-73110
FINAL TECHNICAL EVALUATION
REPORT SM-49109-F

# DEVELOPMENT OF ONE AMPERE-HOUR HEAT STERILIZABLE SILVER-ZINC CELL

PERIOD ENDING JUNE 1967

prepared for

## NATIONAL AERONAUTICS & SPACE ADMINISTRATION
## AMES RESEARCH CENTER

CONTRACT NAS 2-3819

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# DEVELOPMENT OF ONE AMPERE-HOUR HEAT STERILIZABLE SILVER-ZINC CELL

by

A. Himy

prepared for

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July 1967

CONTRACT NAS 2-3819

NASA Ames Research Center
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Douglas Aircraft Company
Newport Beach, California

## ACKNOWLEDGMENT

Acknowledgment is hereby given to Mrs. F. Ruth Sheridan for her full time efforts and valuable contribution to this program.

# CONTENTS

## FIGURES

## TABLES

## 1.0 OBJECTIVES

The prime objective of the program is to design, fabricate, test, and evaluate a primary heat-sterilizable unit cell for space mission applications. The cell must be capable of one recharge and discharge after heat sterilization.

Following is a tentative set of specifications around which the unit cell was to be designed.

Nominal Voltage: 1.5 V

Nominal Capacity: 1 Ah

Load Profile: 3 mA for 72 hours followed by 2 A for two minutes. (Capacity required: 0.30 Ah)

Temperature range during transit and operation: $50^{\circ}F$ to $85^{\circ}F$

Storage temperature in discharged state: $0^{\circ}F$ to $125^{\circ}F$

Sterilization: As per JPL Specification XSO-30275-TST-A

Operating Life: 1 year

Design Objective: The cell shall be sealed during sterilization, transit, and operation.

Environmental Requirements:

1. Shock

   The cell shall withstand three shocks of 18 G's in each direction along each of three mutually perpendicular axes (18 shocks). The wave shape and associated time duration of the input pulse will be one of the following:

   A. Triangular pulse at 10 milliseconds time duration.

   B. Half-sine pulse at 8 milliseconds time duration.

   C. Rectangular pulse at 5 milliseconds time duration.

2. Vibration

   A resonant survey test shall be run on the cell to determine the resonant modes of the cell structure. A sweep shall be made from 5 to 16 cps at 0.368 inch DA and 16 to 200 cps at 5 G peak on each of three mutually perpendicular axes. Measurement shall be made at the point of coupling between the exciting mechanism and the cell. A notation shall be made of all resonances in each direction.

3. Acceleration:

   The cell shall withstand accelerations as shown in the following schedule:

A. 7 G's acceleration for 5 minutes along the longitudinal axis in a direction corresponding to the lift-off of the transporting vehicle.

B. 3 G's acceleration in the opposite direction for 5 minutes duration.

C. 4.5 G's acceleration in both directions along mutually perpendicular axes for 5 minutes duration.

At the end of the program, five unit cells will be delivered to NASA. A parametric study of the current-voltage-temperature relationship will be performed to provide NASA with information about the capability of the cells for a variety of other space missions.

The unit cells will be delivered wet, discharged and sealed, but not sterilized. The sterilization will be performed by NASA prior to use, as per JPL Specification XSO-30275-TST-A, any time within the year following the delivery of the cells.

The cells may be assembled in any configuration desired by NASA to obtain the voltage necessary for the mission. An optimized battery design study will be performed at the end of the program.

## 2.0 SUMMARY OF RESULTS

The program covered a feasibility study on the ability of silver-zinc primary cells to withstand heat sterilization for space missions.

The one-Ah cell was chosen as a test vehicle and the mission profile was tentatively set at a low drain with one high-rate pulse.

The development work involved two distinct but concurrent tasks — mechanical and electrical.

The mechanical problems concerned the terminal and cover-to-case seals. For this reason, two designs were tried. The first one was a cylindrical configuration using a metal case, metal cover and a ceramic-to-metal seal. The cover is welded or soldered to the case. The seal was maintained throughout the entire sterilization. It showed the possibility of using this approach, if necessary.

The second one followed conventional lines, using a rectilinear configuration. The major part of the program was devoted to this approach in agreement with the NASA Project Monitor. Heat resistant plastic materials (polysulfone and polyphenylene oxide) were investigated regarding their sealing capability by heat welding. Two methods were used — ultrasonic and hot gas welding. Both methods showed a strong possibility.

The electrical development covers the cell pack design and its performance evaluation after heat sterilization. After selecting the proper sealant for the separator edges, the design consisted of two positive electrodes and one negative electrode restricted in a cavity made with two rigid inorganic separators and sealed on three edges and partially on the top.

The cell was evaluated under various conditions after sterilization: (1) various discharges ranging from 0.1 A to 2 A at temperatures ranging from 32°F to 125°F; (2) continuous cycling at 1/2-hour charge, 1/2-hour discharge (although a primary, the cell was capable of cycling an average of 200 times); (3) several cells were submitted to shock, vibration, acceleration and survived; (4) cells were left on charged wet stand and discharged for various lengths of time up to five months. Capacity loss was negligible.

This study showed the possibility of using an inorganic separator in the design of a heat sterilizable silver-zinc cell. Voltage and power required by the mission can be obtained through the selection of a custom-made separator for a specific mission.

## 3.0 ESTABLISHMENT OF DESIGN

During the first part of the program, the two following design concepts were considered concurrently because of the difficulty in maintaining reliable seals (terminal-to-cover and case-to-cover) under sterilization conditions.

a. A rectilinear configuration using a plastic case and cover to be joined by different methods — solvent, epoxy cement, heat welding (ultrasonic or hot gas).

b. A cyclindrical configuration using a metal case and a ceramic terminal seal, the cover being welded to the case.

### 3.1 Rectilinear Configuration

The basic components considered are succinctly described.

#### 3.1.1 Separators

Two inorganic separators, 3420-09 and 3420-25, were considered because of their inertness in concentrated potassium hydroxide solution at 145°C for very long periods of time.

The specifications were tentatively set as follows:

Dimensions

| | |
|---|---|
| Length: | 2.030 ± 0.010 inch |
| Width: | 1.900 ± 0.010 inch |
| Thickness: | 0.026 ± 0.001 inch |

Weight

4.0 ± 0.1 grams

Water Absorption (ratio of water pick-up, filling the pores, to the dry weight)

9.5% ± 1%

Apparent Density

2.40 ± 0.1 $g/cm^3$

#### 3.1.2 Interseparators

Interseparators are materials used on either the positive or the negative electrode or both because of their wetting characteristics, i.e., their ability to retain electrolyte and to keep the electrode constantly wetted.

They also serve as cushions between the relatively rigid components (the inorganic separator and the silver electrode).

The following materials were considered and screen-tested:

1. Polyamide fiber felt (Pellon Corp.)
2. Treated nylon fabric (Gelman Acropor Type AN 450)
3. Polypropylene SM-91 (Kendall Mills)
4. Polypropylene EM-476 (Kendall Mills)
5. Potassium Titanate fiber sheet (KT) (duPont)
6. Asbestos fiber sheet (Johns-Manville)
7. Astroflex 4561-7 (Douglas Aircraft)

The latter is an organic-inorganic composition previously developed by Astropower and applied as a coating on the electrode.

### 3.1.3 Electrodes

The active materials and construction types of the electrodes have been kept identical to those used in previous Astropower work. Electrodes were manufactured to the following specifications for all work performed on this program.

#### 3.1.3.1 Positive

| | |
|---|---|
| Material: | Silver powder Silpowder 130 from Handy and Harmon. |
| Grid: | Silver expanded metal 3 Ag 10-3/0. |
| Lead: | Three silver wires (0.016" diameter) spot-welded to grid. |
| Size: | 1.500" x 1.500" (±0.005"). |
| Area: | 14.5 $cm^2$ |
| Thickness: | 0.025" ±0.001" |
| Weight of Active Material: | 4.1 ±0.1 g |

#### 3.1.3.2 Negative

| | |
|---|---|
| Material: | Mix of 98% ZnO and 2% HgO. |
| Grid and Lead: | Same as positive. |
| Size: | 1.500" x 1.500" (±0.005"). |

| | |
|---|---|
| Area: | 14.5 $cm^2$ |
| Thickness: | 0.075" ±0.002" |
| Weight of Active Material: | 7.0 ± 0.1 g |
| Supporting Material: | KT-20 (20 mil thick potassium titanate fiber sheet pressed into the plate to a thickness of about 10 mils). |

### 3.1.4 Cell Assembly

The preliminary cell assembly consists of two positive and one negative electrode. The negative electrode is sandwiched between two inorganic separators, and the edges are sealed resulting in a wafer configuration where the negative is tightly enclosed in a cavity. The silver electrodes are placed on each side of the negative wafer (Figure 1). All preliminary electrical and sterilization tests were conducted on cells of this type.

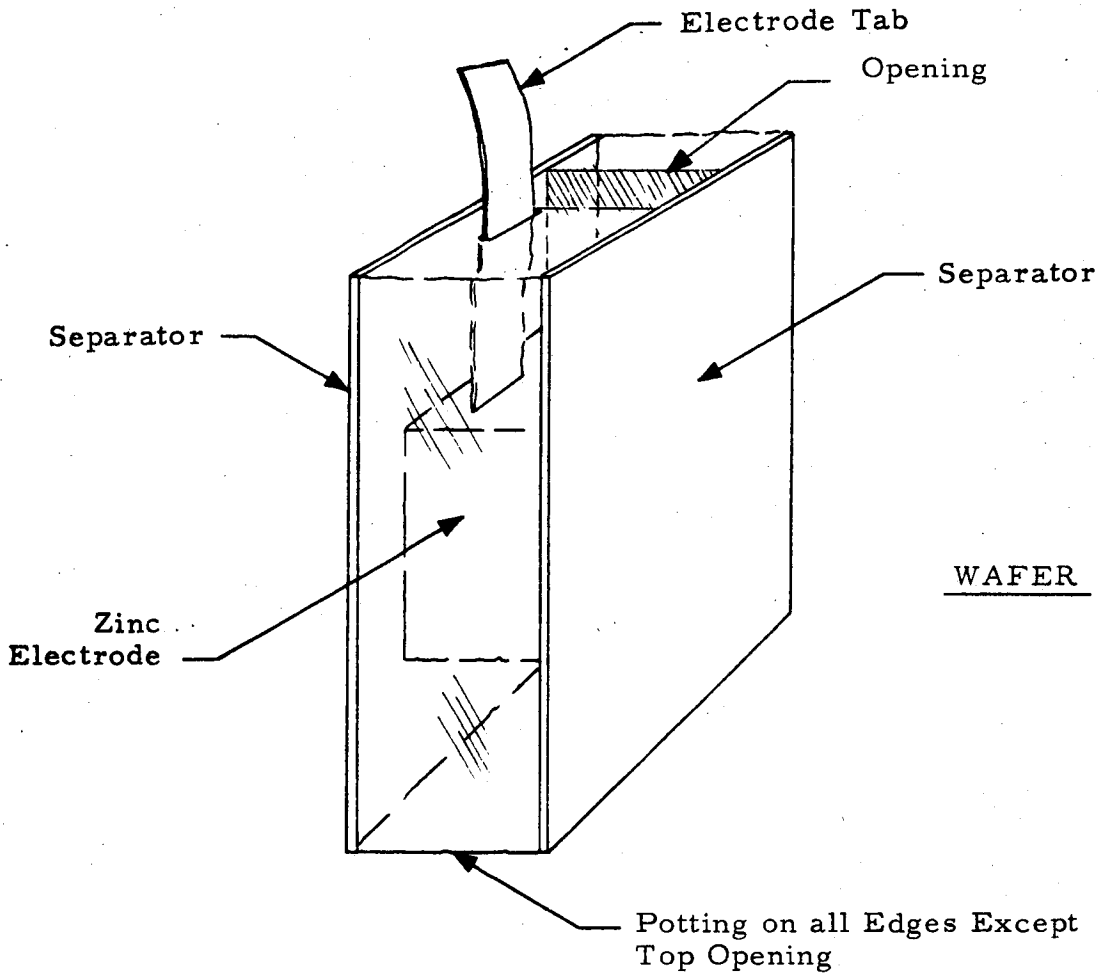
It may be of interest also to use one positive and two negative electrodes, where a large excess of zinc would be more advantageous in avoiding any possibility of zinc limiting charge, producing undesired hydrogen gassing and subsequent pressure build-up, or limiting discharge after prolonged wet stand. The voltage characteristics of the cell will not change, since the current density remains constant; the single silver plate may be designed to provide at least the one-Ah capacity required.

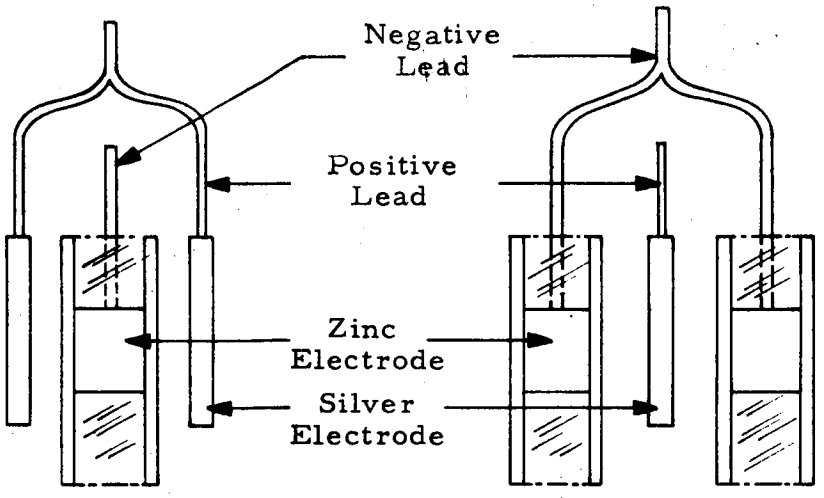
### 3.1.5 Case and Cover

For the sake of convenience and expediency, temporary design of case and cover was established and parts were fabricated from polysulfone flat stock material, grade P-1700 (Union Carbide).

Figure 2 shows a photograph of case and cover with terminals installed. The center hole in the cover is for electrolyte filling and for attaching a pressure gage. However, molds for case and cover will be eventually required. Prime candidate materials are polysulfone P-1700 (manufactured by Union Carbide) and polyphenylene oxide PPO (manufactured by GE) of various grades.

During the course of the program, molded 5-Ah cases and covers available from another NASA Contract[1] were used with a flat polysulfone shim to manufacture cells of the selected design to run all mechanical and electrical tests.




Wafer Assembly

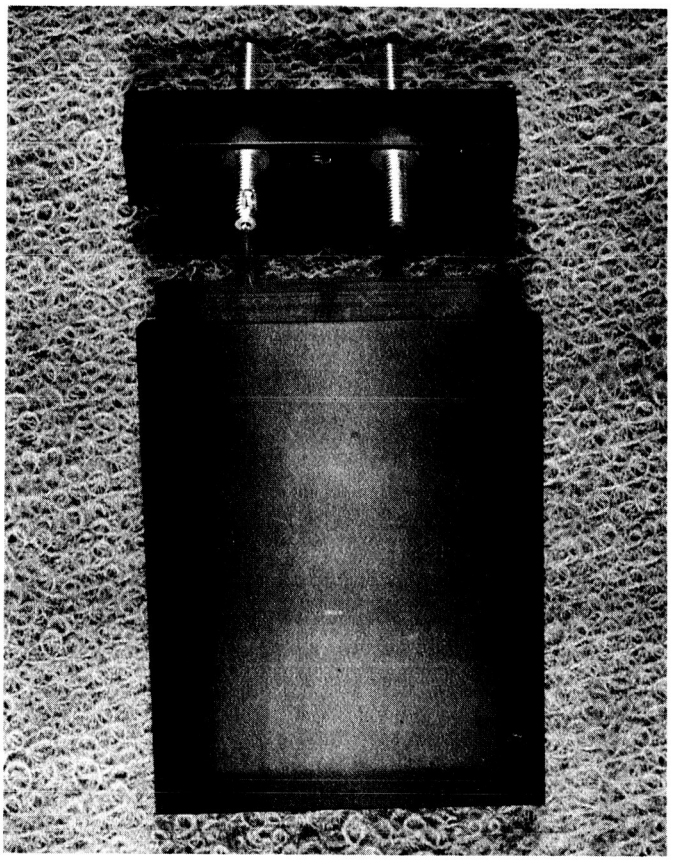
2 Positives
1 Negative

Wafer Assembly

1 Positive
2 Negatives

(320)

Figure 1. Wafer Concept



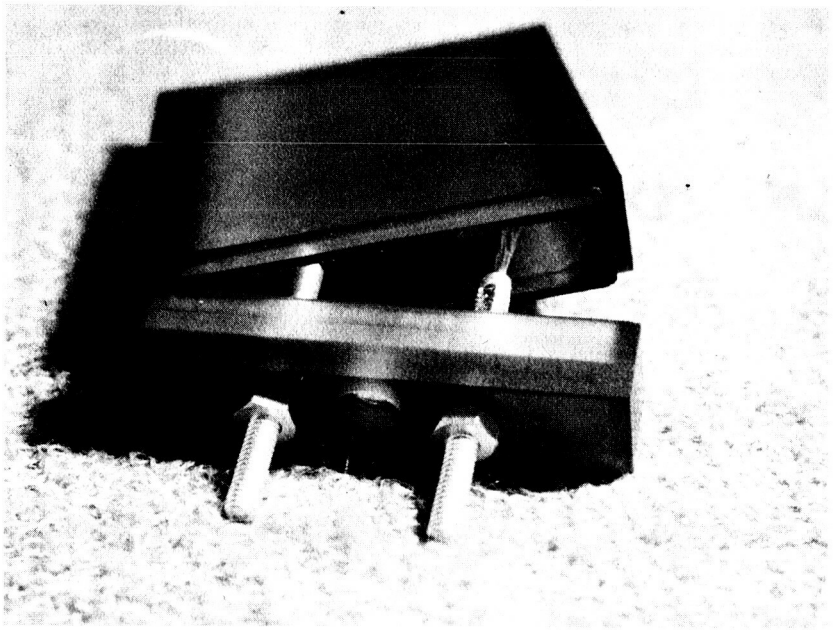


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Figure 2. Case and Cover Assembly Showing One Terminal Crimped With Six Silver Wires

### 3.1.6 Data

This approach was selected for evaluation. All the development work is described in Sections 4.0 through 7.0.

## 3.2 Cylindrical Configuration

In light of the lack of material and molded cases and covers of the proper size and design at the beginning of the program, and in anticipation of the problems involved in sealing a rectilinear configuration, a parallel course was set for a cylindrical case.

The crucial part of the assembly is a separator cup made of inorganic material having the same composition, wall thickness and porosity as the flat inorganic separator.

The case is made of metal or Teflon while the covers are made of Teflon, metal, or both.

### 3.2.1 Case and Cover Design

Several designs of cases and covers are shown in Figures 3 through 8. They vary only in the cover seal configuration.

The first five models feature a closed bottom cylindrical case, made of silver plated soft brass, which has the negative polarity and serves as a current collector.

Designs #1 and #4 (Figures 3 and 6) have a Teflon cover screwed on the metal case. Here a terminal seal (positive polarity) is also needed.

Designs #2 and #3 (Figures 4 and 5) do not present a terminal seal problem since the positive leads can be soldered to the metal cover. A threaded Teflon insulator, screwed on the case and cover, seals both the case and cover threads.

Design #5 (Figure 7) features a welded metal cover with a ceramic seal around a center terminal.

The design #6 (Figure 8) is an open-end Teflon cylinder with circular grooves provided on the walls of both ends for an "O" ring seal.

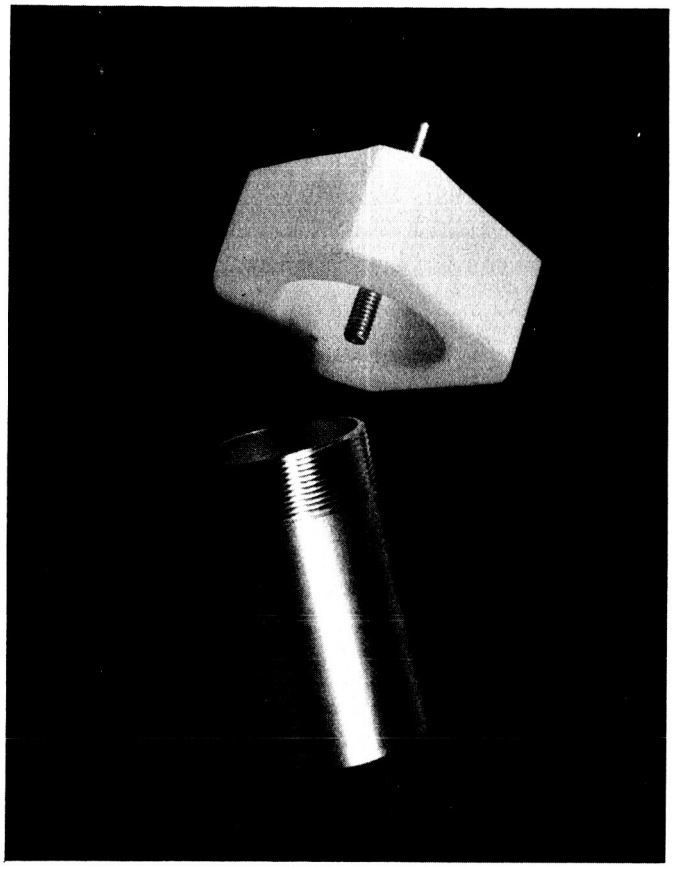

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Figure 3. Cylindrical Cell, Design #1

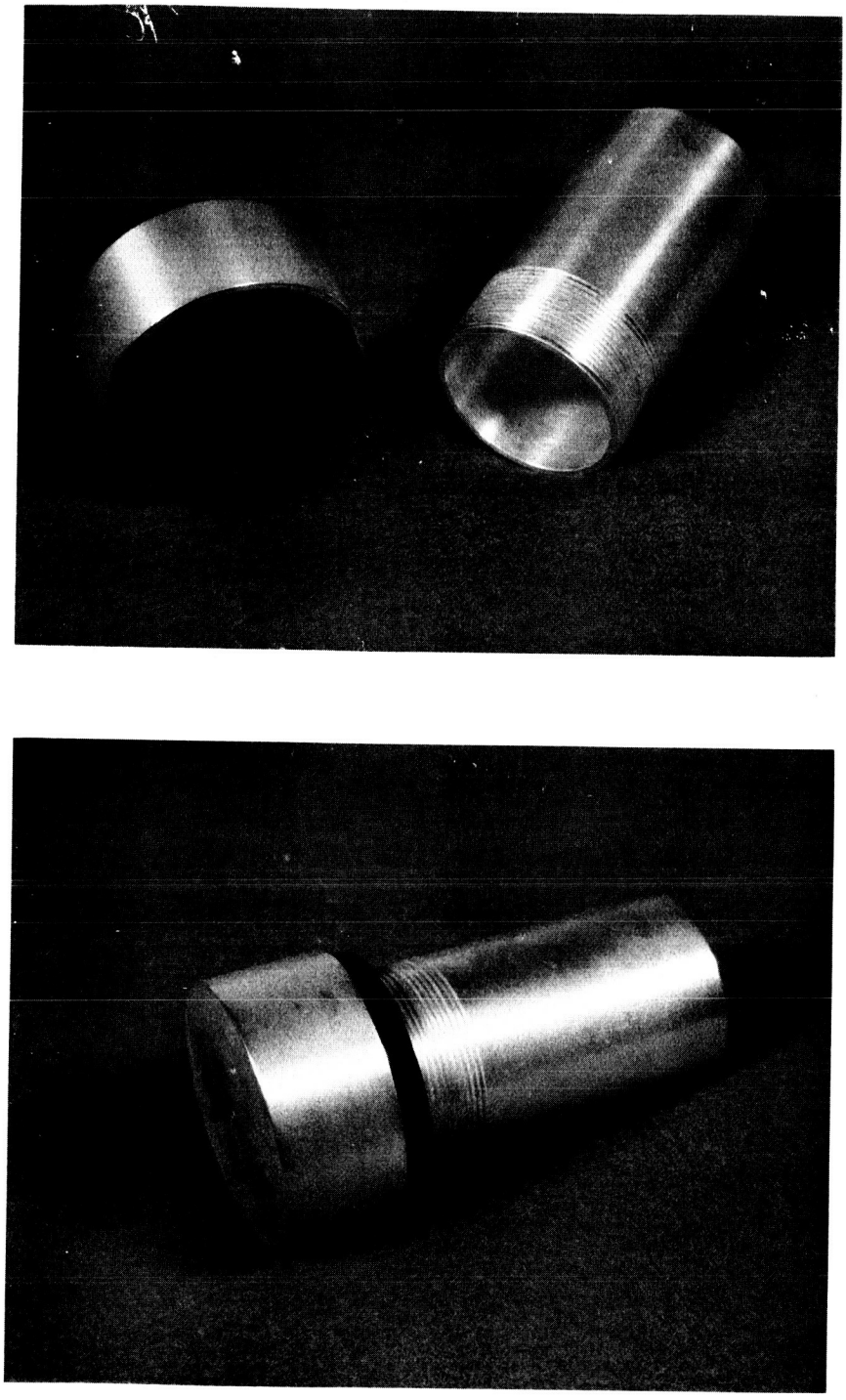

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Figure 4. Cylindrical Cell, Design #2

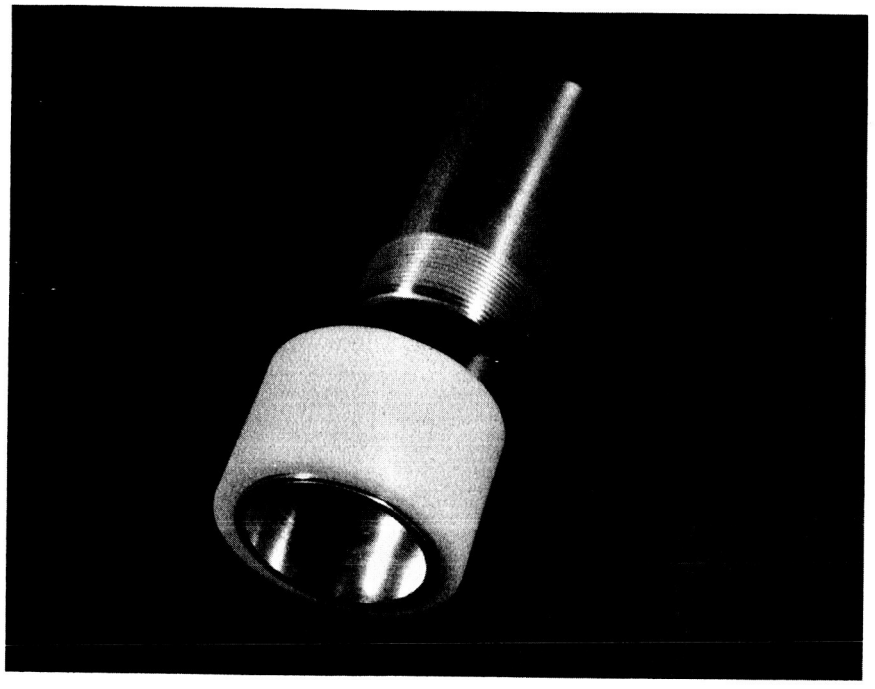

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Figure 5. Cylindrical Cell, Design #3

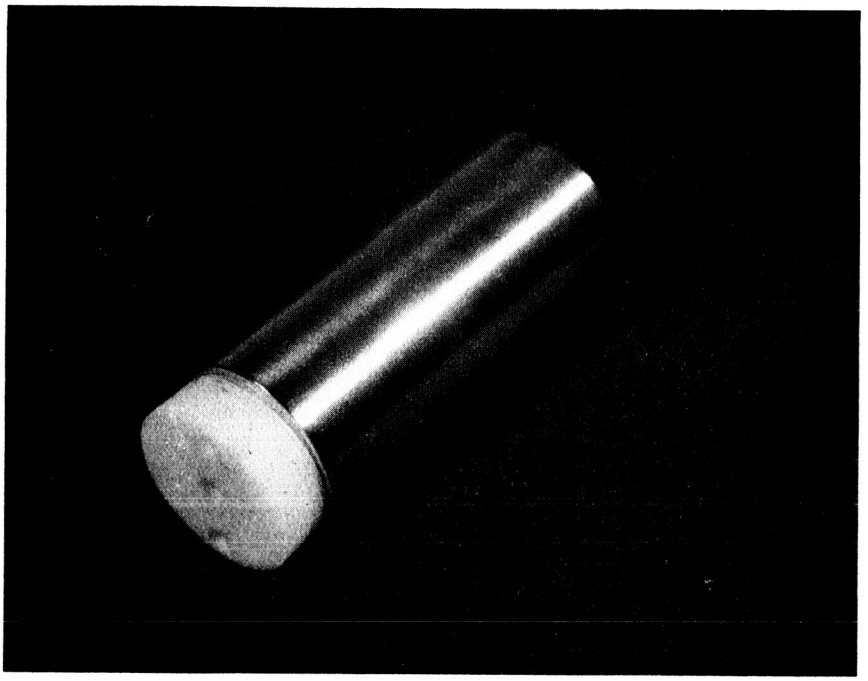

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Figure 6. Cylindrical Cell, Design #4

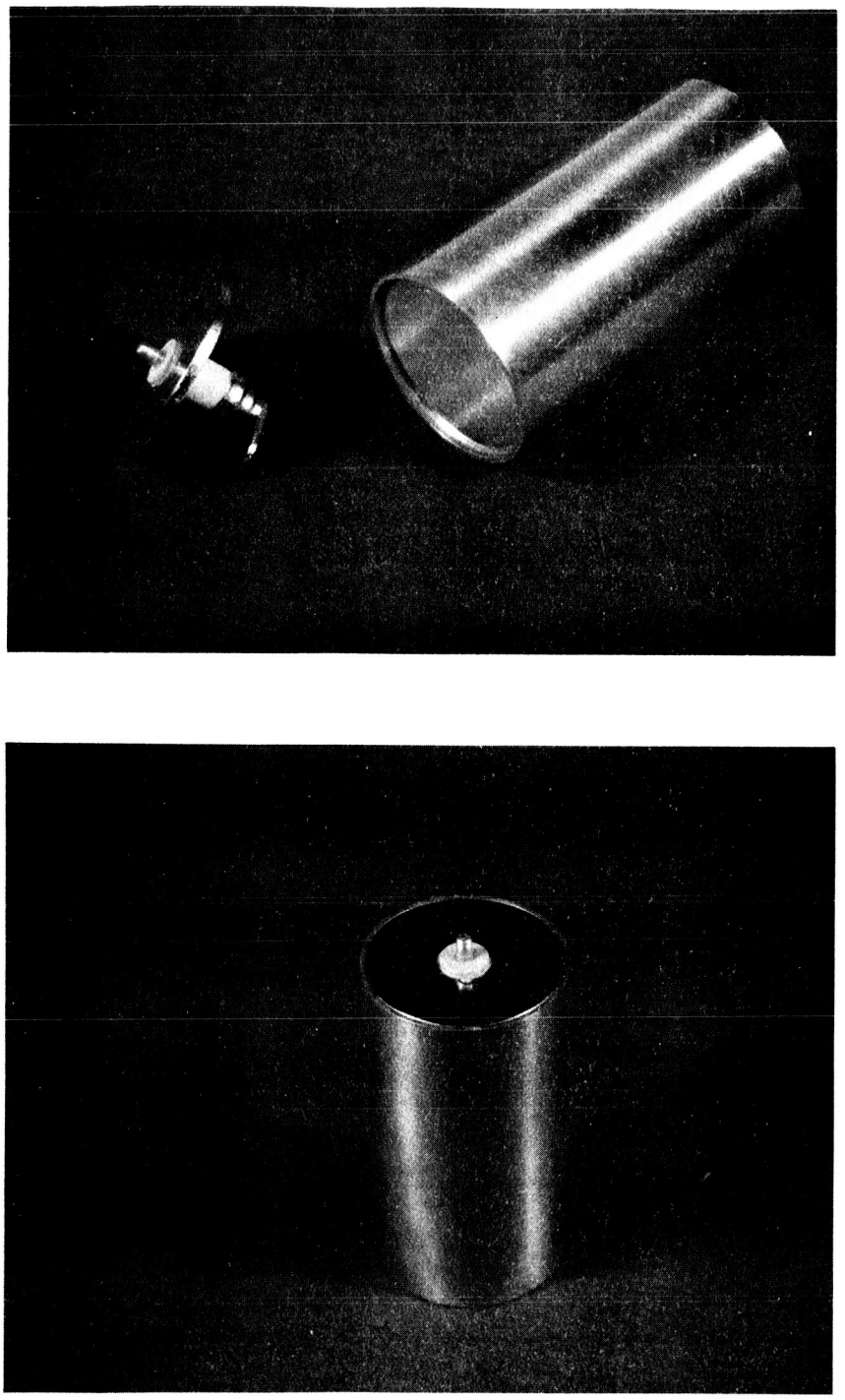

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Figure 7. Cylindrical Cell, Design #5

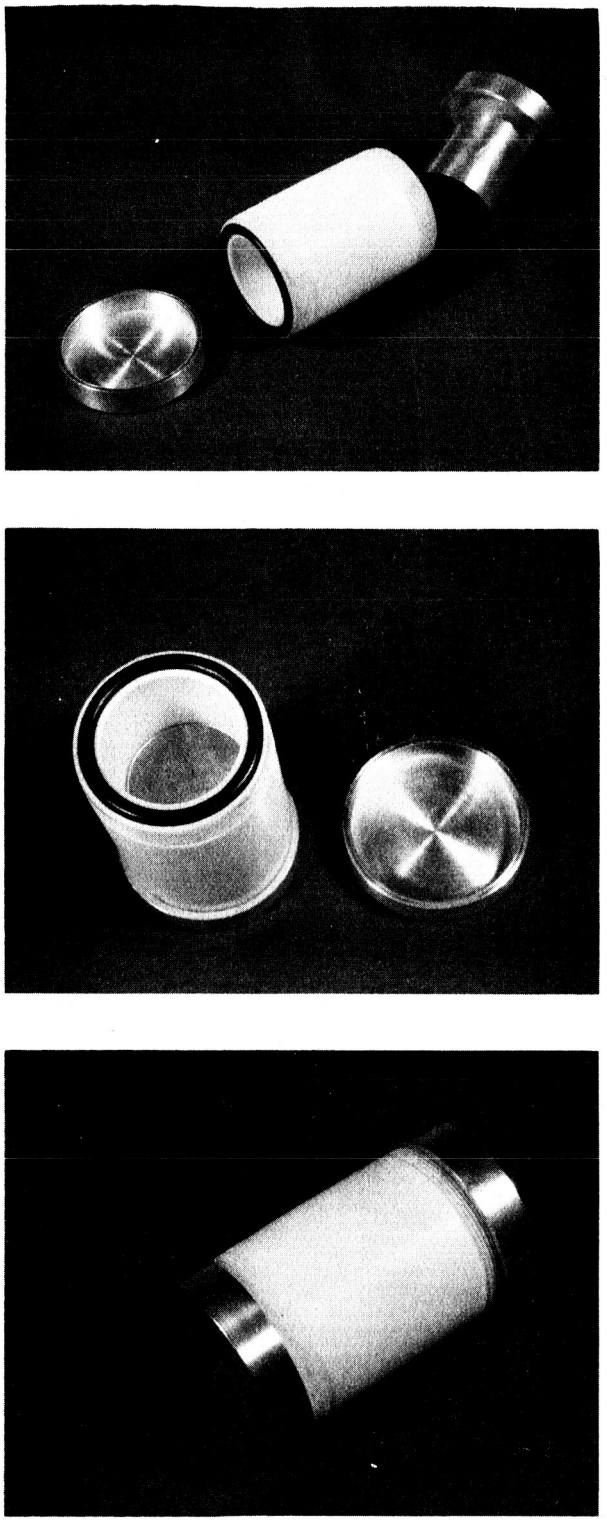

Figure 8. Cylindrical Cell, Design #6

a metal cap is screwed on each end, thus tightening the "O" ring. As an improvement, a silver collector is welded to the bottom cap which is the negative polarity. The silver collector is circular and fits in the Teflon cylinder snugly, thus providing a large area for the current collection.

### 3.2.2 Cell Design and Assembly

The design approach consists of a separator cup which contains the silver electrode and is placed at the center of the cylindrical case (Figure 9). The positive plate is a cylindrical slug of silver powder of the proper design parameters, pressed around a silver collector. The silver wires are attached to the positive terminal in the cover (if plastic), or soldered to the cover itself (if metal).

As an improvement, KT material is added around and on the top of the silver slug.

### 3.2.3 Separator Cup

A typical separator cup is shown in Figure 10. The cups are made of the same inorganic materials used in the flat separator configuration. Porosity and thickness were kept identical so that the electrical area resistivity in KOH remains as close as possible to the one obtained for flat separators. The lateral area exposed to the passage of ions was arbitrarily selected to be 19 $cm^2$ until electrical polarization data could be obtained as well as mechanical data relative to the best sealing approach.

A cup was submitted to the sterilization procedure by itself at 145°C for 108 hours. No visual damage was noted.

### 3.2.4 Data

This approach was not selected for final evaluation; all data pertinent to mechanical and electrical testing may be found in the Appendix.

## 3.3 Comments

As required by the contract, a mid-term, informal oral presentation was made at NASA-Ames Research Center, to report on the status of the technical effort and to discuss the various alternatives encountered in

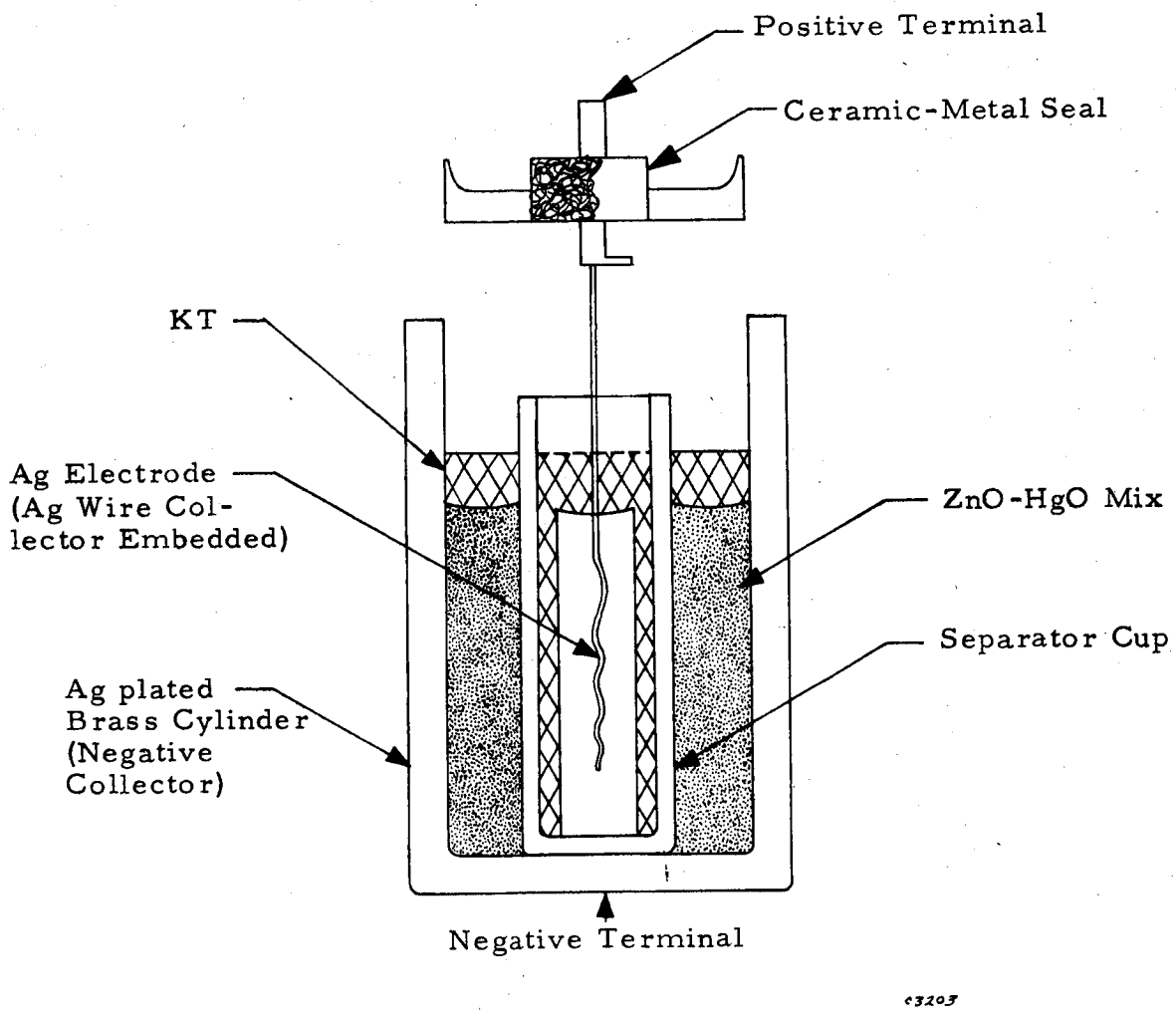


Figure 9. Typical Cylindrical Cell Assembly

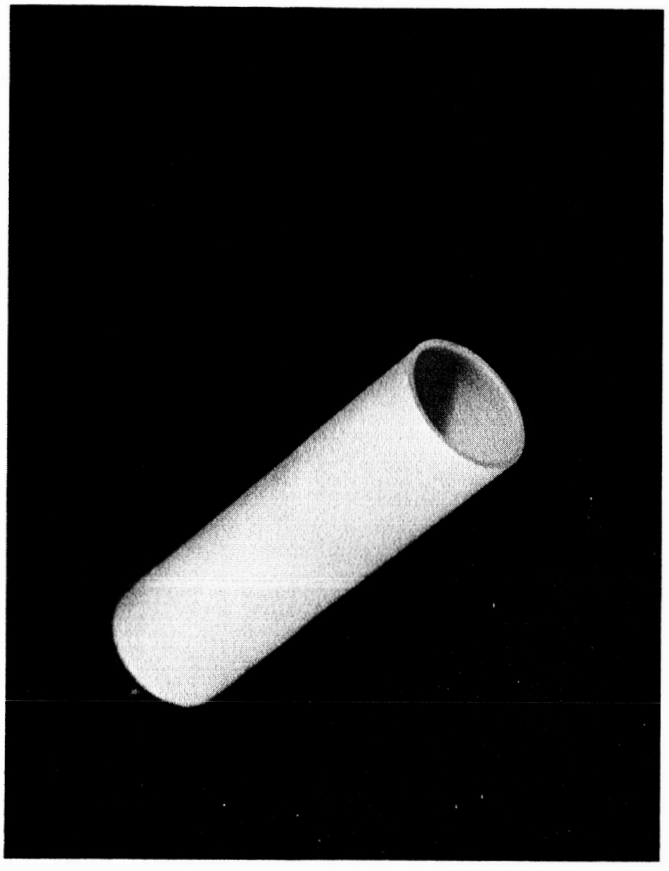

Figure 10. Separator Cup

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order to concentrate on one design and obtain the maximum information on that design for the remainder of the contract.

Because of the cost and long delivery of the ceramic-to-metal seals, the consensus was not to pursue the cylindrical configuration any further. The positive seal demonstrated in this case could be used in a rectilinear configuration if a rectilinear metal case would be needed in the future. On the other hand, the cover-to-case seal in the plastic case requires that the two parts, cover and case, be molded in molds properly and purposely designed for polysulfone or PPO. Purchasing such a mold is beyond the scope of this program so the consensus again was to employ existing molded plastic cases available from another NASA contract[1] designed for five ampere-hour cells. These were to be used with shims to reduce the internal volume of the case to that needed by the actual cell pack tested on this program. Thus, the cover-to-case sealing would be done on properly designed parts.

## 4.0 EFFECT OF HEAT STERILIZATION ON CELL COMPONENTS

A series of tests was run as a screening procedure to determine the capability of all cell components to withstand heat sterilization. To save time by quickly eliminating any component that might be adversely affected by prolonged periods in hot KOH, it was decided that a continuous 108-hour period at 145°C in an air ambient with a short temperature rise and fall period (2 to 2-1/2 hours) would be far more severe than the required three 36-hour periods in a dry nitrogen environment, with temperature rise and fall not to exceed 30°C/hour, as called out in the JPL Specification XSO-32075-TST-A of 8/24/63, modified 8/23/65.

Consequently, all individual parts were heat sterilized at 145°C for 108 hours, plus 4 to 5 hours spent during temperature rise and fall. Figure 11 shows a typical temperature profile.

After sterilization each part was examined closely and all active components were assembled with fresh counterparts and tested against controls.

The test set-up was as follows: Each item was separately placed in 30% and 45% KOH in a polysulfone case and sealed into a stainless steel pressure vessel monitored with a pressure gauge and an inside thermocouple. The pressure vessel was placed in an air-circulation drying oven and the temperature monitored to within ± 3° to 145°C. Figure 12 is a photograph of the test set-up. Each component was then evaluated separately.

### 4.1 Cases and Covers

Initial tests were carried out on polysulfone P-1700 to determine the compatibility of the material in 30% and 45% KOH under the heat-sterilization conditions (145°C for 108 hours).

Molded cases (5 Ah-size) used on NASA contract NAS 3-7639 were indirectly tested. The components described in the next paragraphs were sterilized in polysulfone cases placed in sealed steel pressure chambers. This procedure allowed us to submit several cases to sterilization at the same time. No thermal degradation was observed, although some cases have spent more than 400 hours at 145°C from repeated use in different sterilization set-ups.

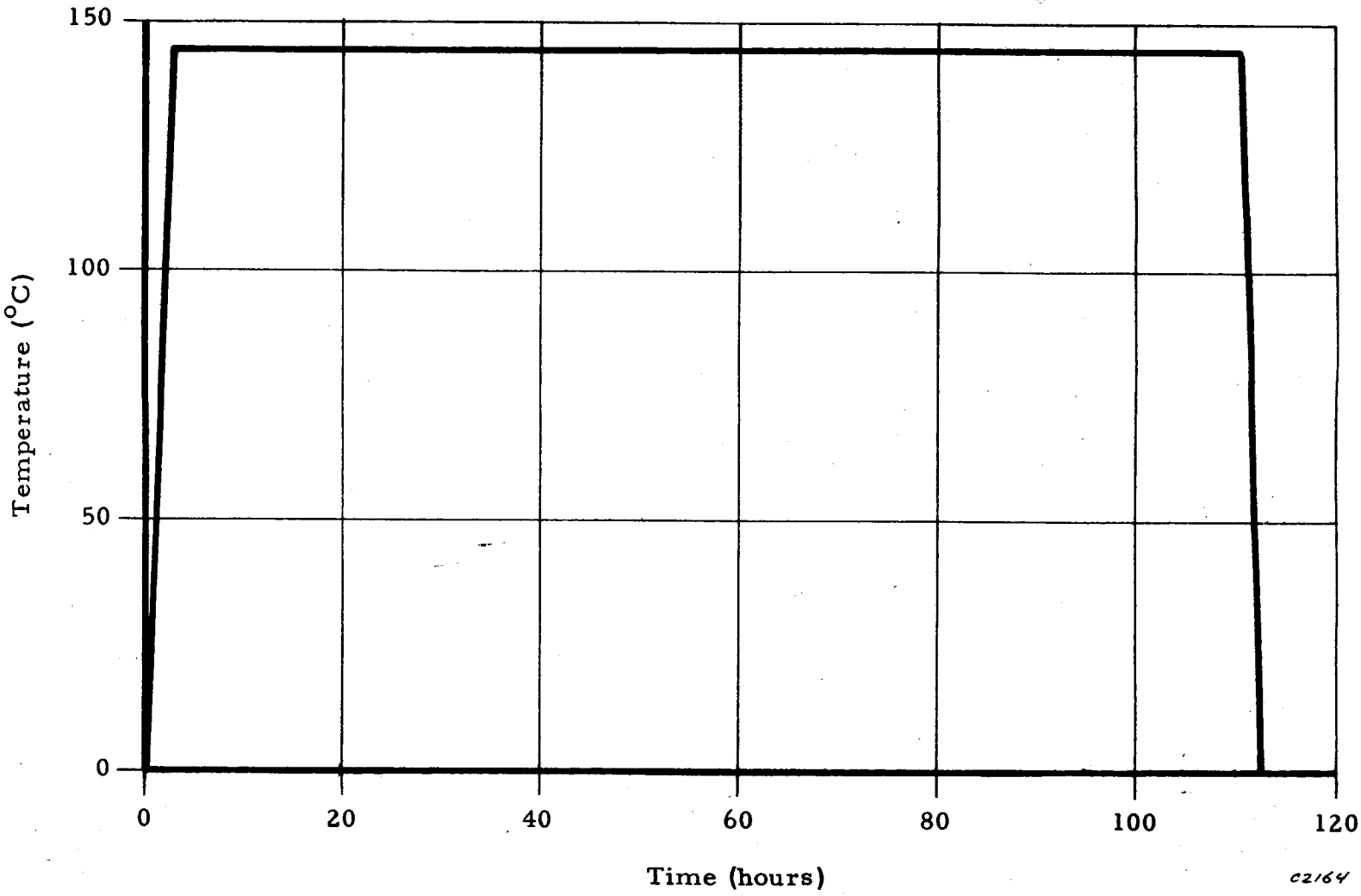


Figure 11. Heat Sterilization Temperature Profile Used for Screening Tests

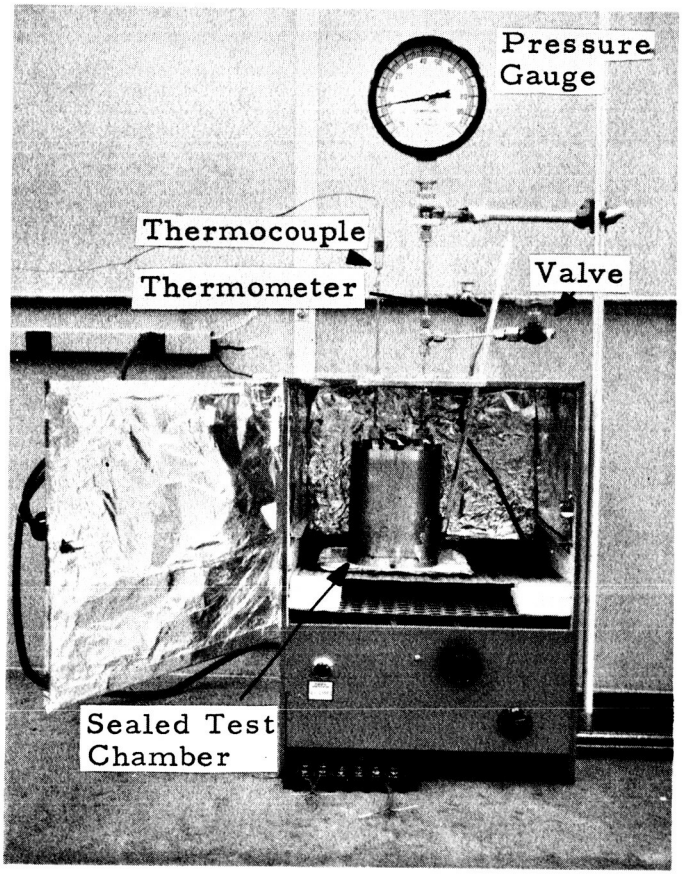


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Figure 12. Oven Set-Up

## 4.2 Inorganic Separators

### 4.2.1 Manufacture

Twenty separators were selected at random from several different manufacturing batches and checked for dimensional, weight, and absorption tolerance to specifications. Table I is the result of this check. All separators were within the required tolerances.

### 4.2.2 Sterilization Test

Five of the above separators were sterilized for 108 hours at 145°C in 45% KOH, then rechecked to see if there was any change in their weight, dimension, or absorption. The results after sterilization are summarized in Table II. All figures are still within the specification tolerances. There were no significant changes in dimension, weight, density, or absorption.

## 4.3 Interseparator Materials

The materials listed in Section 3.1.2 were submitted to the sterilization procedure in 30% KOH, 145°C for over 108 hours.

Polyamide fiber felt (Pellon) and Gelman Acropor disintegrated upon sterilization and were deemed unsuitable.

The most promising materials are listed in Table III.

Comments: An interesting factor to consider is the capability of the material to retain KOH after the sterilization procedure. In this respect, both polypropylene SM-91 and EM-476 seemed to have increased their wettability.

KT and asbestos were still intact, but too fragile to be weighed or washed free of alkali.

Another aspect is the pressure generated during sterilization. As expected, organic materials build up higher pressure than the inorganic materials.

Also noteworthy is the fact that the polypropylene SM-91 produces quite a considerable amount of carbonation in the electrolyte.

Special Treatment: The polypropylene materials undergo a certain amount of shrinkage when immersed in KOH at 145°C. It was found to be desirable to pre-shrink them before the sterilization procedure.

## TABLE I

## TOLERANCE TO SPECIFICATIONS CHECK OF RANDOM SAMPLES OF SEPARATORS

| | Sep. No. | Length (inches) | Width (inches) | Thickness (mils) | Volume (cc) | Mass (grams) | Apparent Density (g/cc) | % Absorption (water pickup) |
|---|---|---|---|---|---|---|---|---|
| Specs | | 2.030 ± 0.010 | 1.900 ± 0.010 | 26 ± 1 | 1.65 ± 0.10 | 4.0 ± 0.15 | 2.4 ± 0.25 | 9.5 ± 1 % |
| | 1 | 2.0275 | 1.899 | 26.5 | 1.67 | 4.04 | 2.42 | 9.1 |
| | 2 | 2.023 | 1.897 | 26.5 | 1.67 | 4.01 | 2.40 | 9.4 |
| | 3 | 2.0235 | 1.900 | 26.0 | 1.64 | 3.92 | 2.39 | 9.6 |
| | 4 | 2.035 | 1.909 | 25.5 | 1.62 | 3.85 | 2.38 | 9.8 |
| | 5 | 2.032 | 1.903 | 26.5 | 1.68 | 4.03 | 2.40 | 9.4 |
| | 6 | 2.036 | 1.909 | 25.5 | 1.62 | 3.89 | 2.39 | 9.6 |
| | 7 | 2.035 | 1.908 | 25.5 | 1.62 | 3 89 | 2.40 | 9.4 |
| | 8 | 2.030 | 1.903 | 25.5 | 1.61 | 3.88 | 2.41 | 9.2 |
| | 9 | 2.030 | 1.905 | 25.5 | 1.62 | 3.87 | 2.39 | 9.5 |
| | 10 | 2.035 | 1.904 | 25.5 | 1.62 | 3.89 | 2.40 | 9.4 |
| | 11 | 2.036 | 1.908 | 26.5 | 1.69 | 4.07 | 2.41 | 9.2 |
| | 12 | 2.034 | 1.907 | 26.5 | 1.68 | 4.01 | 2.39 | 9.6 |
| | 13 | 2.032 | 1.905 | 25.5 | 1.62 | 3.91 | 2.41 | 9.2 |
| | 14 | 2.034 | 1.096 | 25.5 | 1.62 | 3.88 | 2.39 | 9.6 |
| | 15 | 2.030 | 1.905 | 25.5 | 1.62 | 3.89 | 2.40 | 9.4 |
| | 16 | 2.037 | 1.907 | 25.5 | 1.62 | 3.92 | 2.42 | 9.1 |
| | 17 | 2.034 | 1.905 | 26.5 | 1.68 | 4.00 | 2.38 | 9.8 |
| | 18 | 2.031 | 1.902 | 26.5 | 1.68 | 4.00 | 2.38 | 9.8 |
| | 19 | 2.0255 | 1.899 | 25.5 | 1.61 | 3.87 | 2.40 | 9.4 |
| | 20 | 2.025 | 1.898 | 26.5 | 1.68 | 4.03 | 2.39 | 9.6 |

TABLE II

SPECIFICATION CHECK ON FIVE RANDOMLY SELECTED SEPARATORS
HEAT STERILIZED 108 HOURS AT 145°C IN 45% KOH

| | Sep. No. | Length (inches) | Width (inches) | Thickness (mils) | Volume (cc) | Mass (grams) | Apparent Density (g/cc) | % Absorption (water pickup) |
|---|---|---|---|---|---|---|---|---|
| Specs | | 2.030 ± 0.010 | 1.900 ± 0.010 | 26 ± 1 | 1.65 ± 0.10 | 4.0 ± 0.15 | 2.4 ± 0.10 | 9.5 ± 1 % |
| | 1 | 2.0275 | 1.900 | 27.0 | 1.72 | 4.10 | 2.38 | 9.8 |
| | 2 | 2.026 | 1.899 | 27.0 | 1.70 | 3.99 | 2.35 | 10.3 |
| | 3 | 2.0265 | 1.902 | 26.5 | 1.64 | 3.98 | 2.43 | 8.9 |
| | 4 | 2.035 | 1.910 | 26 0 | 1.66 | 3.96 | 2.39 | 9.6 |
| | 5 | 2.034 | 1.905 | 27.0 | 1.71 | 4.08 | 2.39 | 9.6 |

TABLE III

INTERSEPARATOR MATERIALS STERILIZED IN 30% KOH

| Material | KOH Absorption Before Test | Hours At 145°C | Maximum Pressure In Vessel mm Hg | KOH Absorption After Test | Carbonation of Electrolyte | % Area Shrunk |
|---|---|---|---|---|---|---|
| Polypropylene SM-91 pre-shrunk | 15 mg/cm$^2$ | 113 | 3290 | 30 mg/cm$^2$ | considerable | 0 |
| Polypropylene EM-476 pre-shrunk | 9 mg/cm$^2$ | 114 | 3500 | 16 mg/cm$^2$ | slight | 44% |
| Potassium Titanate Sheet (KT) | F | 111 | 3260 | F | slight | 0 |
| Asbestos Sheet | 48 mg/cm$^2$ | 113 | 2260 | F | none | 0 |
| Coating 4561-7 on Ag Plate | 7 mg/cm$^2$ | 130 | – | 7 mg/cm$^2$ | none | 0 |

F = Intact, but too fragile for measurement

The treatment consisted of a soaking in a 45%, KOH solution at 145°C for approximately 40 hours. The samples cut were carefully measured. The data are tabulated below for two samples of each material.

| Material | Original Dimensions | After Treatment | After Sterilization |
|---|---|---|---|
| EM-476 | 27.6 $cm^2$<br>27.0 $cm^2$ | 25.0 $cm^2$<br>25.2 $cm^2$ | 13.40 $cm^2$<br>14.70 $cm^2$ |
| SM-91 | 26.80 $cm^2$<br>25.75 $cm^2$ | 23.25 $cm^2$<br>22.10 $cm^2$ | 23.25 $cm^2$<br>22.10 $cm^2$ |

After treatment, EM-476 shrank by 7 to 9% and SM-91 by 13 to 14%.

After sterilization and comparing to the new post-treatment area, the shrinkage percentage was respectively 48% and 0%.

The physical appearance also showed a difference: whereas the EM-476 became quite stiff, the SM-91 remained soft and flexible.

The SM-91 propylene may be retained as a candidate in further development. However, the KT material has been used extensively in sterilization and electrical tests and did not show any adverse effects. It was kept as a constant on this program.

### 4.4 Sealants

This section deals exclusively with the problem of finding a sealant suitable for bonding the inorganic separators placed on either side of the zinc electrode, thus encapsulating the electrode and making a wafer element as depicted in Figure 1.

Several sealants tested at room temperature in concentrated KOH solution maintained the integrity of the seal for a long time without apparent degradation or chemical attack. At 100°C and higher, the integrity of the seal is a function of the length of time of exposure to alkali and, depending on the material, may vary from days to months. Therefore, it was important to determine if there would be a sufficiently good retention of the seal at the sterilization conditions.

The sealants per se were tested in 45% KOH at 145°C for 108 hours in pressure vessels. They were

1. Unichrome U-218X (M&T Chemicals, Inc.)
2. Chem-O-Sol PK4706 (Plas-Kem Corp.)
3. Uralane (Furane Plastics, Inc.)
4. RTV-102 (G.E.)
5. Si-O-Flex SS-831 (Stauffer Chemicals Co.)
6. Allbond epoxy resin (Allaco)
7. Neoprene Cement (Acorn 724)

The first five broke down. Allbond was mildly attacked and all preliminary work on negative wafers was done with this epoxy. However, during the course of the program, accumulation of data on the neoprene cement, Acorn, showed that this material can withstand heat sterilization in KOH without losing its bonding characteristics. It was exclusively used in the design for final evaluation (Section 7.0).

### 4.5 Silver Electrodes

Unformed sterilized silver electrodes show no visible evidence of any changes due to heat sterilization. The ability to accept charge, the maintenance of open circuit voltage, and discharge capacity were well within the expected performance range of unsterilized control plates, after the sterilized plates were assembled against unsterilized components. Figure 13 shows discharge curves of a sterilized silver electrode and of an unsterilized control.

### 4.6 Negative Wafers

Negative wafers made by sandwiching a negative electrode between two inorganic separators and sealing, in this case with an epoxy resin, showed no effect from the heat sterilization except a tendency for the ZnO to boil out through the small filling hole in the top of the wafer. The loss was small but will be eliminated in the future by packing the top of the wafer with Armalon felt before sealing. Previous experience has proven this to be very effective in containing the ZnO.

Figure 14 shows discharge curves of a sterilized negative wafer assembled with unsterilized counterparts and of an unsterilized control. The

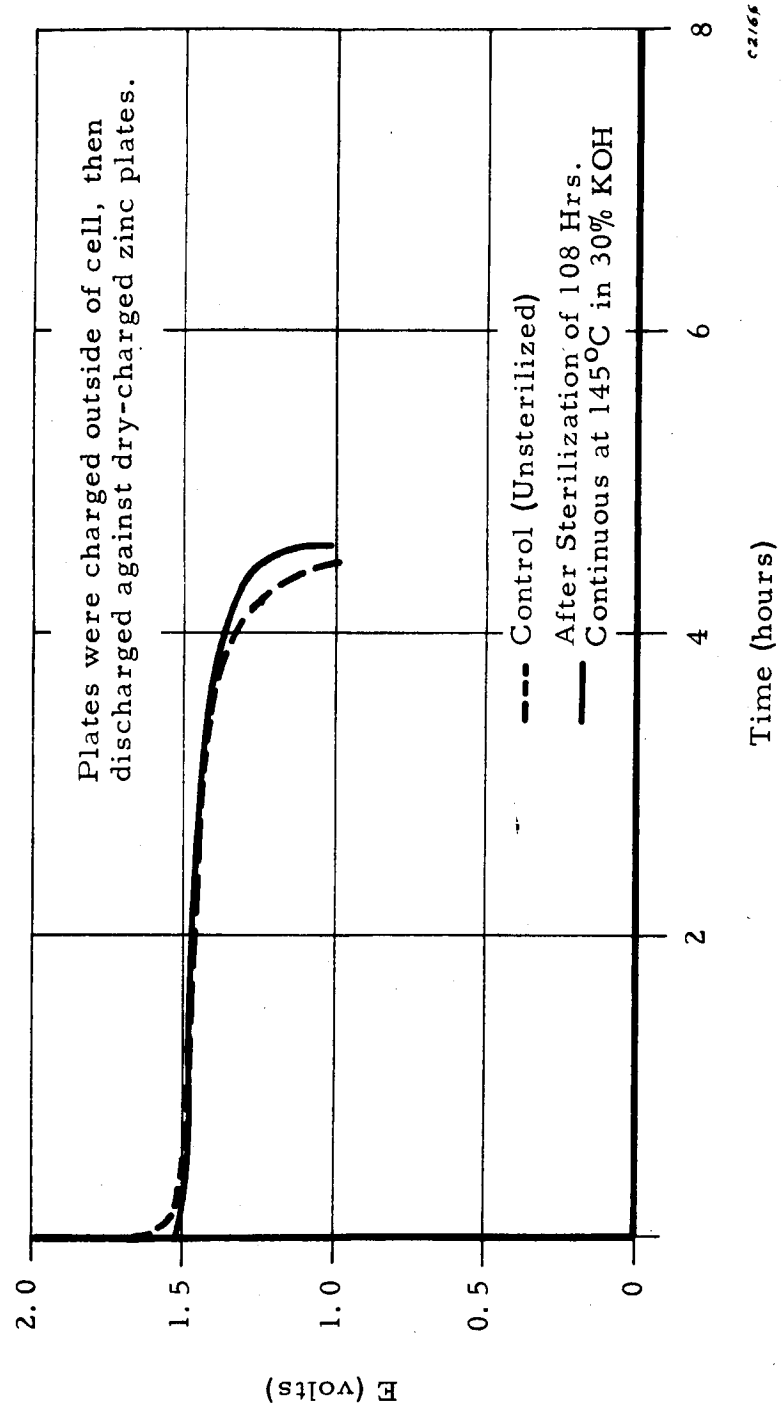


Figure 13. Discharge of Silver Plates at 300 mA (Current Density 10 mA/cm$^2$)

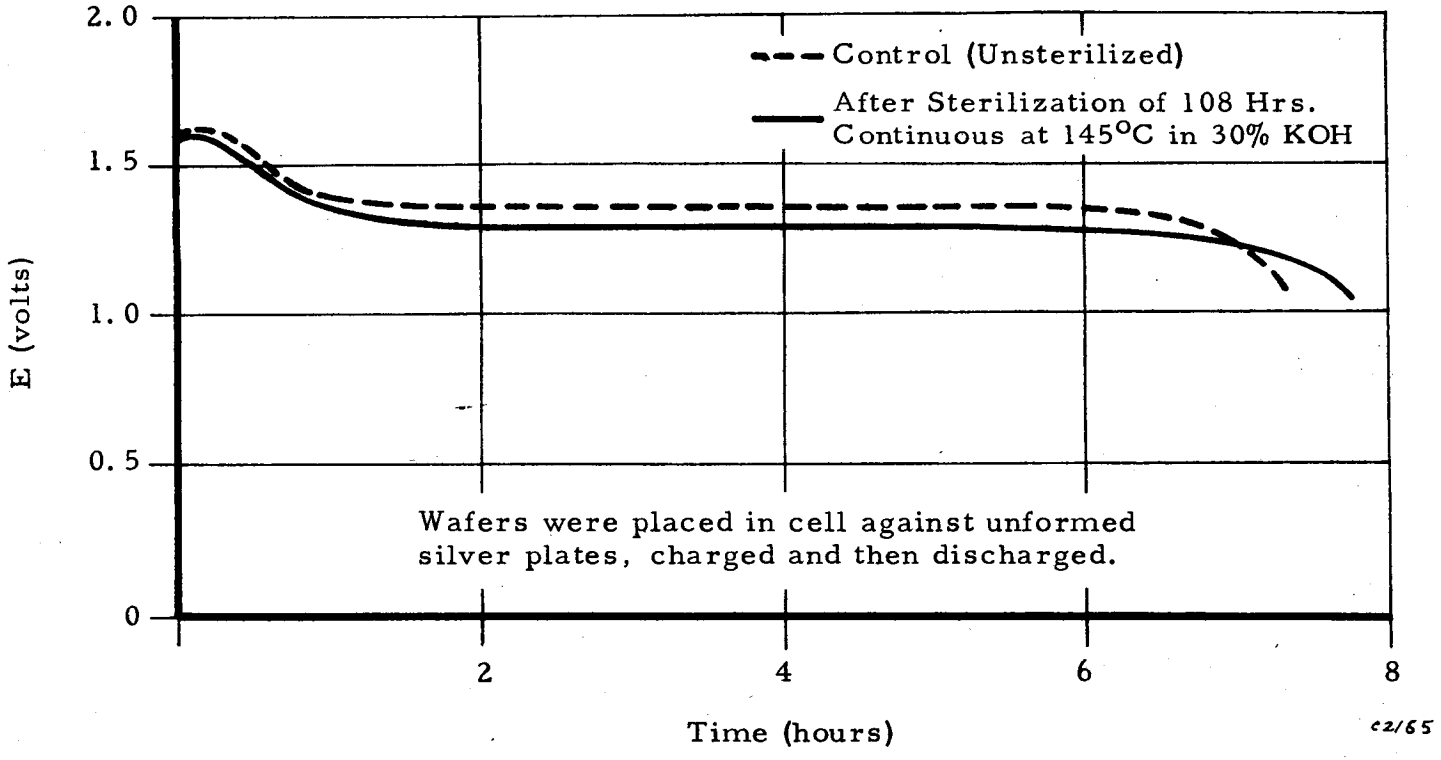


Figure 14. Discharge of Negative Wafers at 300 mA (Current Density 10 mA/cm$^2$)

capacity obtained is comparable to that of the unsterilized wafer, but there is some degradation of the voltage plateau.

## 4.7 Pressure Determination

### 4.7.1 Approach Philosophy

The pressure in the sealed cell during heat sterilization is caused mainly by the water vapor pressure of the alkali solution, the expansion of gases caused by the elevation of temperature, and by gas evolution caused by chemical processes (dissolution of material in electrolyte, reduction, local actions, etc.). If the active materials are taken in their original form (pure silver and pure zinc oxide) the amount of gas evolved due to chemical action is practically nil and can be measured to verify it. The pressure caused by temperature rise can be minimized if maximum evacuation of the cell is done at room temperature before sealing.

The main factor, then, is the vapor pressure. It is known that at a given temperature the vapor pressure decreases as the KOH concentration increases. This compels us to consider the two extreme concentrations, 30% and 45%, with relation to pressure and electrical performance and establish the optimum point on the basis of trade-off.

Figures 15 and 16 give data on KOH pressure against temperature at various concentrations.

Since ZnO dissolves readily in KOH at high temperature, the vapor pressure of a saturated zincate solution of 45% KOH may even yield a lower pressure. Consequently, our tests utilized this type of electrolyte as well.

Another consideration is the relative succession of the sterilization procedure and the cell formation procedure; which one must precede is a matter of experimentation rather than of judgment, since the order of procedures is debatable.

On one hand, the sealed cell, when still unformed, will develop relatively low pressures only of the order of magnitude of the KOH vapor pressures at 145°C. The active materials sterilized in their original raw form are not expected to degrade, so that after sterilization the cell can

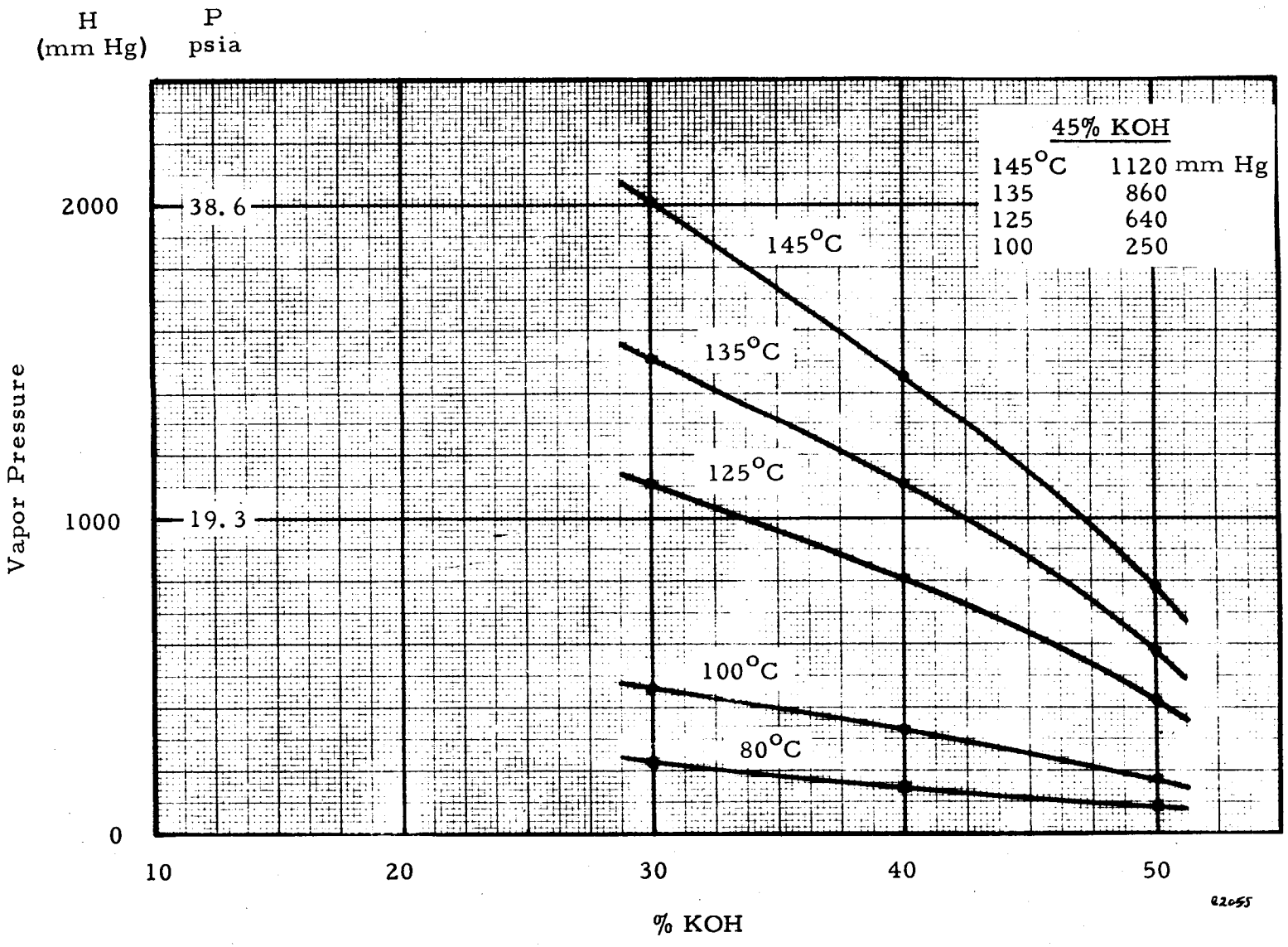


Figure 15. Vapor Pressure of KOH

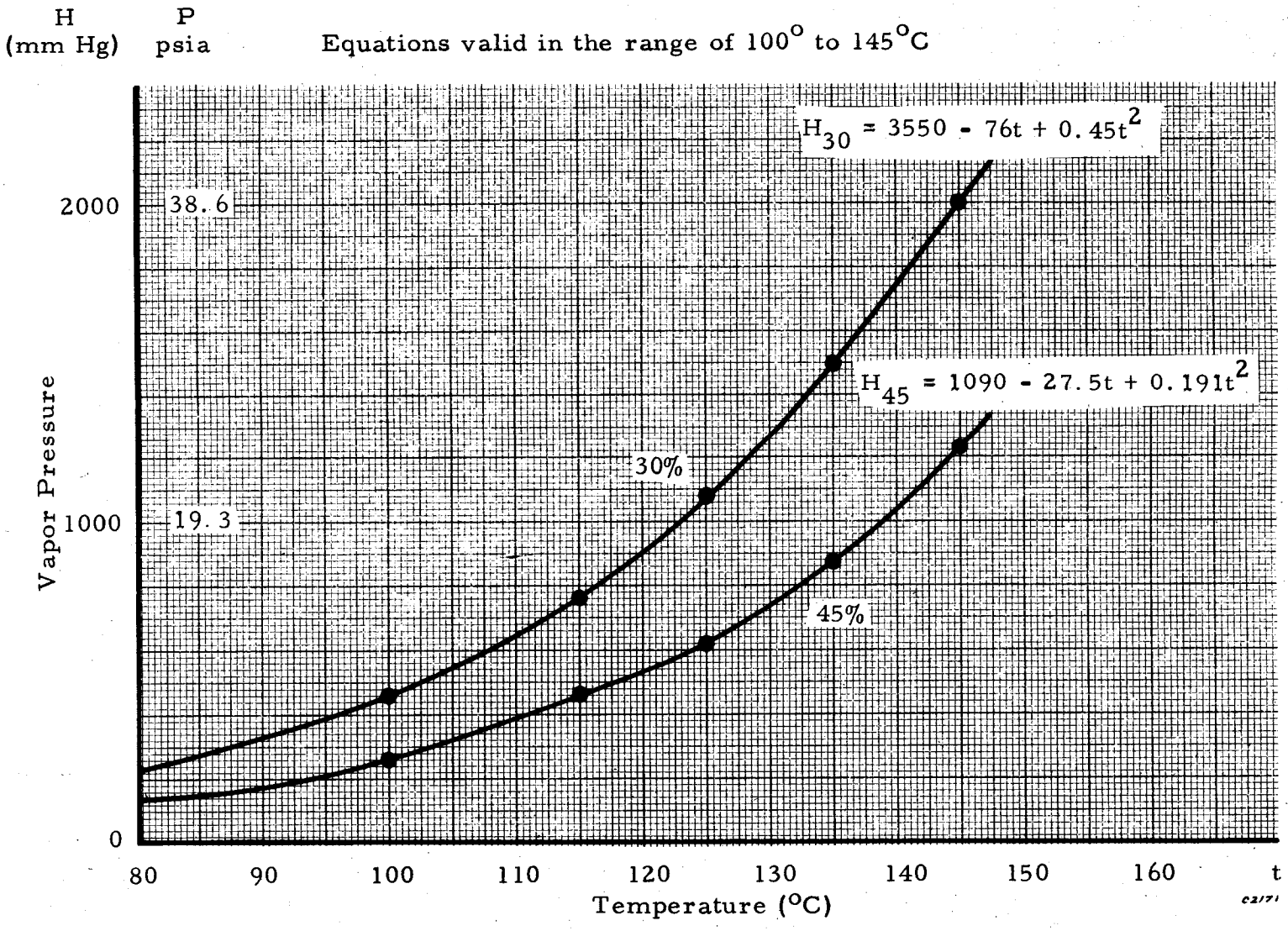


Figure 16. Vapor Pressure of KOH

be submitted to a regular formation, which is nothing more than a normal charge and discharge cycle.

On the other hand, starting with the formation would establish the cell performance and eliminate any possibility of fabrication deficiency before the cell is sealed and delivered to be mounted on the spacecraft and submitted to sterilization. However, after the formation discharge, the zinc electrode does not return to its original state of zinc oxide and mercuric oxide. At the sterilization temperature, the zinc electrode may undergo more degradation and therefore evolve hydrogen profusely. This approach was also tested to determine the extent of the degradation and the pressure build-up.

Cells were evaluated side by side, formation following sterilization and vice-versa.

### 4.7.2 Pressure Determination

Tests carried out on cell components being sterilized were also intended to determine the range of pressures to be encountered during the sterilization procedure from all sources: vapor pressures, gas expansion caused by elevated temperature, gas evolution caused by chemical processes or degradation.

The tests also serve to determine how much of a reduction in pressure can be expected by using 45% KOH over 30%, and by using zincate (KOH saturated with ZnO) rather than pure KOH solution.

The contribution of each cell component, alone or in combination, to pressure rise at 145°C is determined while they are undergoing regular sterilization experiments. The time spent at 145°C varies from a short time after the pressure stabilizes (approximately 3 hours) to full length test procedure (108 hours).

Table IV lists the pressures obtained for each component in different electrolytes. Table V categorizes them by electrolyte type.

Most promising candidates are listed with their maximum pressures in Figure 17.

TABLE IV

PRESSURES CATEGORIZED BY COMPONENTS

| Item | Electrolyte | Pressures (mm Hg Absolute) Start @ 25°C | Peak @ 145°C | End @ 25°C | Gas Sample |
|---|---|---|---|---|---|
| Unformed Silver Electrode | 45%-Z | 760 | 1690 | * | |
| | 45% KOH | 760 | 1900 | * | |
| | 30%-Z | 760 | 2620 | * | |
| | 30% KOH | 760 | 2670 | * | |
| Negative Mix A | 45%-Z | 760 | 2050 | * | |
| | 30%-Z | 760 | 2830 | * | |
| Unformed Silver Electrode and ZnO | 45%-Z | 760 | 2050 | * | |
| | 30%-Z | 760 | 2620 | * | |
| 3420-09 Separator | 45%-Z | 760 | 1430 | 630 | |
| | 45% | 760 | 2210 | * | X |
| | 30%-Z | 760 | 2620 | 630 | |
| Unf. Silver Electrode ZnO & 3420-09 Sep. | 45%-Z | 760 | 2210 | * | |
| | 30%-Z | 760 | 3140 | * | |
| Allbond Sealant | 45% KOH | 760 | 1790 | * | X |
| Potassium Titanate Fiber Sheet (KT) | 45% KOH | 760 | 2310 | * | X |
| | 30% KOH | 760 | 3260 | * | X |
| Asbestos Fiber Sheet | 45% KOH | 760 | 2210 | * | X |
| | 30% KOH | 760 | 2260 | * | X |
| Polypropylene SM-91 | 45% KOH | 760 | 2360 | * | X |
| | 30% KOH | 760 | 3290 | * | X |
| Polypropylene EM-476 | 45% KOH | 760 | 2050 | * | X |
| | 30% KOH | 760 | 3500 | * | X |
| Wafer with Dummy Electrode, Allbond Sealant | 45%-Z | 760 | 2470 | * | |
| | 30%-Z | 760 | 2310 | * | |
| Negative Wafer Allbond Sealant | 45%-Z | 760 | 1850 | 510 | |
| | 30%-Z | 760 | 3090 | 630 | |
| | 30% KOH | 760 | 3600 | * | X |

*No end pressure was noted because the chamber was opened before complete return to ambient temperature.

Z = KOH saturated with ZnO

## TABLE V

## PRESSURES CATEGORIZED BY ELECTROLYTE

| Electrolyte | Item | Pressures (mm Hg Absolute) Start @ 25°C | Peak @ 145°C | End @ 25°C | Gas Sample |
|---|---|---|---|---|---|
| 45%-Z | Unformed Silver Electrode | 760 | 1690 | * | |
| | Negative Mix A | 760 | 2050 | * | |
| | Unformed Silver Electrode & ZnO | 760 | 2050 | * | |
| | 3420-09 Separator | 760 | 1430 | 630 | |
| 45%-Z | Unformed Silver Electrode, ZnO & 3420-09 Separator | 760 | 2210 | * | |
| | Wafer with Dummy Electrode — Allbond Sealant | 760 | 2470 | * | |
| | Negative Wafer — Allbond Sealant | 760 | 1850 | 510 | |
| 45% KOH | Unformed Silver Electrode | 760 | 1900 | * | X |
| | 3420-09 Separator | 760 | 2210 | * | X |
| | Allbond Sealant | 760 | 1790 | * | X |
| | Potassium Titanate Fiber Sheet (KT) | 760 | 2310 | * | X |
| | Asbestos Fiber Sheet | 760 | 2210 | * | X |
| | Polypropylene SM-91 | 760 | 2360 | * | X |
| | Polypropylene EM-476 | 760 | 2050 | * | X |
| 30%-Z | Unformed Silver Electrode | 760 | 2620 | * | |
| | Negative Mix A | 760 | 2830 | * | |
| | Unformed Silver Elec. & ZnO | 760 | 2620 | * | |
| | 3420-09 Separator | 760 | 2620 | 630 | |
| | Unf. Silver Electrode, ZnO & 3420-09 Separator | 760 | 3140 | * | |
| | Wafer with Dummy Electrode— Allbond Sealant | 760 | 2310 | * | |
| | Negative Wafer—Allbond Sealant | 760 | 3090 | 630 | |
| 30% KOH | Unformed Silver Electrode | 760 | 2640 | * | |
| | Potassium Titanate Fiber Sheet (KT) | 760 | 3260 | * | X |
| | Asbestos Fiber Sheet | 760 | 2260 | * | X |
| | Polypropylene SM-91 | 760 | 3290 | * | X |
| | Polypropylene EM-476 | 760 | 3500 | * | X |
| | Negative Wafer—Allbond Sealant | 760 | 3600 | * | X |

*No end pressure was noted because the chamber was opened before complete return to ambient temperature.

Z = KOH saturated with ZnO

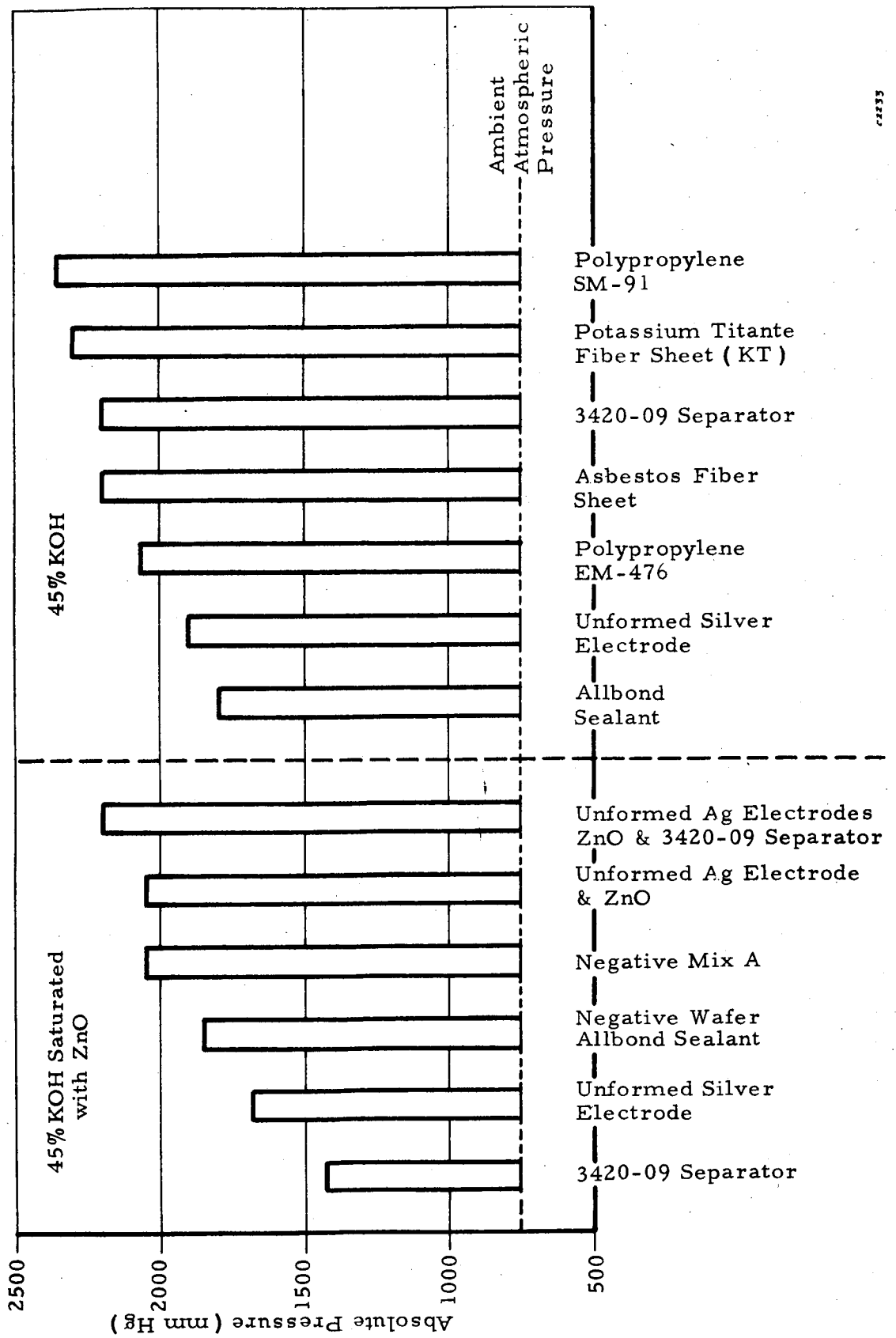


Figure 17. Pressures Generated by Most Promising Cell Components

The pressure obtained is not entirely predictable in nature because of the partial pressures other than water vapor pressure.

Most of the tests were started at atmospheric pressure with ambient air sealed in the vessel. The pressure reaches a peak at 145°C throughout the sterilization time; at the end, upon cooling off, the pressure drops and does not necessarily return to atmospheric. It may be higher, due to generation of new gases or lower due to oxygen dissolution in electrolyte or oxidation of organic materials by available oxygen. The gas analysis in one instance (see Section 4.8) shows a drastic diminution of the oxygen content (from 21% in the original ambient down to 3%) while the nitrogen content goes up to 92%. This discovery leads naturally to the idea of flushing the cell with oxygen before sealing, thus removing all nitrogen, or else simply evacuating the cell to the maximum before sealing. It will be possible to reduce the maximum pressure to a level close to the vapor pressure of the electrolyte being used.

## 4.8 Gas Analyses

The sealed vessels where the components are sterilized are provided with valves. At the end of the sterilization, samples of the internal gas mixture can be obtained through the valves by means of a special syringe. The gas mixture sample is injected in a gas chromatograph and analyzed qualitatively and quantitatively. Organic gases were detected by means of an I-R spectrophotometer. Several gas analyses were performed. At the same time, sterilized electrolyte was checked for carbonation.

Table VI gives the gas analyses for only some selected components which are worth considering.

Selection of the best sterilizable components must be based on minimum hydrogen evolution, minimum carbonation, and minimum pressure. However, diminution of oxygen pressure is often connected with heavy carbonation. A trade-off may be necessary.

## TABLE VI

## RESULTS OF GAS ANALYSES ON SELECTED COMPONENTS

| Material | Time @ 145°C | $N_2$ % | $O_2$ % | $H_2$ % | $CO_2$ % | CO % | $CH_4$ % | $H_2O$ Vapor % | Misc. Gases % | Electrolyte Carbonation |
|---|---|---|---|---|---|---|---|---|---|---|
| Polysulfone Case | 36 hrs. | 80.2 | 18.4 | — | trace | — | — | trace | 1.4 | — |
| | 108 hrs. | 80.0 | 16.0 | — | — | trace | — | trace | 4.0 | slight |
| 3420-09 Separators | 112 hrs. | 83.0 | 16.0 | — | trace | — | 1.0 | trace | — | slight |
| Cured Allbond Sealant | 18 hrs. | 92.0 | 3.0 | 3.0 | trace | — | 2.0 | trace | traces | considerable |
| Potassium Titanate Fiber Sheet (KT) | 41 hrs. | 83.0 | 16.0 | — | trace | trace | trace | trace | traces | slight |
| Asbestos Fiber Sheet | 113 hrs. | 81.0 | 16.0 | 2.0 | trace | — | 1.0 | trace | — | none |
| Polypropylene EM-476 - pretreated | 114 hrs. | 80.0 | 20.0 | — | trace | — | trace | trace | — | slight |
| Polypropylene SM-91 - pretreated | 113 hrs. | 84.0 | 16.0 | — | trace | — | — | trace | — | considerable |

## 5.0 ELECTRICAL DEVELOPMENT

### 5.1 Separator Box Design

In the first part of the program, in the absence of a sealant capable of withstanding heat sterilization in KOH, another type of assembly was tried where no sealant was used. The separator consisted of one single rectilinear vessel in the form of the wafer used, with two flat sides as regular separators. This integral piece is made by pressing the inorganic material isostatically in a mold and machining it to the proper dimensions (Figures 18 and 19).

Only a small amount of work was done and any further testing was discontinued when a suitable cement was found for the standard assembly (wafer design) described in Section 3.1 and reported in detail in Section 5.2.

The electrical tests run on the separator box design are reported here.

#### 5.1.1 First Run

Two cells were built with a one ampere-hour nominal capacity. One cell was sterilized unformed and one cell was used as control and not sterilized.

Control: A-20-2

| | |
|---|---|
| Output: | 1.30 Ah |
| 2A-pulse voltage: | 1.28 V |
| 2A-pulse power: | 1.56 W |
| Automatic cycling regime: | 162 cycles |

Test Cell: A-20-1

| | |
|---|---|
| Sterilized unformed; evacuated: | 25 mm Hg |
| Maximum pressure during sterilization: | 2180 mm Hg |
| Gas analysis: | $N_2$ = 91%, $O_2$ = 9% |
| Output: | 1.10 Ah |
| 2A-pulse voltage: | 1.13 V |
| 2A-pulse power: | 2.26 W |
| Automatic cycling regime: | 141 cycles |

Figure 20 shows voltage characteristics of the two cells.

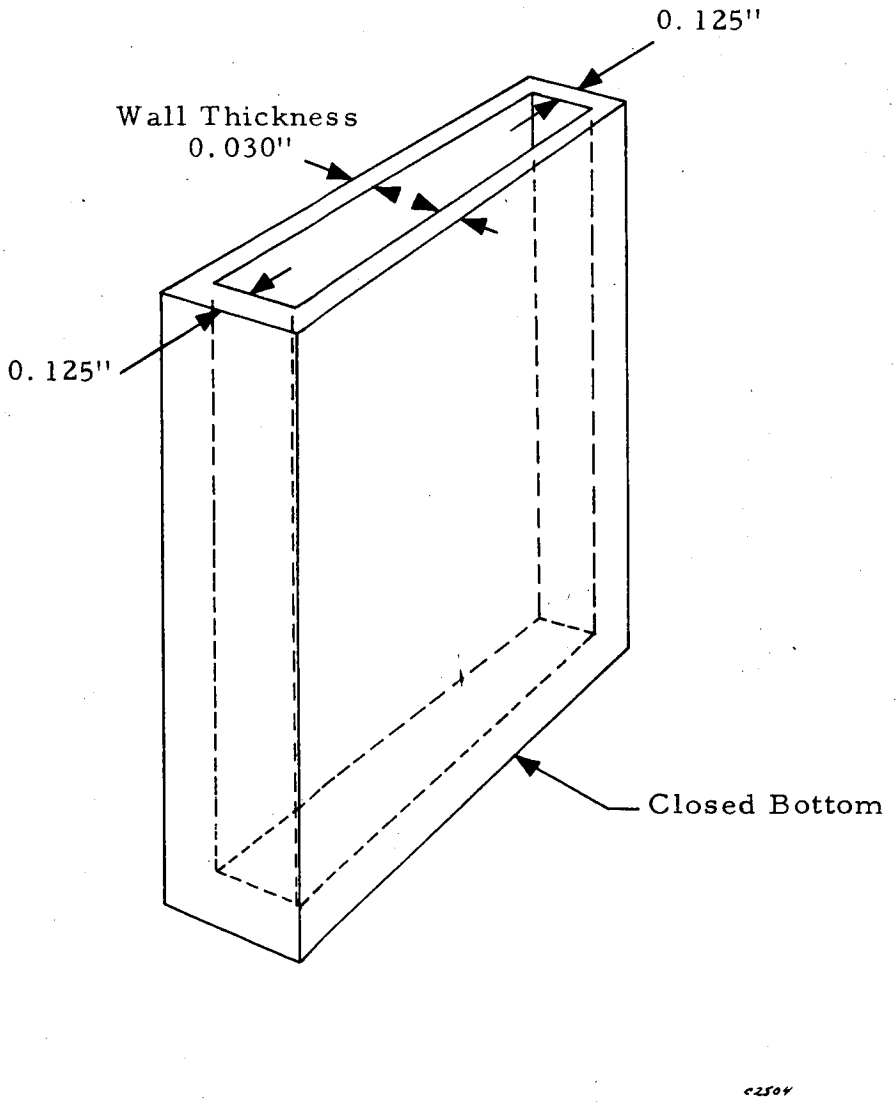


Figure 18. Separator Box

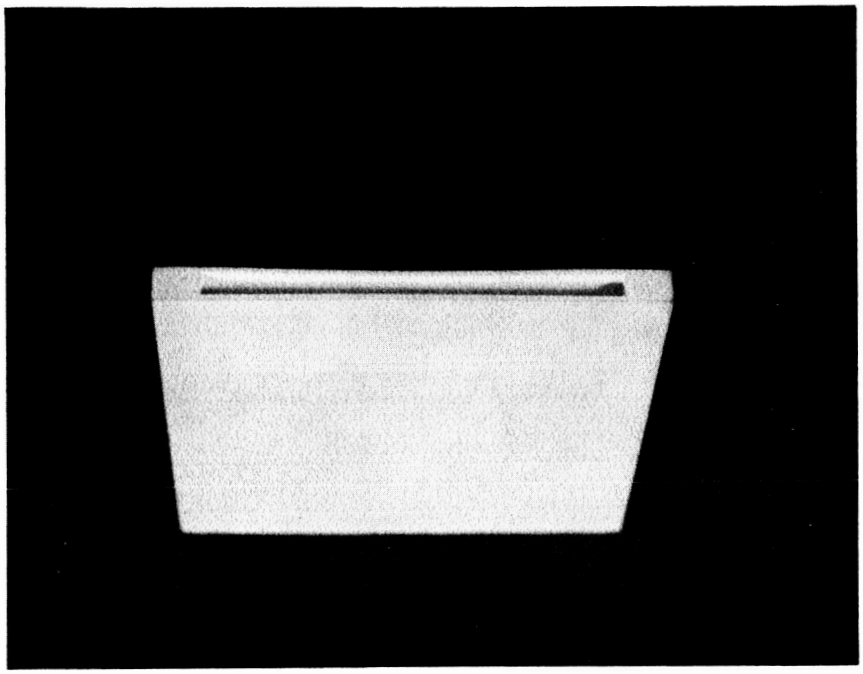

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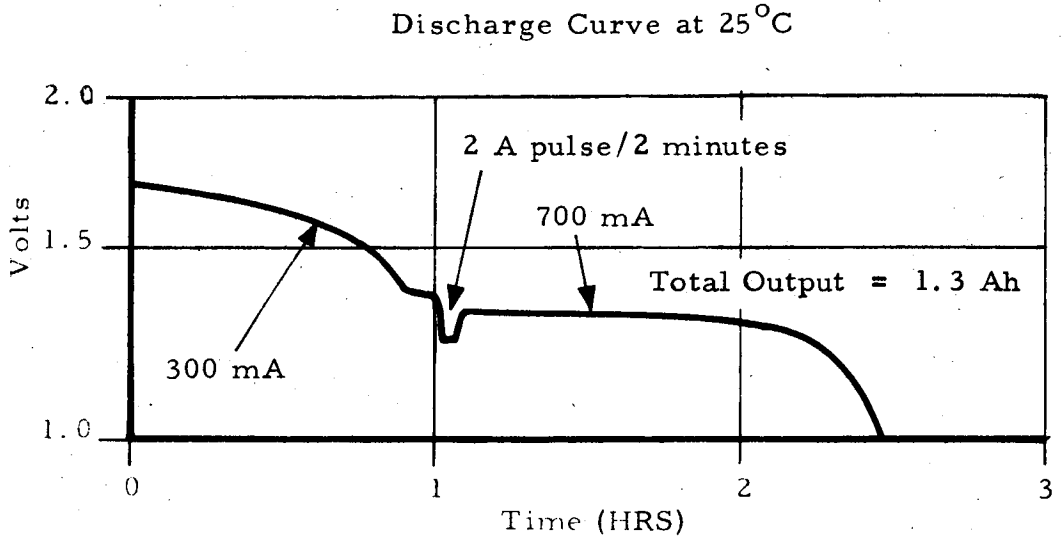
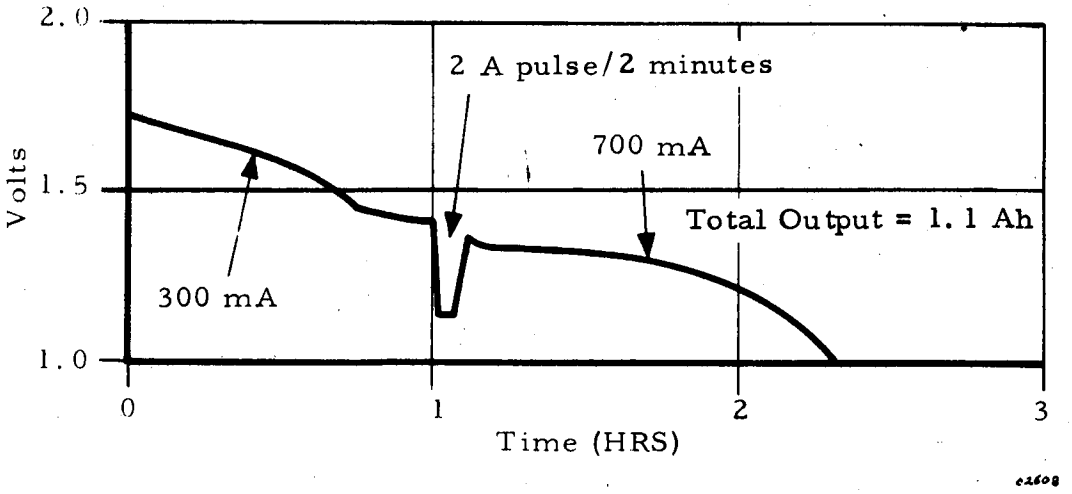
Figure 19. Sterilized Separator Box




Unsterilized Control Cell A-20-2




Sterilized Cell (A-20-1)
116 hrs @ 145°C

Figure 20. Box Design – Nominal Capacity: 1 Ah

### 5.1.2 Second Run

Again two cells were built and tested as follows: one cell left unformed, then sterilized and one cell formed, discharged, then sterilized. Each cell was subsequently recharged and discharged on a duty cycle.

Results:

Cell A-39-1: Unformed
Sterilized at 145°C for 111 hours
Charged and discharged
Output: 1.2 Ah
2A-pulse voltage: 1.29 volts
2A-pulse power: 2.58 watts

Cell A-39-6: Formed and discharged
Output: 1.1 Ah
Sterilized at 145°C for 108 hours
2A-pulse voltage: 1.23 V
Charged and discharged
Output: 0.9 Ah
2A-pulse voltage: 1.06 volts
2A-pulse voltage: 2.12 watts

Discharge characteristics are shown in Figures 21 and 22, respectively.

## 5.2 Wafer Configuration Design

Samples of inorganic separator used in building wafer assemblies are shown in Figure 23 — one before sterilization, one after sterilization, and one after sterilization and cycling.

Two identical cells coded A-22-1 and A-22-2 were built using inorganic separators and KT interseparators on both the positive and negative electrodes and filled with 45% KOH at the same time. Allbond was used as a sealant.

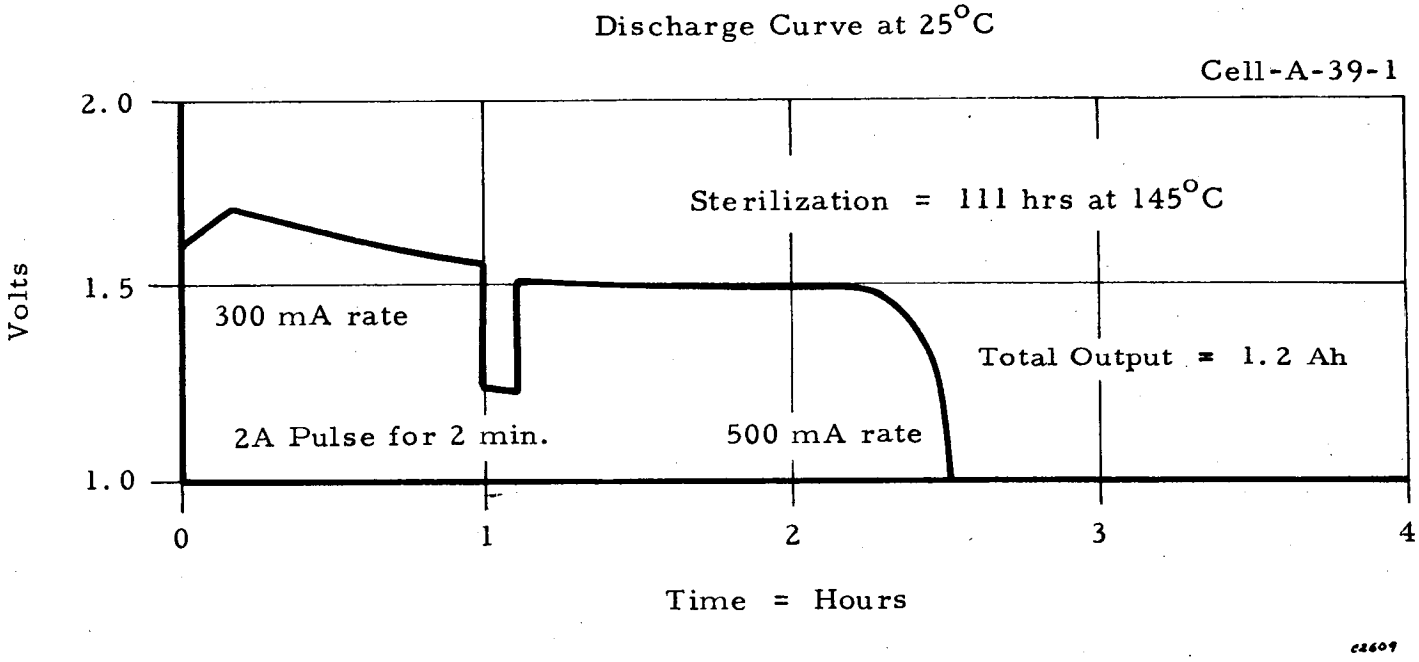



Figure 21. Box Design: Cell Sterilized, Unformed — Nominal Capacity: 1 Ah

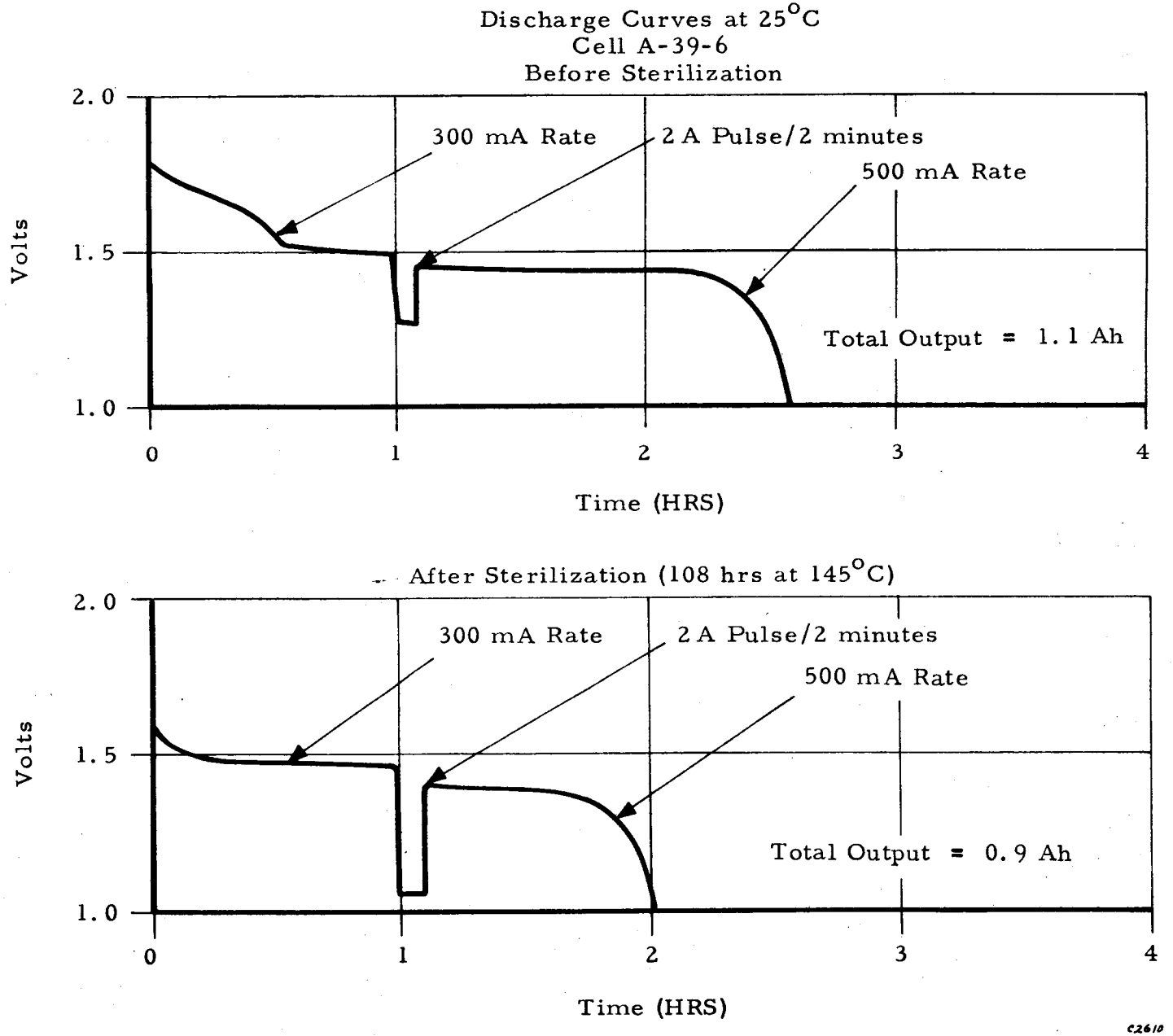
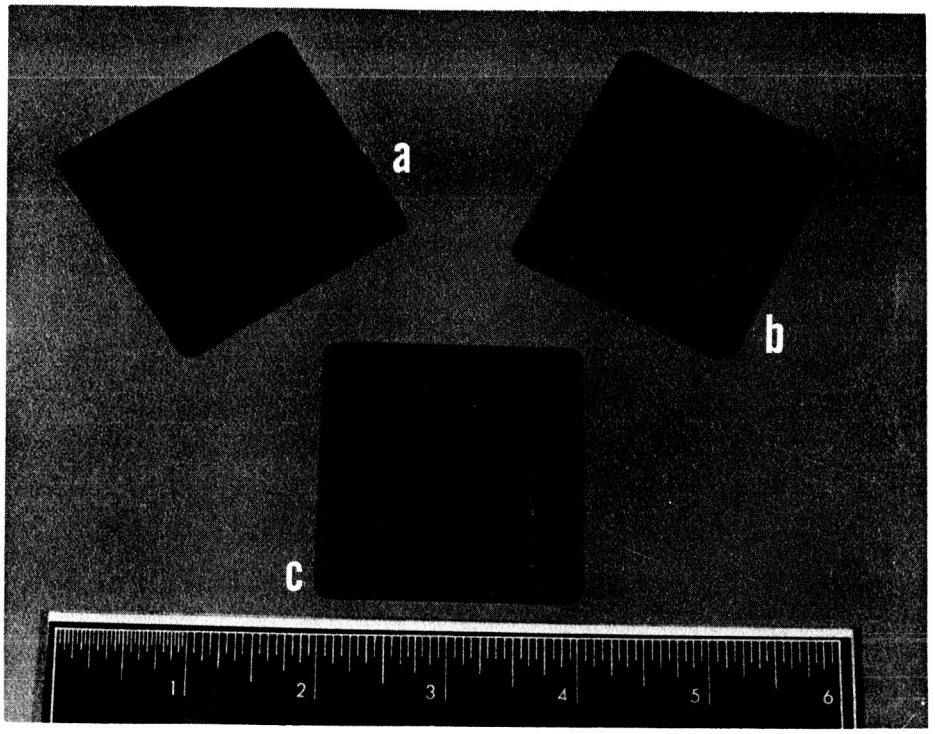



Figure 22. Box Design Cell Sterilized After Formation and Discharge — Nominal Capacity: 1 Ah




a. Before Sterilization

b. After Sterilization

c. After Sterilization and Continuous Cycling (274 Cycles)

c2621

Figure 23. Flat Separator Before Sterilization, After Sterilization, and After Cycling

Cell A-22-1 was submitted to heat-sterilization without formation, then charged and discharged. On the other hand, cell A-22-2 was given a formation cycle (charge and discharge) first, then heat-sterilized.

All charges were carried out at 100 mA and 2.10 V for total capacity determination.

Pressure and gas analyses were recorded for each pressure vessel.

Data are summarized in Table VII. As expected, the pressure is higher in the preformed cell and hydrogen evolution much higher. In anticipation of high pressure build-up, the pressure vessel containing the preformed cell was evacuated. The maximum reached was approximately 55 psig, compared to 27 psig for the unformed cell.

After the duty cycles, the cells were placed on a simulated automatic cycling regime. The equivalent capacity of the duty cycle (0.3 Ah) is removed and put back on a one-hour cycling period as follows: discharge at 0.6 A for 1/2 hour; recharge at 0.72 A for 1/2 hour, thus simulating a low-orbital flight regime. Characteristic curves are given in Figures 24 and 25 for their duty cycles and in Figures 26 and 27 for their continuous automatic cycling until failure. Their actual capacity was approximately 2 Ah.

The wafer configuration design was therefore selected as the most convenient vehicle for characterizing the rectilinear cell. The flexible sealant (neoprene type) was used to seal the electrode compartment as it proved successful. A discharge of a typical cell on a duty cycle is shown in Figure 28 after sterilization and recharge. The cell was then put on automatic cycling — 1/2 hour discharge at 0.6 A = 0.3 Ah (discharge capacity of a duty cycle) and 1/2 hour recharge at 0.72 A.

The cell completed 502 cycles before it could not accept any recharge due to cell pack drying.

TABLE VII

UNFORMED VS. PREFORMED CELL DATA
$2^+/1^-$ WAFER, KT INTERSEPARATOR

| | A-22-1, Unformed | A-22-2, Preformed |
|---|---|---|
| Cycle 1 (charge & discharge) | | |
| Output to 1.0 V | | 2.2 Ah |
| 2 A pulse: voltage | | 1.18 V |
| 2 A pulse: power | | 2.36 W |
| Sterilization at 145°C | 104 hrs | 98 hrs |
| Initial pressure (before sterilization) | 760 mm Hg | 51 mm Hg |
| Maximum pressure (during sterilization) | 2,160 mm Hg | 3,600 mm Hg |
| Gas Analysis | | |
| $N_2$ | 93.5% | 16% |
| $O_2$ | 3.0% | trace |
| $H_2$ | 3.5% | 82% |
| $CH_4$ | trace | 2% |
| Cycle 1 (charge & discharge) | | |
| Output to 1.0 V | 2.5 Ah | |
| 2 A pulse: voltage | 0.96 V | |
| 2 A pulse: power | 1.92 W | |
| Cycle 2 | | |
| Output | 2.30 Ah | 1.90 Ah |
| 2 A pulse: voltage | 0.97 V | 1.03 V |
| 2 A pulse: power | 1.94 W | 2.06 W |
| Cycling Regime (0.3 Ah) at 25°C | | |
| Discharge: 1/2 hr x 0.6 A | | |
| Charge: 1/2 hr x 0.72 A | | |
| Cycles to Failure | 274 | 167 |

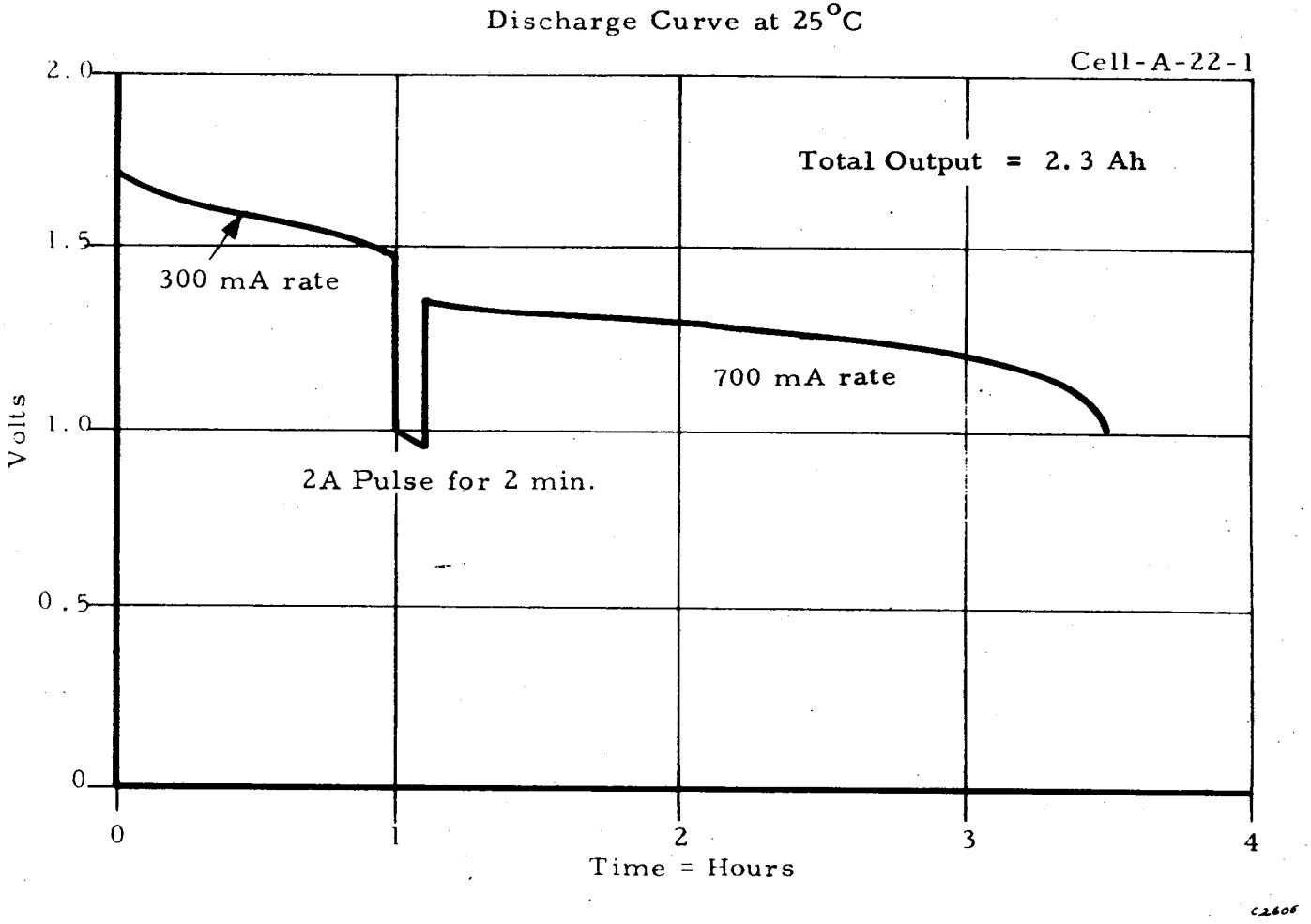



Figure 24. Standard Design: Cell Sterilized, Unformed — Nominal Capacity: 2 Ah

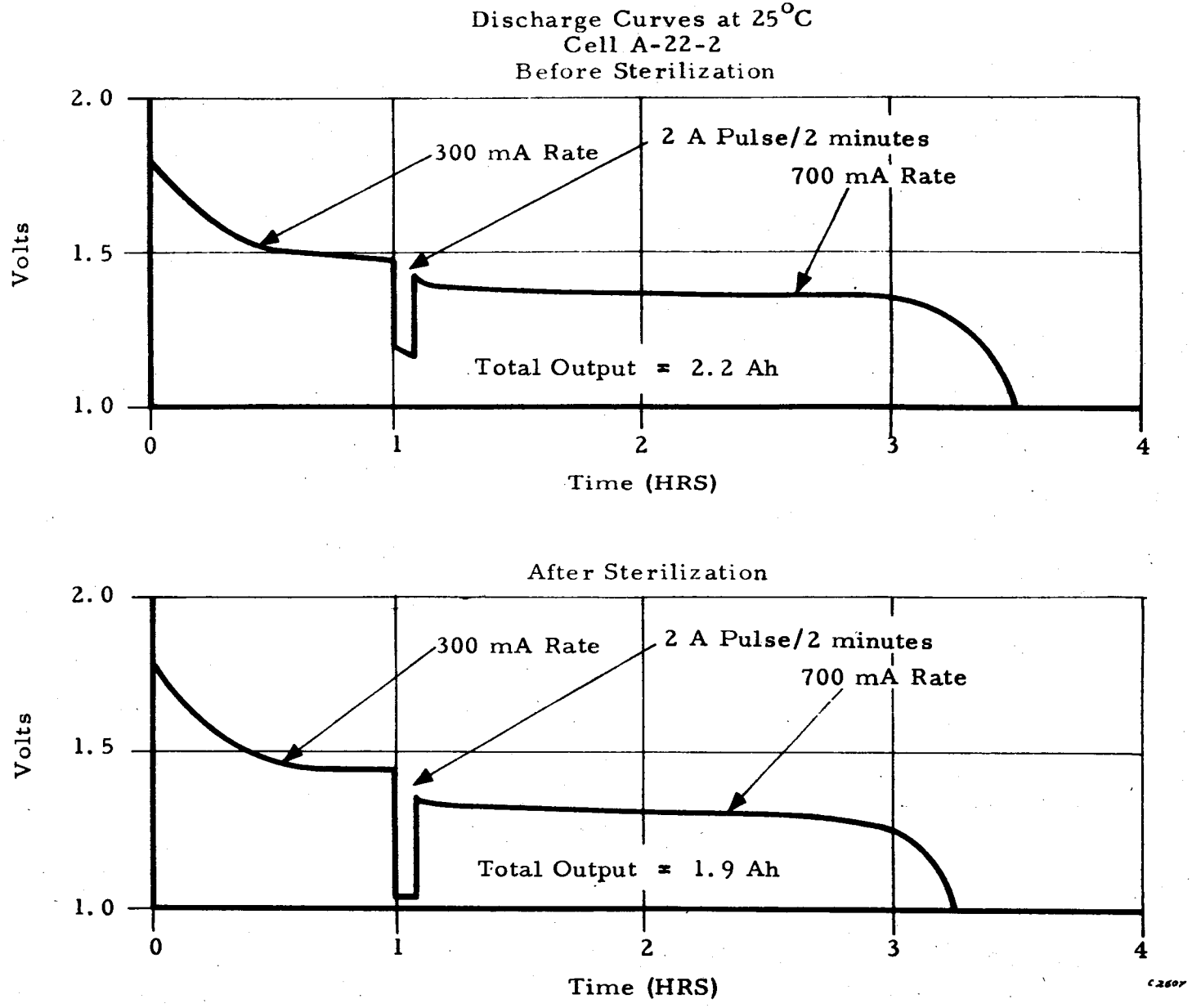
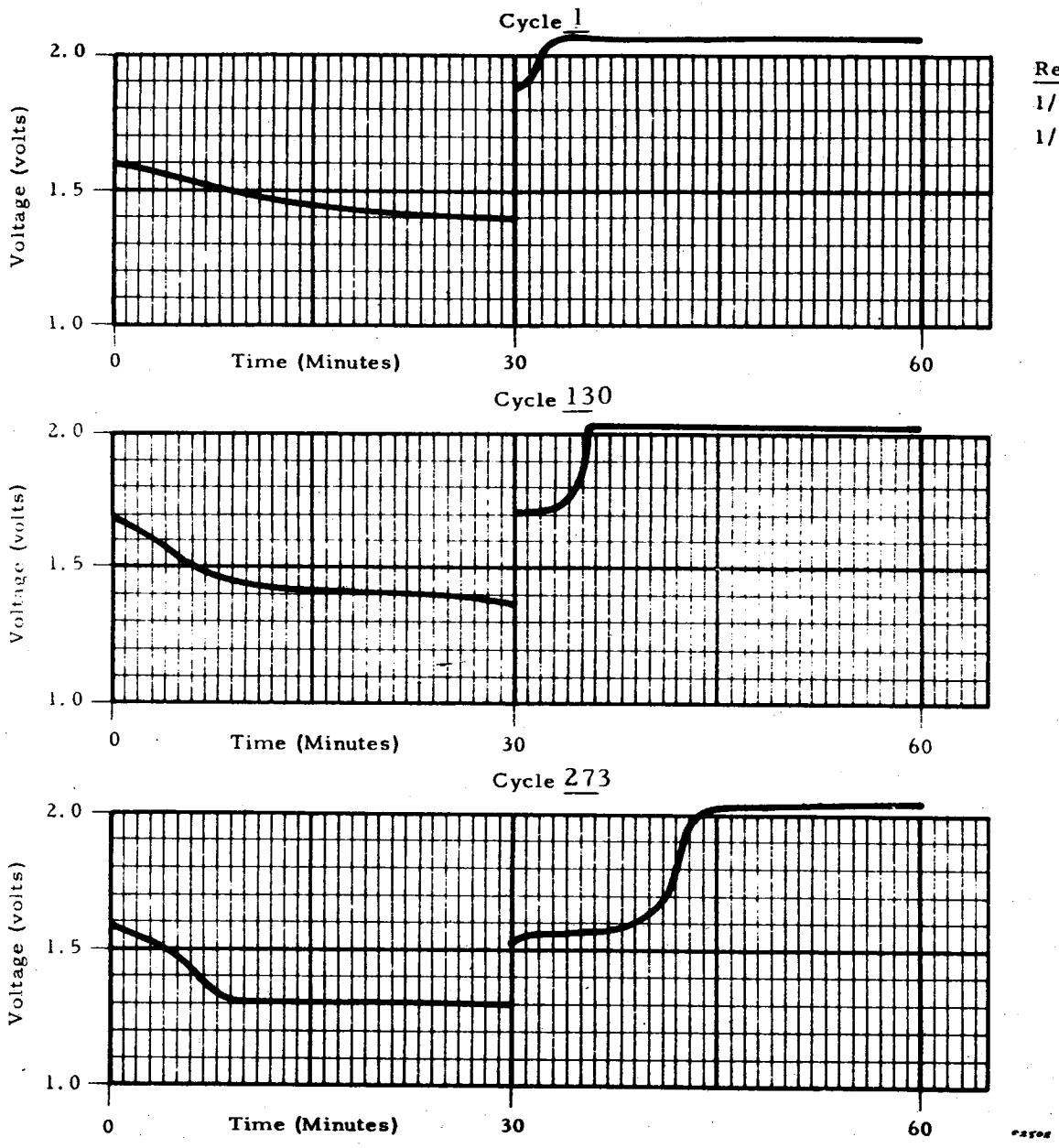



Figure 25. Standard Design Cell Sterilized After Formation and Discharge — Nominal Capacity: 2 Ah



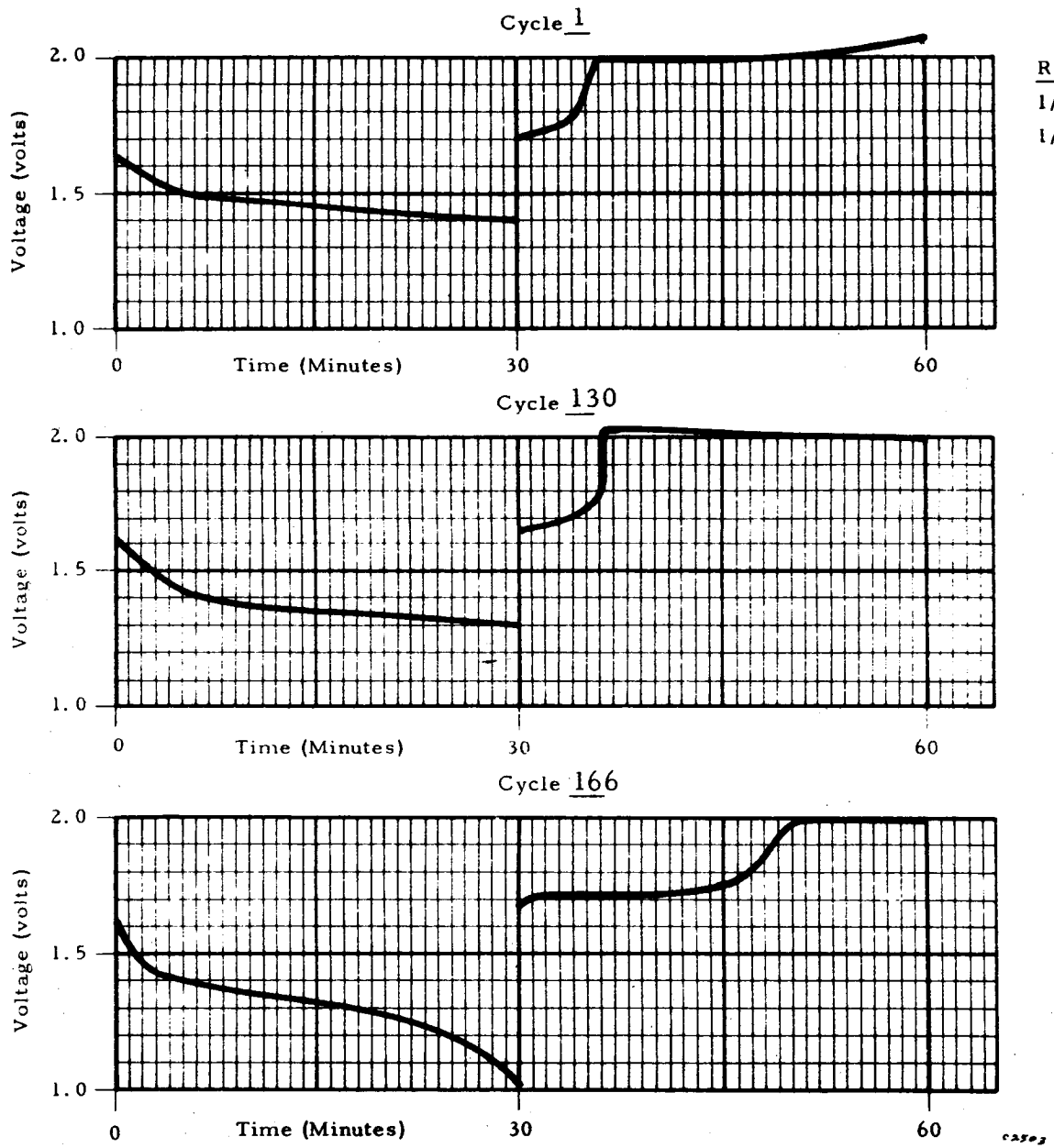

Cell No. A-22-1

Regime 25°C

1/2 hr-discharge: 0.6 A

1/2 hr-charge: 0.72 A

Figure 26. Characteristics of Cell Sterilized, Unformed, on Automatic Cycling




Cell No. A-22-2

Regime 25°C

1/2 hr-discharge: 0.6 A

1/2 hr-charge: 0.72 A

Figure 27. Characteristics of Cell Sterilized, Preformed, on Automatic Cycling

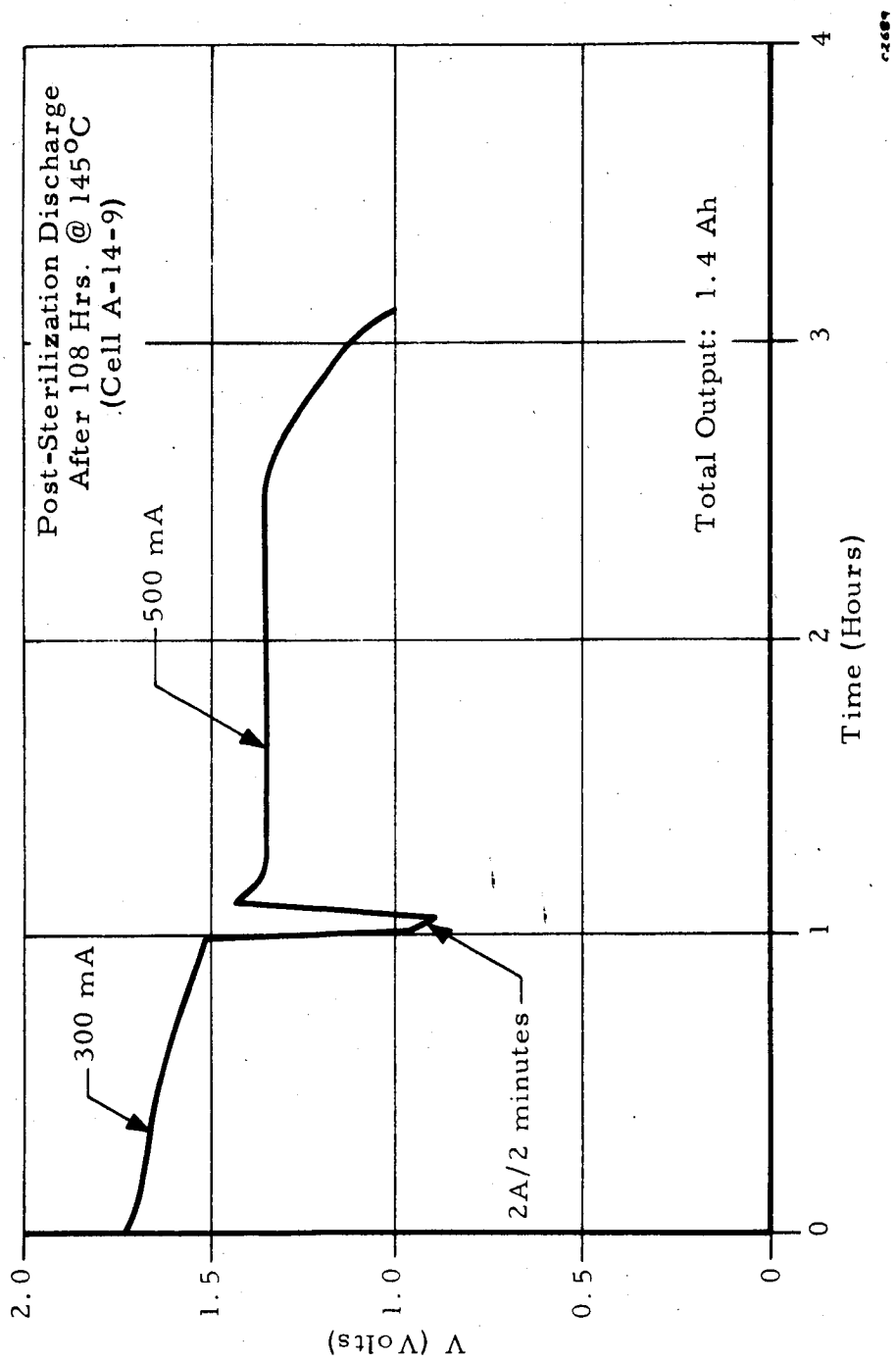



Figure 28. Standard Flat Design (Sterilized Unformed) With Flexible Sealant

## 6.0 MECHANICAL DEVELOPMENT

Under this section is consolidated all work done during the course of the program on terminals, cases and case-to-cover seals. This work was carried out concurrently with the electrical development and evaluation work until the end of the program.

### 6.1 Terminals

Several models of terminals, terminal-to-cover seals and lead attachment to the terminal base were considered throughout the program. The design evolution can be seen in Figures 29 through 33.

Model #1 consists essentially of an 8-32 screw fitted in a threaded hole in the plastic cover and sealed with Teflon tape. It is screwed from the bottom until its flat median section comes to a stop (Figure 29). Designs 1 and 2 of the lead attachments are shown in Figures 32 and 33.

In the first one, the base is slotted and partially flattened. After inserting the electrode tabs in the slot, a nut comes down and tightens the expanded section of the screw.

In the second one, the base is drilled approximately 1/2 inch. The electrode leads (0.016" diameter silver wires) are inserted in the blind hole, and the base is crimped.

The electrode lead attachments to the terminal were tested for good contact. A current of 5 A was applied through the leads up tb the terminal top, and voltage drop was measured between leads and terminal top.

| Design #1 | Run | Voltage |
|---|---|---|
| Two silver tabs 1/8 inch wide, 0.006 inch thick | 1 | 28.5 mV |
| | 2 | 30.0 mV |
| **Design #2** | | |
| Six silver 0.016 inch wires | 1 | 3.2 mV |
| | 2 | 4.6 mV |

Model #2, described in Figure 30, was to be used as a back-up. The design consists of a Teflon circular wedge pressed into a countersink by the terminal base when the terminal top is tightened with a nut. Extra sealing

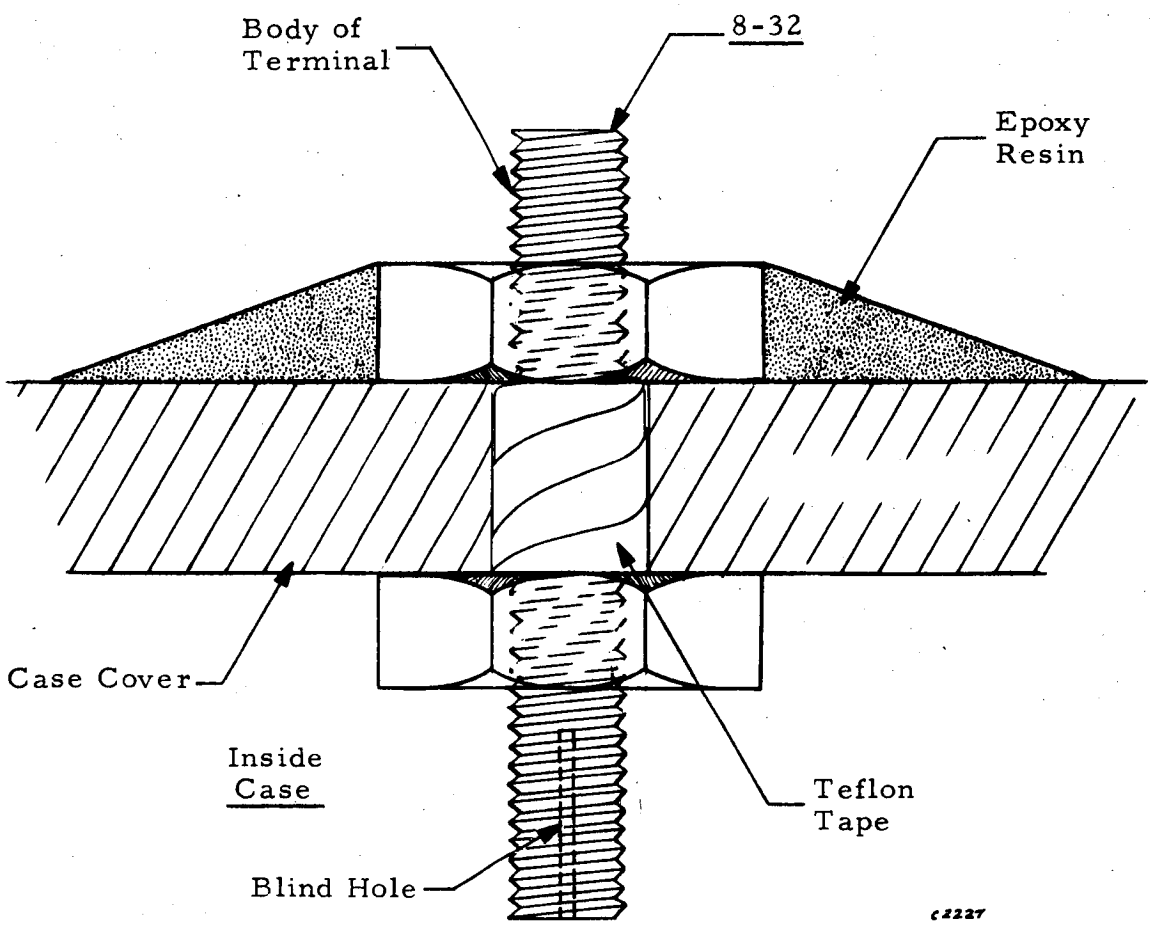



Figure 29. Terminal-to-Cover Seal Assembly — Model #1

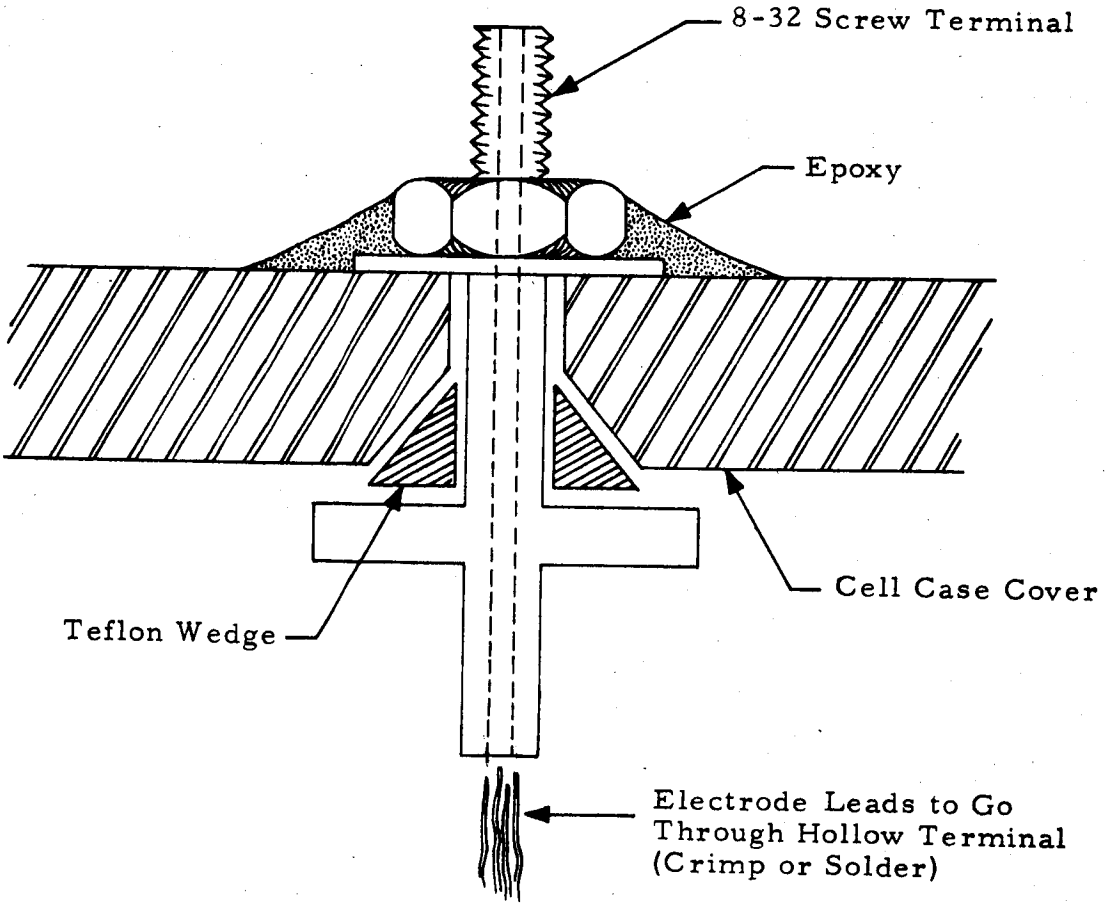



Figure 30. Terminal-to-Cover Seal Assembly – Model #2

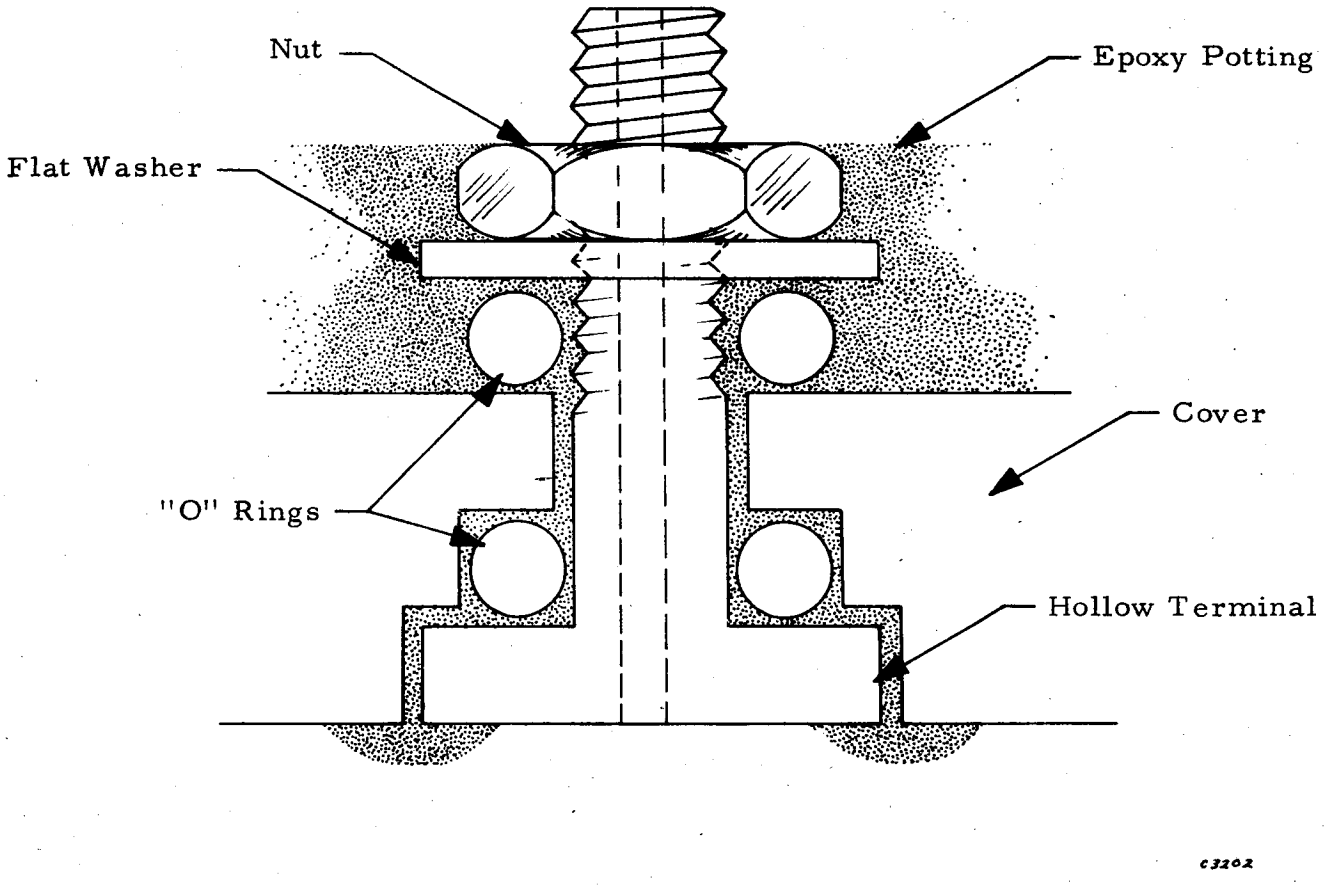



Figure 31. Terminal-to-Cover Seal Assembly, Model #3

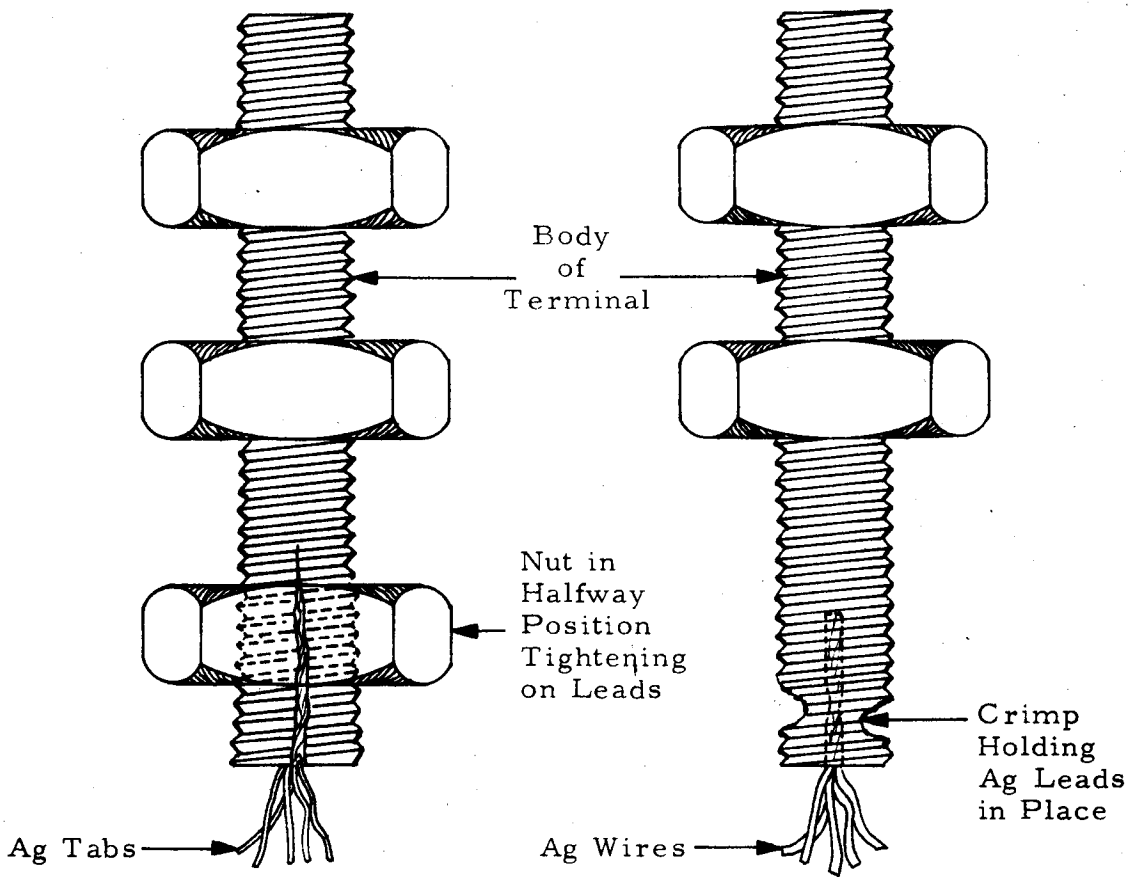



Figure 32. Attachment of Electrode Leads to Terminal

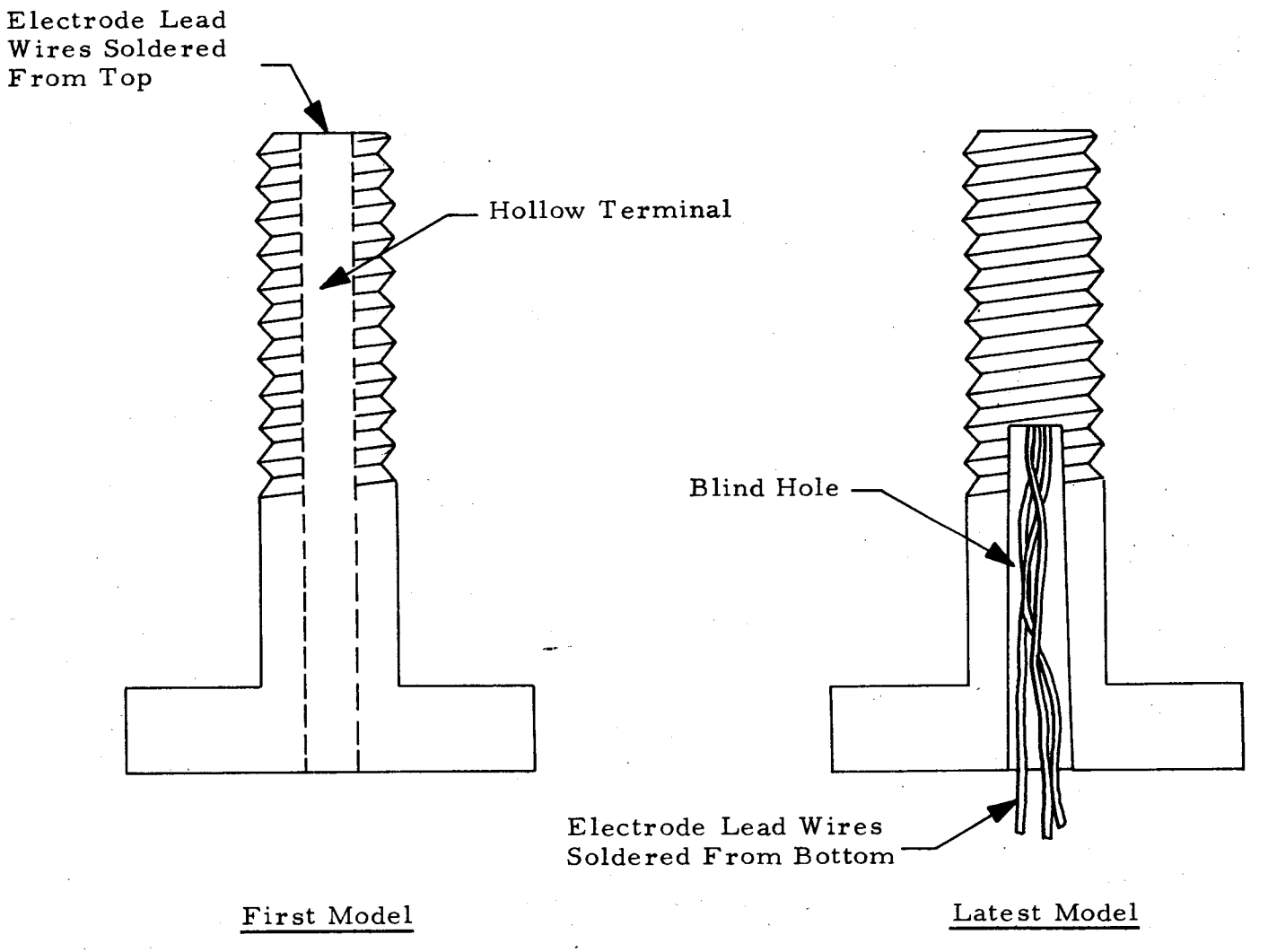



Figure 33. Attachment of Electrode Leads to Terminal (Design #3)

was provided with epoxy resin, filling all void space around the terminal body as well as the top and bottom.

When molded cases and covers were obtained from another NASA contract, the terminal assembly evolved to the model #3 represented in Figure 32. The lead attachment design #3 is shown in Figure 34.

The terminal seals were tested in a special fixture represented in Figures 34 and 35.

The terminal-to-cover seal must be gas tight at the expected test pressures and be unaffected by prolonged exposure to hot KOH. The fixture provided conditions more severe than those experienced during sterilization.

Two terminals were tested. Each held a pressure of 50 to 54 psig (2600 to 2800 mm Hg absolute) at 145°C for more than 108 hours (115 and 122 hours), after which the test was stopped and the terminal top checked for traces of alkali. Neither terminal had any traces.

### 6.2 Case Testing

The three materials used for the molded case were Polysulfone P-1700, PPO grade 534-801 (natural), and PPO grade 541. The cases were pressurized to determine the bursting pressure. The pressure of 100 psi was maintained for five minutes; then the pressure was raised until the case exploded. Test data are presented in Table VIII. Figure 36 shows a picture of molded cases used in our tests.

One case of the material considered was submitted to 145°C and KOH exposure for a very long time and retested for bursting pressure. It appears that the sterilization procedure would not appreciably change the burst strength of the natural PPO case.

Another series of tests was to measure the bulge of the case under continuous stress at 145°C. The cases were unrestrained on their flat faces and a pressure of 50 psi (about 50% over the sterilization pressure) was maintained for four hours. The thickness of the case, taken between the large faces, was measured before the test and after the test at room temperature, so that the bulge noted was a permanent deformation (Table IX).

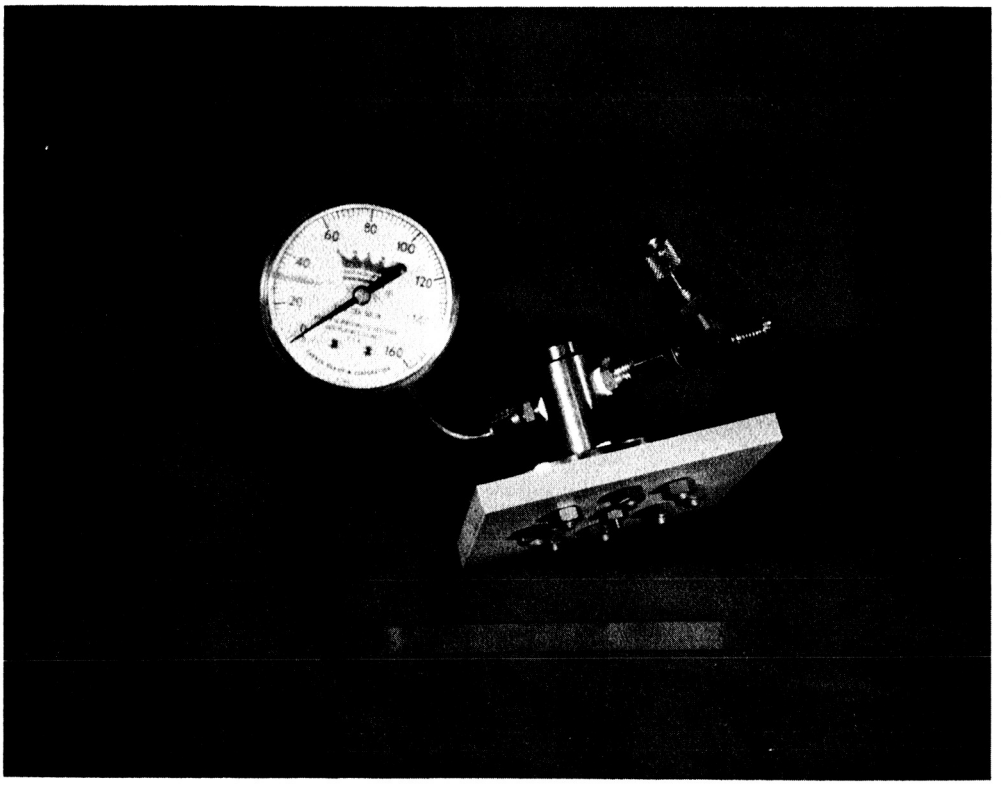



Figure 34. Terminal Test Fixture

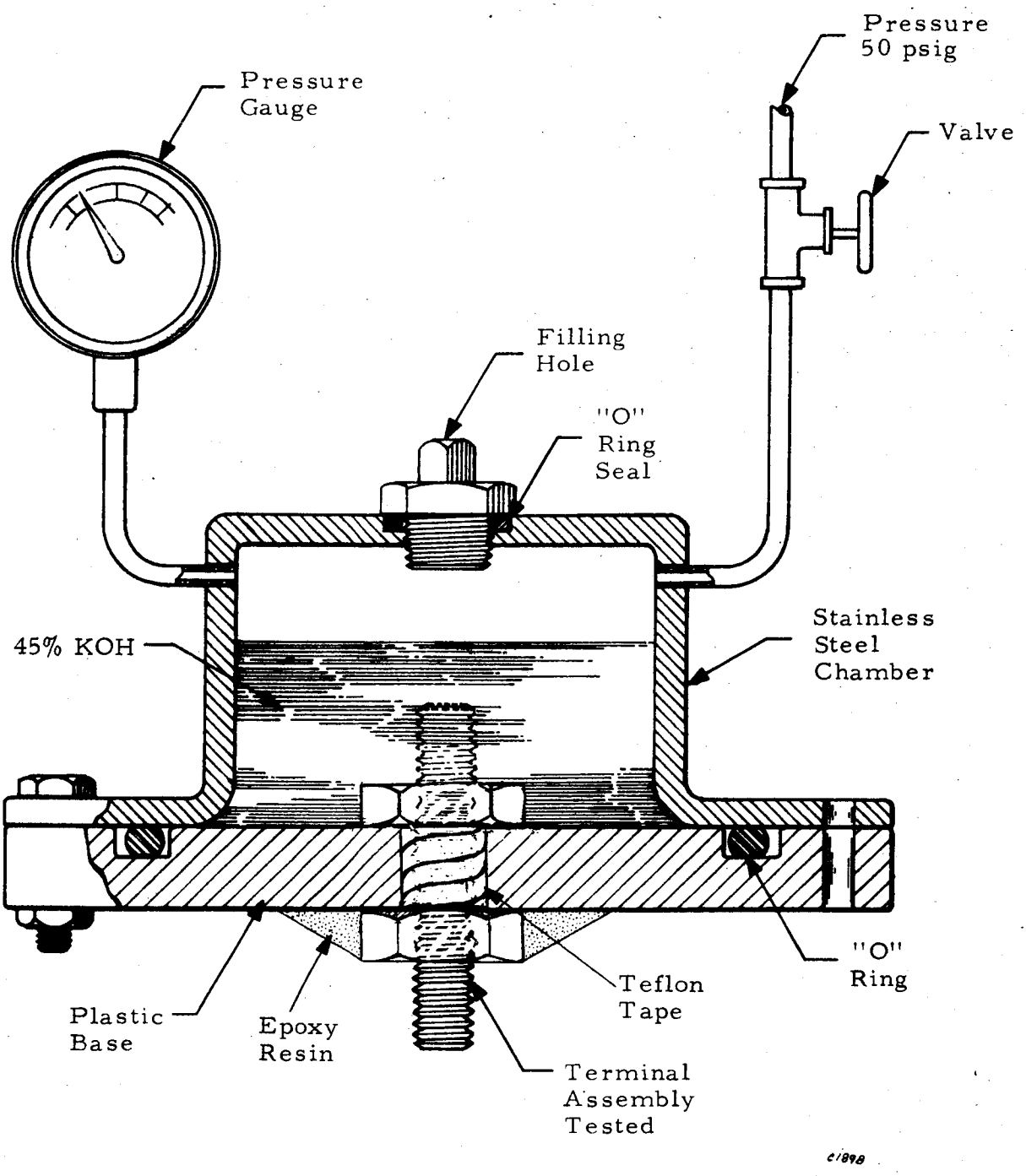



Figure 35. Schematic of Test Fixture for Terminal Seal

## TABLE VIII

## CASE PRESSURE TESTS

| Material | Sample No. | Test 100 psi/5 min. | Bursting Pressure |
|---|---|---|---|
| Polysulfone P-1700 | 1 | Passed | 130 psi |
| | 2 | Failed | 100 psi after 4 min. |
| | 3 | Failed | 100 psi after 30 sec. |
| | 4 | Failed | 100 psi after 20 sec. |
| PPO Natural (Grade 534-801) | 1 | Passed | Over 140 psi* |
| | 2 | Passed | Over 140 psi* |
| | 3 | Passed | Over 140 psi* |
| | 4 | Passed | Over 140 psi* |
| PPO (Grade 541-801) | 1 | Failed | 85 psi |
| | 2 | Failed | 85 psi |
| | 3 | Failed | 85 psi |
| | 4 | Failed | 75 psi |

| Material | Sample No. | Time at 145°C | Bursting Pressure |
|---|---|---|---|
| Polysulfone | 39 | 108 hrs. | 97 psi |
| PPO Natural | 25 | 317 hrs. | Over 140 psi* |

*Test was discontinued at 140 psi, which was the valve relief setting on the pressure regulator.

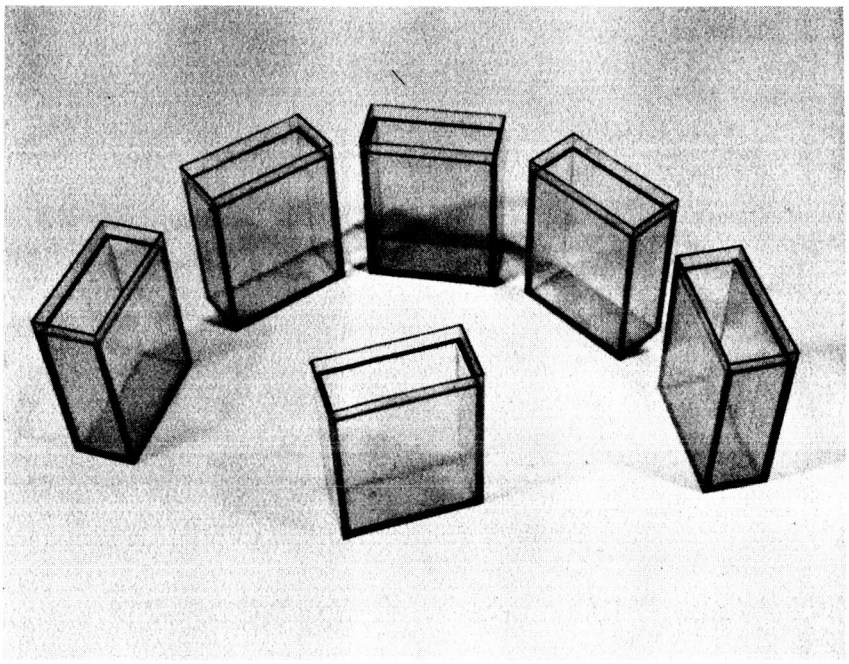


Figure 36. Molded Polysulfone Cases

TABLE IX

PERMANENT DEFORMATION OF UNRESTRAINED CASES AT 145°C UNDER 50 PSI

| Material | Sample No. | Thickness (Inches) | |
|---|---|---|---|
| | | Before | After |
| Polysulfone P-1700 | 1 | 1.030 | 1.100 |
| | 2 | 1.030 | 1.183 |
| | 3 | 1.032 | 1.097 |
| | 4 | 1.030 | 1.067 |
| PPO Natural (534-801) | 1 | 1.026 | 1.132 |
| | 2 | 1.027 | 1.101 |
| | 3 | 1.027 | 1.107 |
| | 4 | 1.033 | 1.160 |
| PPO Filled (Grade 541-801) | 1 | 1.037 | 1.059 |
| | 2 | 1.036 | 1.066 |
| | 3 | 1.037 | 1.070 |
| | 4 | 1.038 | 1.070 |

However, it should be kept in mind that the cell is restrained in a battery assembly and that the sterilization pressure is much lower.

## 6.3 Cover-To-Case Seal

### 6.3.1 Epoxy Seal

The integrity of the cover-to-case seal was tested on a few cases. The method used was the epoxy resin cementing. Two epoxy resins were selected: BR-92 (American Cyanamid Company) recommended for polysulfone; and Allbond (Allaco).

After plugging the terminal holes and sealing the cover to the case, the assembly was pressurized in increments of 10 psig, and the pressure held for five minutes. This was done at 25°C and 145°C until a leak developed or 100 psig was reached.

The procedure is considered only as a screening test. The actual test would be exposure at 145°C for 108 hours.

Below is a tabulation of the preliminary data.

| | BR-92 | Allbond |
|---|---|---|
| 25°C | 100 psig, 5 min.<br>No leak | 100 psig, 5 min.<br>No leak |
| 145°C | 40 psig, 5 min.<br>No leak<br><br>50 psig, 2 min.<br>Pinhole leak | 40 psig, 5 min.<br>No leak<br><br>48 psig, leak<br>56 psig, case broke |

Figure 37 depicts another type of sealing. The cell case is held in an inverted position, fitting into the cover where a fresh layer of epoxy resin 1/8 inch thick has been poured. The epoxy is also spread between the junction surfaces of the case and cover until it is squeezed out. Fiberglas tape (3M Scotch 361) impregnated with fresh epoxy is wrapped around the case at the junction line.

Before assembling the case, the holes for terminals were plugged and a polyethylene tube was temporarily fitted into the filling hole to leave the opening free for adapting the pressure devices.

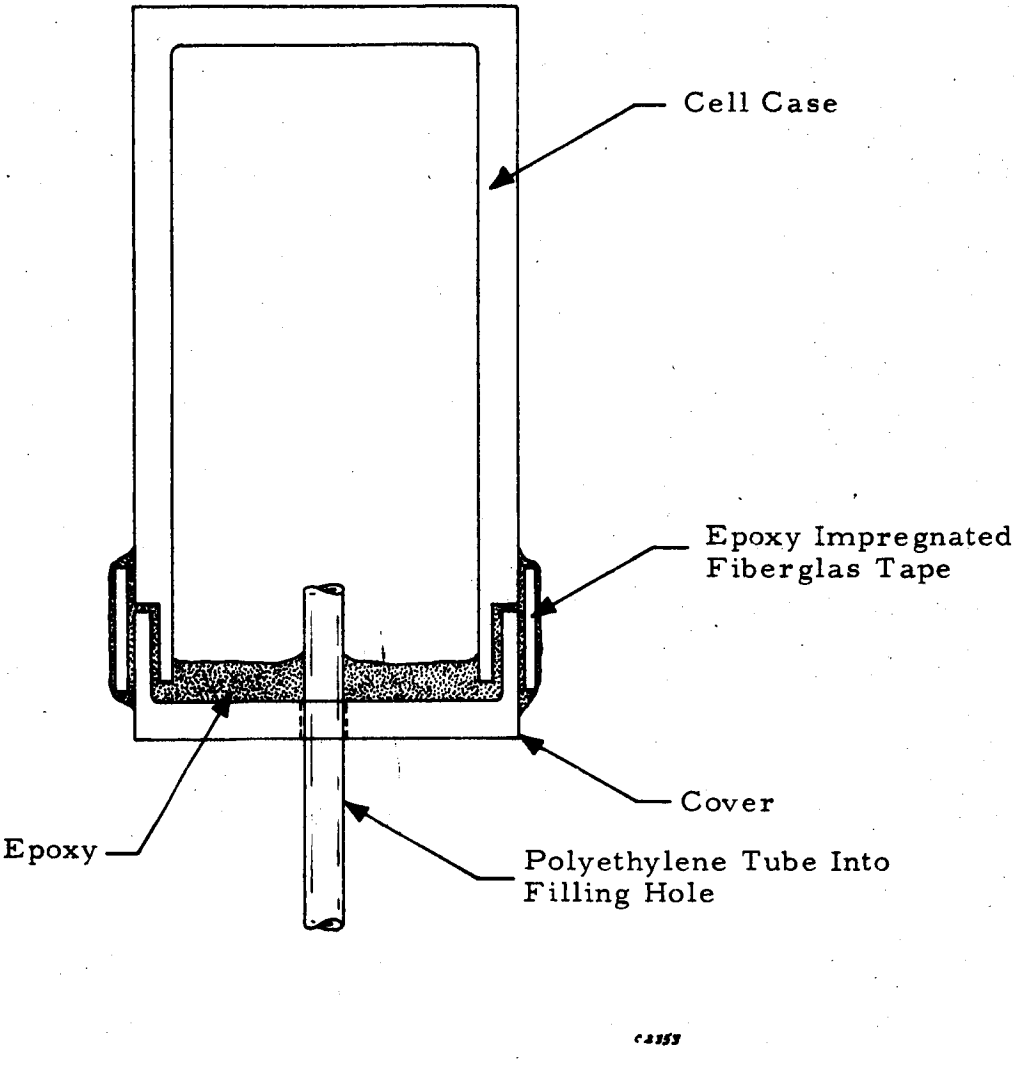



Figure 37. Cover-to-Case Sealing

After curing the epoxy at 120°C for two to three hours, the assembly was subjected to 145°C. After 10 hours, it developed a leak around the pressure fitting in the center hole at about 16 psig.

The test was repeated more carefully. The case was partly filled with 45% KOH, fitted with a pressure gauge and placed in an oven at 145°C. After holding a constant pressure of 14 psig for 20 hours, it developed a minute leak and the pressure gradually dropped to 0 over the next 20 hours. A minute crack was found on the junction line.

This type of seal does not appear to be reliable for several reasons: The cases and covers tested were machined out of polysulfone sheet. This operation introduces stresses in the material and the sterilization temperature certainly enhances its aptitude to crazing. On the other hand, the epoxy sealant does not provide a good surface bond to polysulfone, mainly at high temperature and in an alkaline medium. The pressures encountered during sterilization are not so high as to promote a catastrophic mechanical failure of the seal. The failure is more related to a wear-out or fatigue of the bonded and bonding materials, due to prolonged exposure to high temperature and alkaline medium.

Rather than trying to improve this approach on this limited program, it was decided to scan rapidly other alternatives.

### 6.3.2 Ultrasonic Welding

In order to eliminate the effect of machining, tests were carried out on molded five ampere-hour cases and covers available from another NASA program.[1]

A horn for ultrasonic welding was fabricated to fit the molded five ampere-hour case and cover. Several polysulfone cases were welded at various settings (three cases per setting):

| Identification | Weld Time (sec) | Hold Time (sec) | Horn Pressure (lbs) |
|---|---|---|---|
| #1 | 1.25 | 0.50 | 60 |
| #3 | 1.00 | 0.50 | 60 |
| #5 | 0.80 | 0.50 | 69 |

The cases were pressure-tested in succession as follows: (Any subsequent test was done only on cases having passed the previous test.)

1. Pressure tested at 10 psig for 5 minutes at 25°C.
2. Pressure tested at 50 psig for 5 minutes at 25°C.
3. Submitted to 145°C ambient for 16 hours, then pressure tested at 50 psig for 5 minutes at 25°C.

At this point all cases were potted with epoxy (Allbond) along all the welded seams to plug leakage paths. The epoxy would be used as a sealant rather than an adhesive since the ultrasonic weld seemed to offer a strong joint, but possibly an incomplete seal.

The cases were put back on test as follows:

4. Pressure tested at 50 psig for 5 minutes at 25°C.
5. Submitted to 145°C ambient for 16 hours, then pressure tested at 50 psig for 5 minutes at 145°C.

Results

| Weld Identification | Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|---|
| | | Without Epoxy | | | With Epoxy | |
| | | 1 | 2 | 3 | 4 | 5 |
| #1 | 11 | f | | | P | f |
| | 12 | P | f | | P | f |
| | 13 | P | P | | P | P |
| #2 | 21 | P | f | | P | f |
| | 22 | f | | | f | |
| | 23 | f | | | P | P |
| #3 | 31 | f | | | f | |
| | 32 | f | | | f | |
| | 33 | f | | | f | |

f = failed; P = passed; Blank = not done.

The testing procedure was made more stringent and modified as follows:

Test #1: 25°C

(a) 50 psig for five minutes

(b) If it leaks, use potting and retest

Test #2: 145°C, 50 psig for five minutes

Test #3: 145°C, for 108 hours

(a) 50 psig for five minutes

(b) 30% KOH partially filled and sealed

Polysulfone – Weld time was varied from 1.0 second to 1.35 seconds. A summary of the tests run on polysulfone cases is shown in Table X.

Evaluation of ultrasonic weld on polysulfone cases is as follows (percentage of success):

| Weld Time | Test #1 | Test #2 | Test #3 |
|---|---|---|---|
| 1.0 sec. | 73% | 75% | 50% |
| 1.25 sec. | 81% | 15% | 0% |
| 1.35 sec. | 0% | | |

It appears therefore that ultrasonic welding on polysulfone cases as presently designed will not be more than 50% reliable. However, the data necessarily limited are not conclusive and must be repeated on several cases.

Polyphenylene Oxide – Ultrasonic welding was also carried out on cases molded out of polyphenylene oxide (PPO). Two types were considered:

1. Natural PPO (grade 534-801) – Results are shown in Table XI. Out of five cases, five passed through successive tests #1, #2, and #3, which classifies the weld as 100% successful under sterilization conditions.
2. Filled PPO (grade 541) – Results are shown in Table XII. The sterilization test (#3) was only 66% successful, regardless of the weld time.

## TABLE X

## ULTRASONIC WELDING ON POLYSULFONE CASES

| Weld Time | Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|---|
| | | 1 | | 2 | 3 | |
| | | (a) | (b) | | (a) | (b) |
| 1.0 sec. | 3.1 | f | f | | | |
| | 3.2 | f | f | | | |
| | 3.3 | f | P | P | | f |
| | 3.4 | f | f | | | |
| | 3.5 | f | P | P | P | |
| | 3.6 | f | P | f | | |
| | 3.7 | f | P | P | P | |
| | 3.8 | f | P | f | | |
| | 3.9 | f | P | P | f | |
| | 3.10 | f | P | P | P | |
| | 3.11 | f | P | P | f | |
| 1.25 sec. | 1.1 | f | P | f | | |
| | 1.2 | f | P | f | | |
| | 1.3 | P | | P | f | |
| | 1.4 | P | | P | | f |
| | 1.5 | f | P | f | | |
| | 1.6 | f | f | | | |
| | 1.7 | f | P | f | | |
| | 1.8 | f | P | f | | |
| | 1.9 | f | P | f | | |
| | 1.10 | f | P | f | | |
| | 1.11 | f | f | | | |
| | 1.12 | f | P | f | | |
| | 1.13 | f | P | f | | |
| | 1.14 | f | P | f | | |
| | 1.15 | f | P | f | | |
| | 1.16 | f | f | | | |
| 1.35 sec. | 6.1 | f | f | | | |
| | 6.2 | f | f | | | |
| | 6.3 | f | f | | | |
| | 6.4 | f | f | | | |
| | 6.5 | f | f | | | |

f = Failed
P = Passed

## TABLE XI

## ULTRASONIC WELDING ON NATURAL PPO (GRADE 534-801)

| Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|
| | 1 | | 2 | 3 | |
| | (a) | (b) | | (a) | (b) |
| 7.1 | f | P | P | P | |
| 7.2 | P | | P | P | |
| 7.3 | P | | P | P | |
| 7.4 | f | P | P | P | |
| 7.5 | P | | P | P | |
| % success | 100% | | 100% | 100% | |

Weld Time: 1.25 sec.

Hold Time: 0.50 sec.

f = Failed

P = Passed

TABLE XII

ULTRASONIC WELDING ON
FILLED PPO (GRADE 541)

| Weld Time | Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|---|
| | | #1 | | #2 | #3 | |
| | | (a) | (b) | | (a) | (b) |
| 1.3 sec. | 8.1 | P | P | P | P | |
| | 8.2 | f | P | P | P | |
| | | f | P | P | f | |
| | % Success | 100% | | 100% | 66% | |
| 1.5 sec. | 9.1 | f | P | P | P | |
| | 9.2 | P | | P | f | |
| | 9.3 | P | | P | P | |
| | % Success | 100% | | 100% | 66% | |

f = Failed

P = Passed

Comments: Failure was mainly due to case bulging at 145°C under continuous pressure and consequent stress on the stress joint. Actually, the cells in an assembled battery are constrained rigidly and tightly and no bulging or distortion of the plastic is possible. Restraining the cell case between flat pieces of steel will simulate this condition and may increase the chances of success. This procedure was applied to all subsequent tests.

The limited number of tests give an indication that the ultrasonic welding may solve the cover-to-case seal problem. An ultrasonic welder was therefore acquired. It is recommended to further the investigation beyond the point of statistical testing at various settings of the welder and on various materials on molded cases on hand. A re-evaluation of the case and cover design for ultrasonic welding should be made with the view of optimizing the ultrasonic energy directors and their relative location on the case and cover.

### 6.3.3 Hot-Gas Welding

Hot-gas welding was started on polysulfone cases. A few cases were sent to four different vendors to establish the capability of their equipment. Table XIII gives a summary of the tests run through the procedure outlined in Section 6.3.2.

Considering that the samples were not welded with the proper plastic welding rods, the results cannot be conclusive. Polysulfone and PPO rods, 1/8" thick, were ordered and used on a new batch of cases, welded by one selected welder.

The results are given in Table XIV. Although the last test, 3b, where KOH filled sealed cases were maintained at 145°C for 108 hours, was not entirely successful on polysulfone cases, the amount of water lost was negligible. The cases were brought down to room temperature and pressurized at 50 psi for 5 minutes. No leak occurred. After dissection, the failure was traced to minute crazing around the center hole which was sealed with a threaded pipe screw.

In the PPO cases, the failure was caused by delamination of the covers along the lip of the cases. This may have occurred during the

## TABLE XIII

## HOT-GAS WELDING ON POLYSULFONE CASES

| Welder | Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|---|
| | | 1 | | 2 | 3 | |
| | | (a) | (b) | | (a) | (b) |
| A | A 1 | f | f | | | |
| | A 2 | f | P | f | | |
| | A 3 | P | | P | | P |
| | A 4 | P | | P | | f |
| B | B 1 | | | | | |
| C | C 1 | P | P | P | P | |
| D | D 1 | P | P | P | P | |
| | D 2 | P | P | f | f | |

f = Failed

P = Passed

TABLE XIV

HOT-GAS WELDING (WELDER A)

| Material | Case Identification | Test Identification | | | | |
|---|---|---|---|---|---|---|
| | | 1 | | 2 | 3 | |
| | | a | b | | a | b |
| PS | 1 | f | P | P | | * |
| | 2 | P | P | P | | * |
| | 3 | f | P | P | | * |
| | 4 | f | P | P | | * |
| PPO | 1 | f | P | f | | |
| | 2 | f | P | f | | |
| | 3 | f | P | f | | |
| | 4 | f | P | f | | |

f = failed

P = passed

*Average weight loss = 0.5 g of liquid after 108 hours at 145°C.

intense heat of the welding or during the prolonged heat of the sterilization procedure. It may be traceable also to poor molding.

The method of hot-gas welding should be investigated further. Alone or in combination with ultrasonic welding, it will increase the reliability of the case-to-cover seal.

### 6.3.4 Comments

Based on the data obtained on various materials and sealing methods, natural PPO (grade 534-801) and ultrasonic welding were selected for the cases and covers of the final cells.

## 7.0 EVALUATION OF SELECTED DESIGN

The selected design described in Paragraph 5.2 consists of one negative wafer assembly, sandwiching one negative electrode and sealed with a neoprene cement, and two positive electrodes wrapped in KT paper. The cell uses 45% KOH as electrolyte. The inorganic separator used is 3420-25. A large number of identical cells were tested under various conditions.

### 7.1 Temperature Tests

Cells were tested at various discharge rates and temperatures to establish their capability. After sterilization, formation and recharge, the cells were run for two cycles in groups of two, respectively, on each of the following rates: 0.1 A, 0.5 A, 1 A, 2 A and at each of the following temperatures: 32°F, 77°F and 125°F.

Tables XV and XVI give a summary of the data — average output and average plateau voltage. Only high rates (1 A and 2 A) at 32°F gave erratic voltage, although the output was acceptable. Discharge curves are presented in Figures 38 through 46. Figures 47 and 48 show a family of parametric curves for average capacity and plateau voltages as functions of temperature.

The spectrum temperature-voltage-capacity given here is a mere indication of the capability of the present cell design. It does not, however, give the optimum representation of the performance of a heat-sterilizable cell designed for a specific mission. The present program centered around a feasibility study rather than a design engineering problem.

### 7.2 Cycling Tests

Eight cells taken from the temperature tests were submitted to automatic cycling (1/2-hour, 600 mA discharge and 1/2-hour, 720 mA charge) as done previously on other cells. The average range of cycles obtained without adjustment (electrolyte addition, electrical circuitry, ratio of charge to discharge) was 170 to 240 cycles. Figure 49 depicts discharge and charge cycles at various times for a cell which was capable of 450 cycles. In a primary cell, one useful cycle is required. The present separator lends itself to a secondary mode and is capable of sustaining several charge and discharge cycles. Therefore, the cycling capability shown here can be a measure of the reliability of the system.

TABLE XV

CAPACITY (AVERAGE OF TWO CONSECUTIVE CYCLES FOR EACH CELL)

| Temperature | Current | | | |
|---|---|---|---|---|
| | 0.1 A | 0.5 A | 1.0 A | 2.0 A |
| 32°F | 2.10 Ah | 1.90 Ah | 1.70 Ah | 1.50 Ah |
| | 2.00 Ah | 1.75 Ah | 1.85 Ah | 1.80 Ah |
| 77°F | 2.60 Ah | 2.00 Ah | 2.50 Ah | 2.50 Ah |
| | 2.65 Ah | 2.30 Ah | 2.50 Ah | 2.40 Ah |
| 125°F | 2.50 Ah | 2.20 Ah | 2.10 Ah | 2.30 Ah |
| | 2.35 Ah | 2.25 Ah | 2.30 Ah | 2.20 Ah |

TABLE XVI

AVERAGE PLATEAU VOLTAGE
(TWO CELLS – TWO CYCLES)

| Temperature | Current | | | |
|---|---|---|---|---|
| | 0.1 A | 0.5 A | 1.0 A | 2.0 A |
| 32°F | 1.40 V | 1.00 V | Erratic | Erratic |
| 77°F | 1.45 V | 1.28 V | 1.20 V | 1.00 V |
| 125°F | 1.50 V | 1.47 V | 1.20 V | 1.02 V |

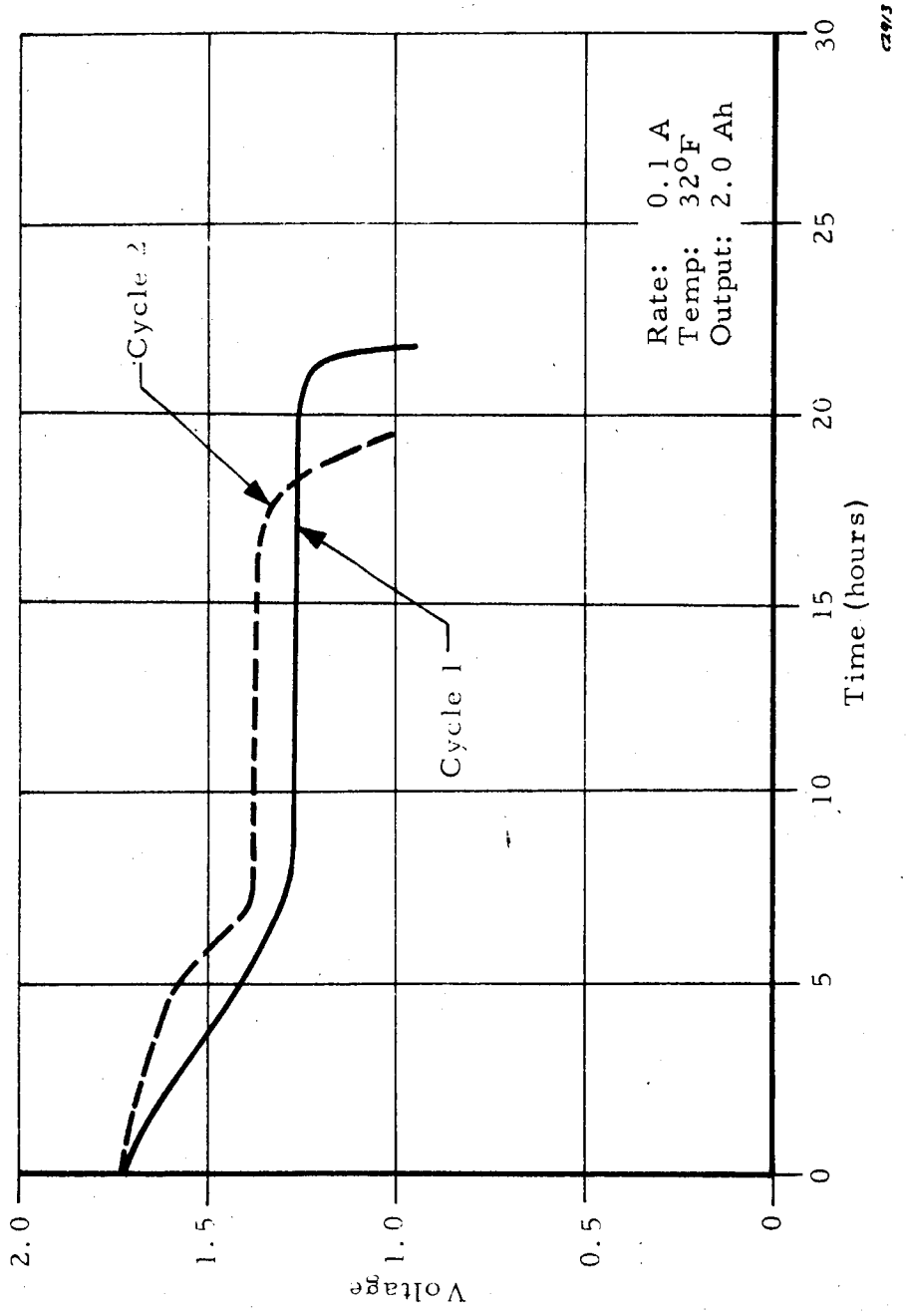



Figure 38. Discharge Curves After Sterilization (Cell A-56-15)

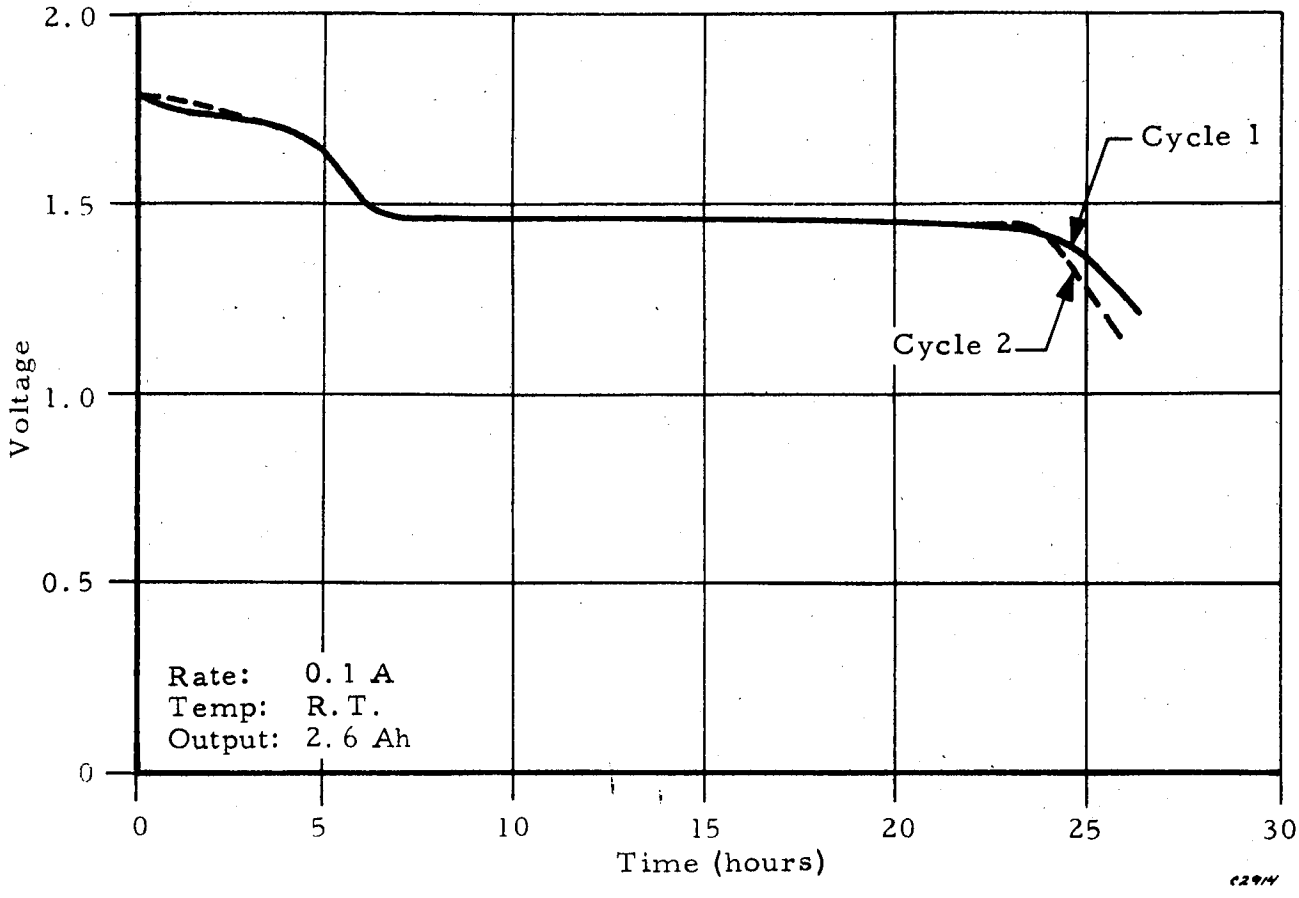



Figure 39. Discharge Curves After Sterilization (Cell A-56-7)

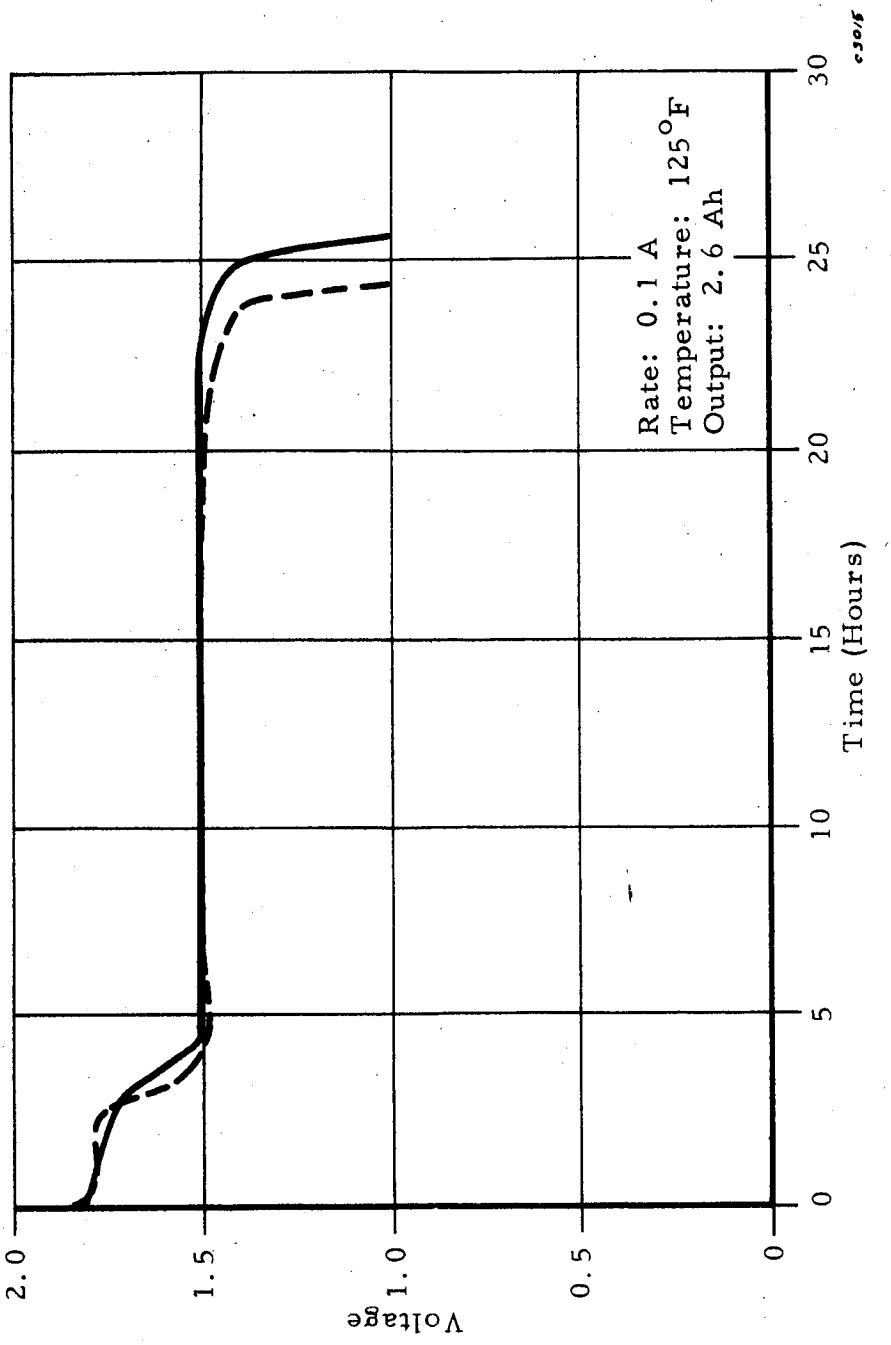



Figure 40. Discharge Curves After Sterilization (Cell A-56-23)

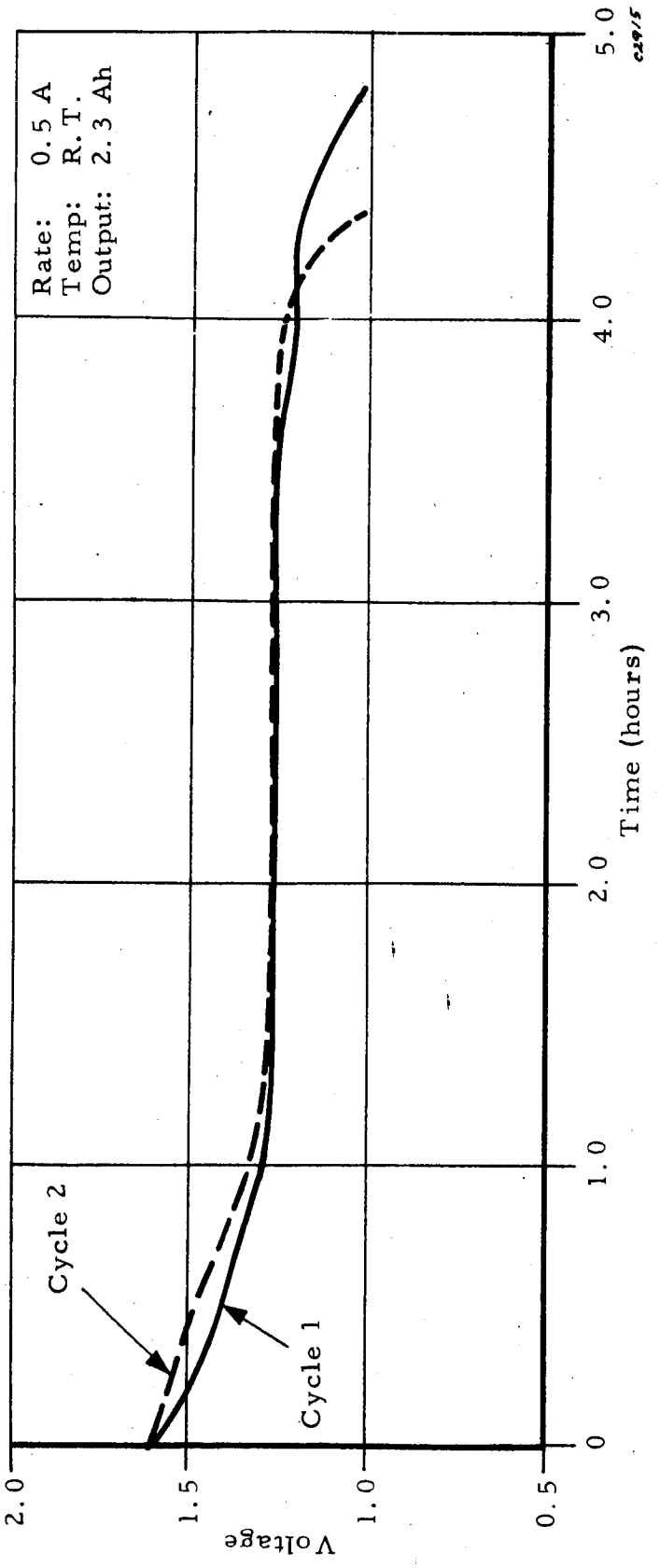



Figure 41. Discharge Curves After Sterilization (Cell A-56-5)

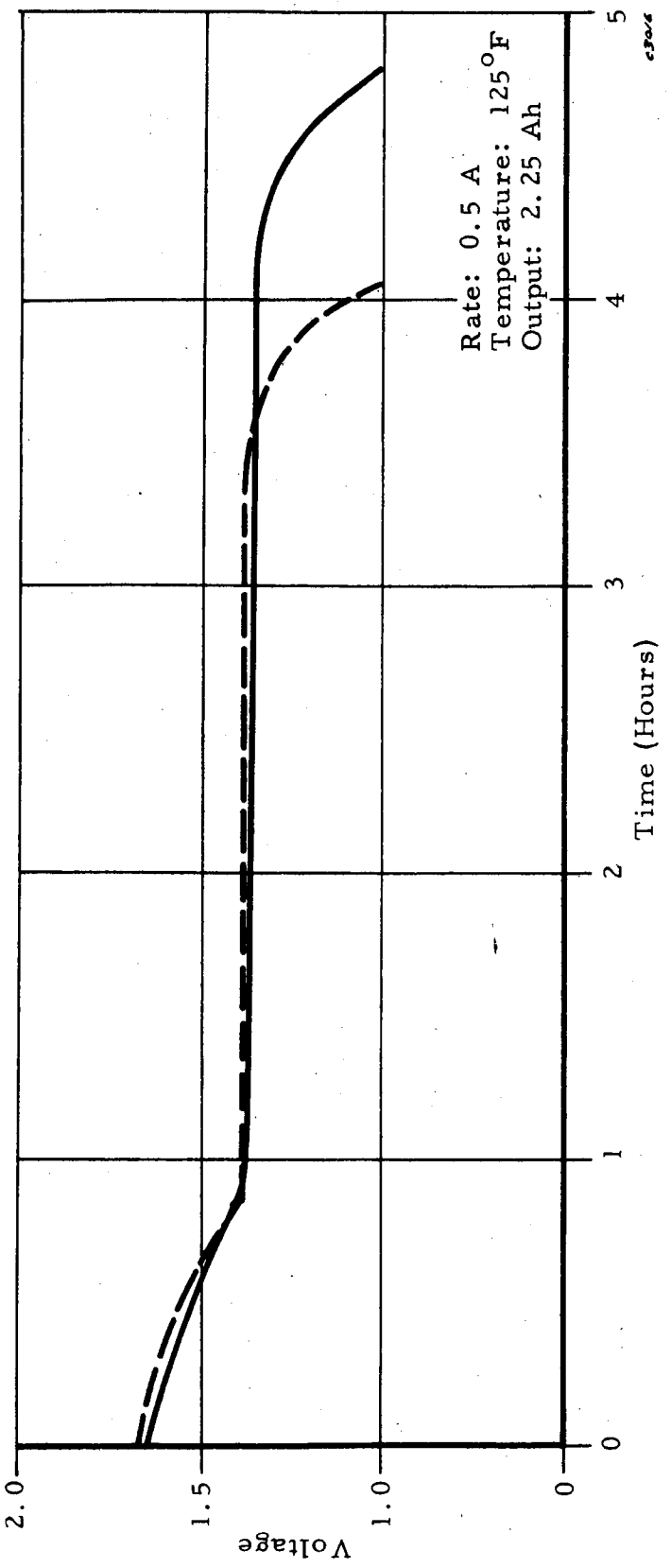



Figure 42. Discharge Curves After Sterilization (Cell A-56-22)

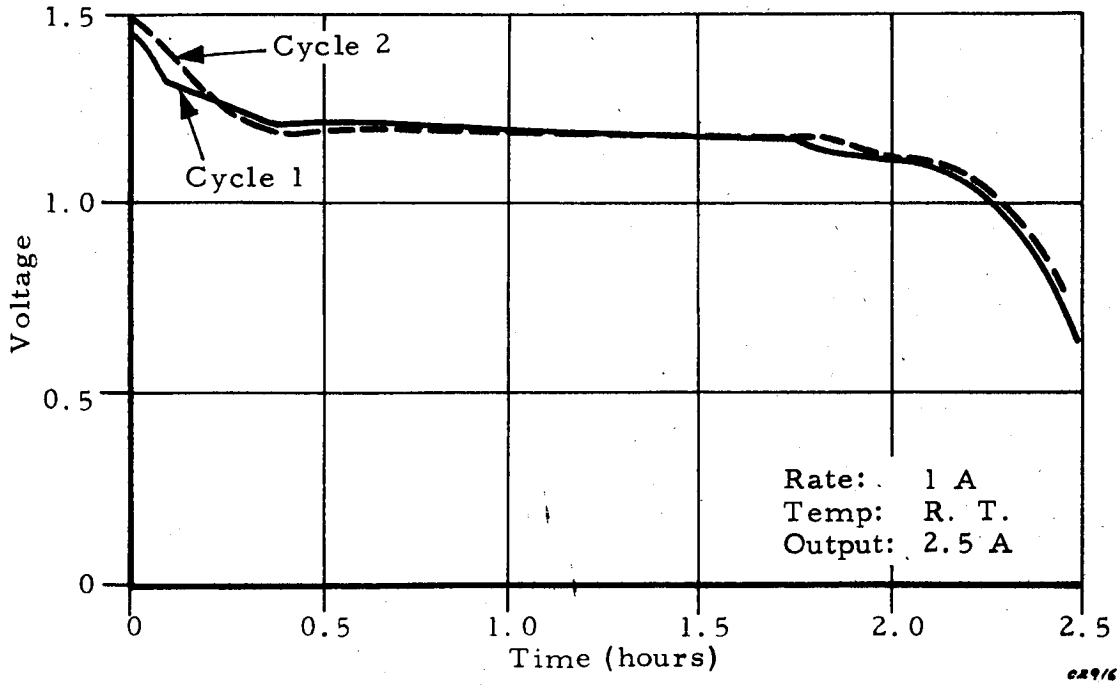



Figure 43. Discharge Curves After Sterilization (Cell A-56-4)

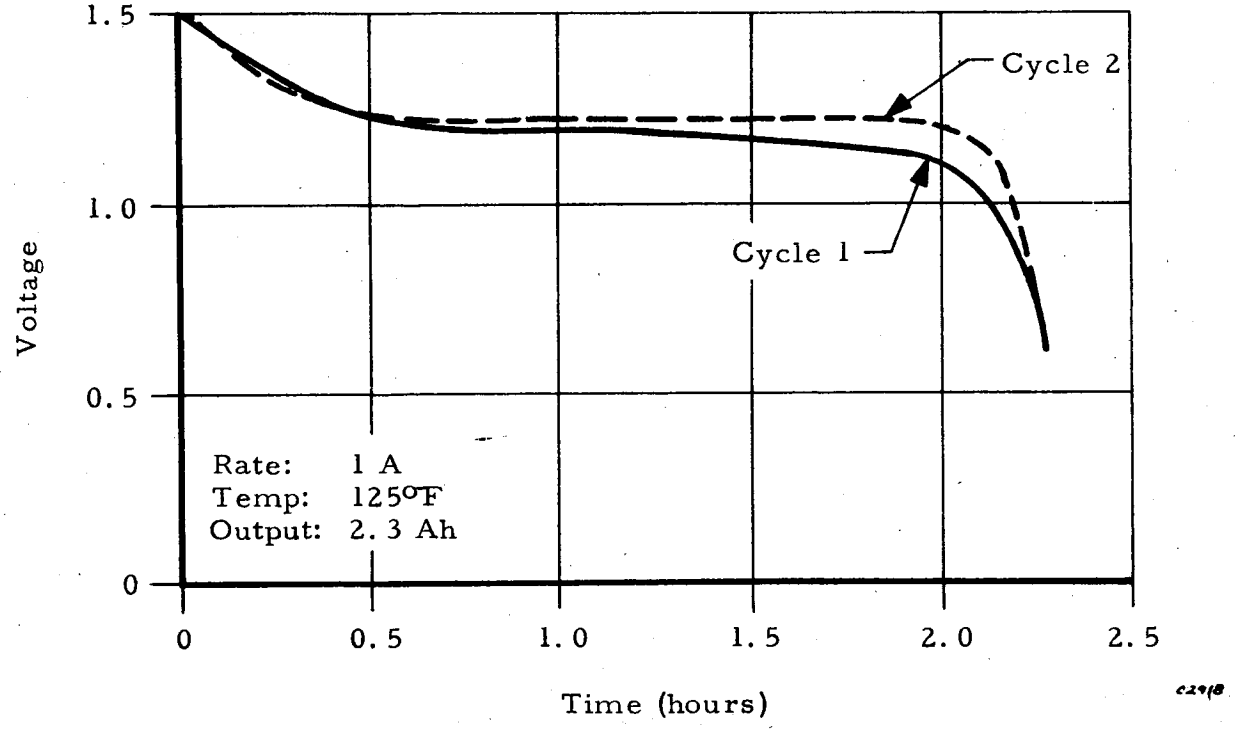



Figure 44. Discharge Curves After Sterilization (Cell A-56-17)

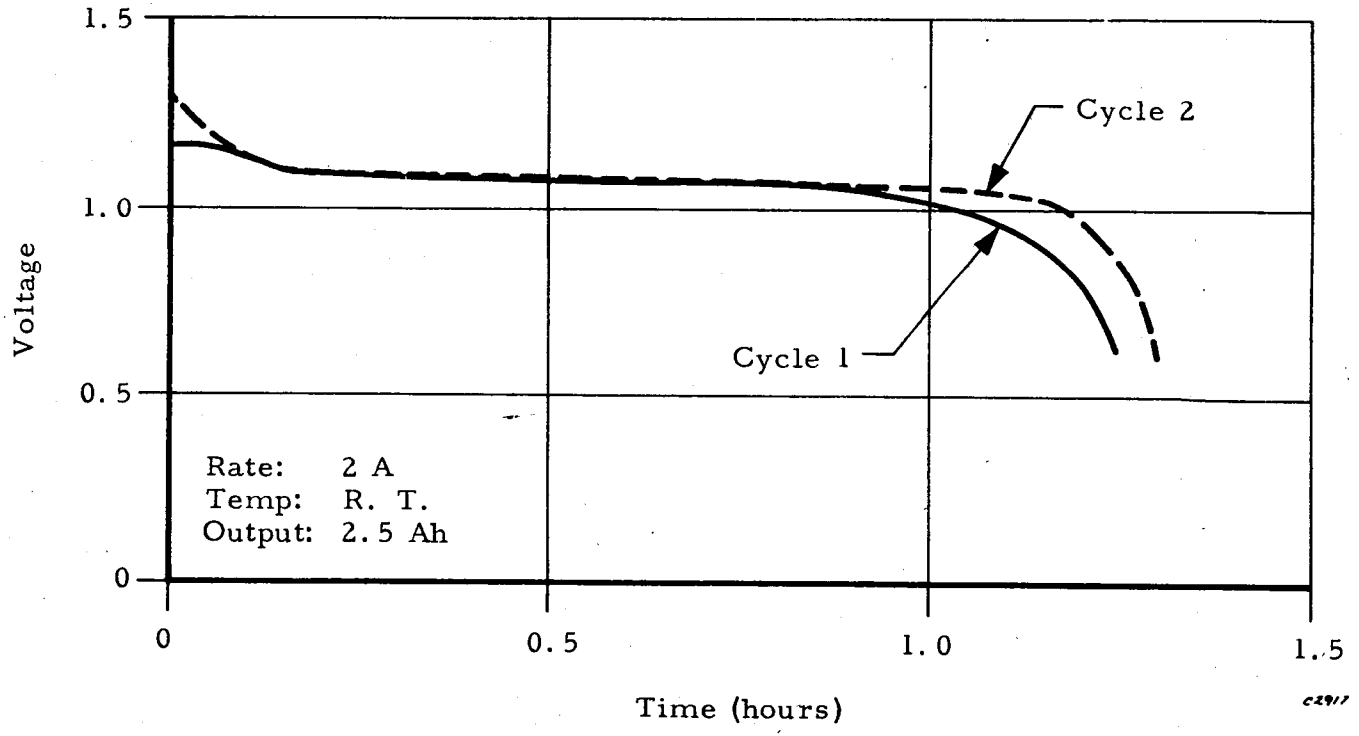



Figure 45. Discharge Curves After Sterilization (Cell A-56-1)

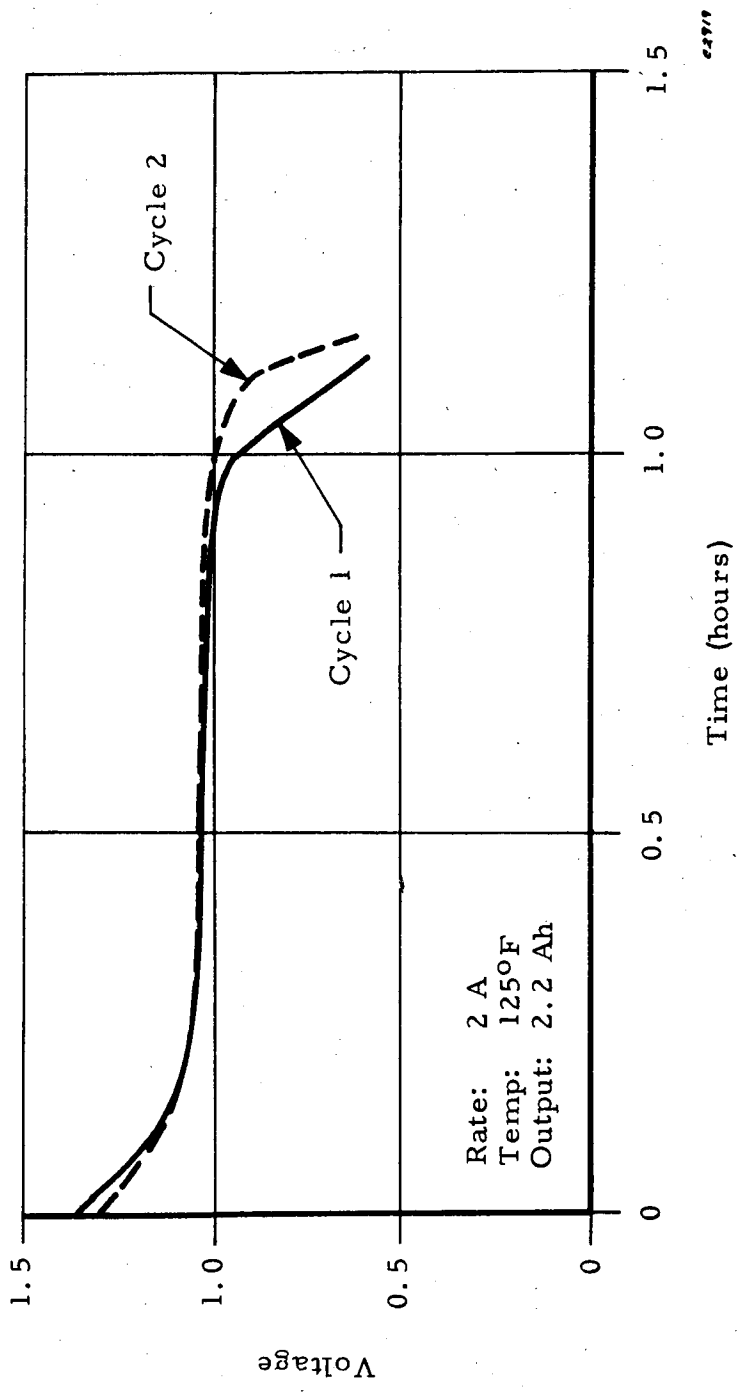



Figure 46 Discharge Curves After Sterilization (Cell A-56-19)

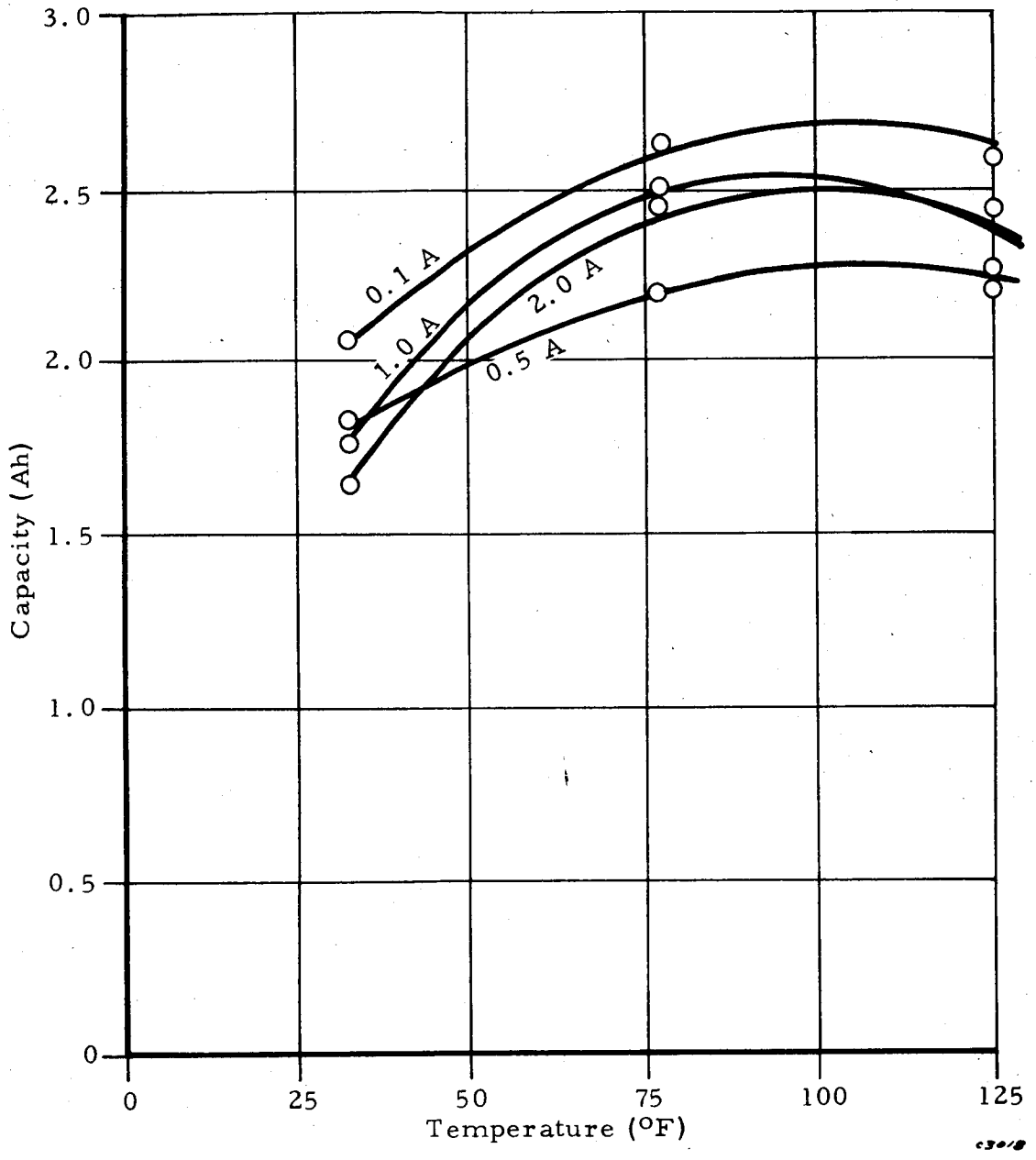



Figure 47. Average Capacity Vs. Temperature For Discharge at Various Rates

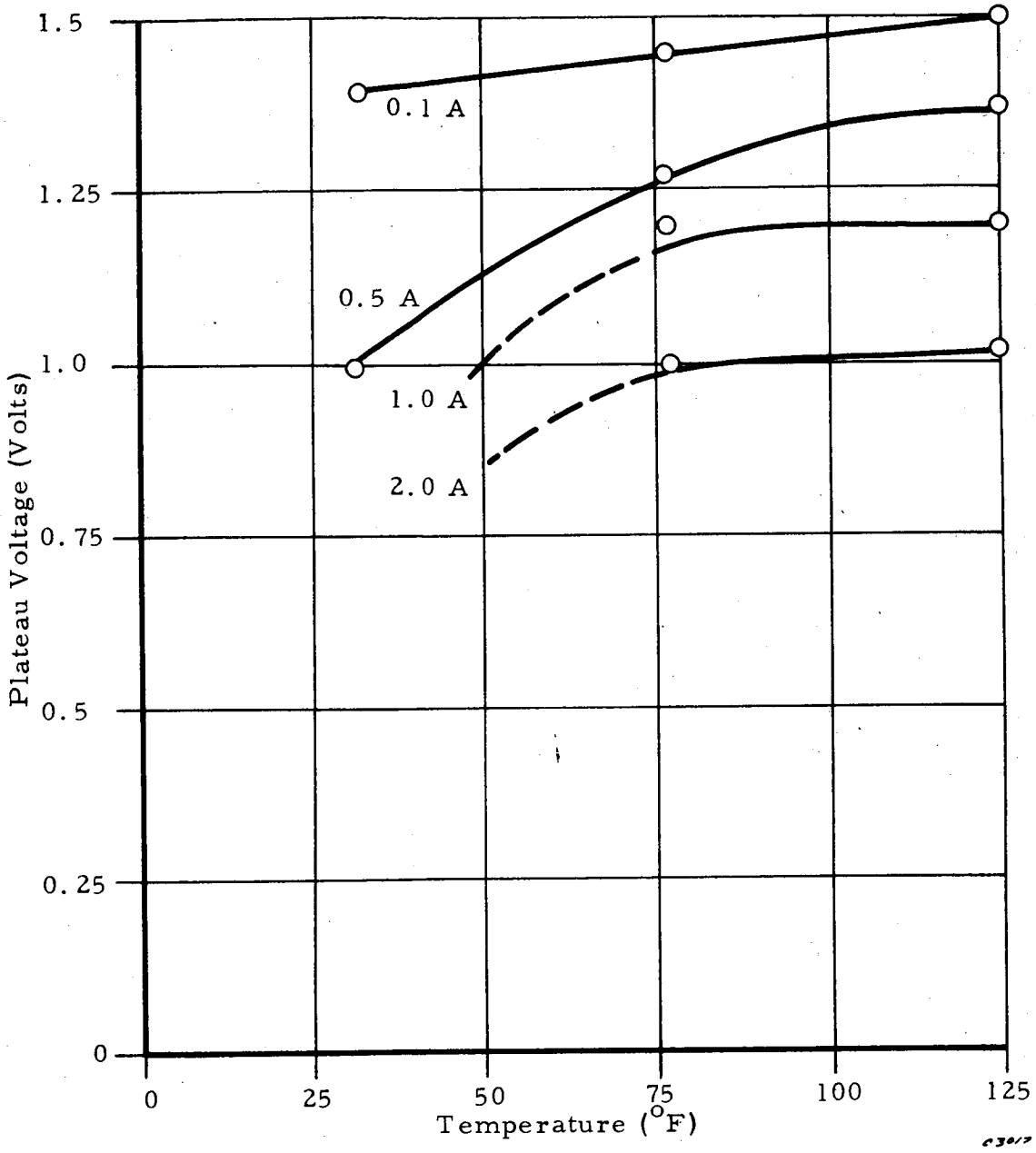



Figure 48. Average Plateau Voltage Vs. Temperature For Discharge at Various Rates

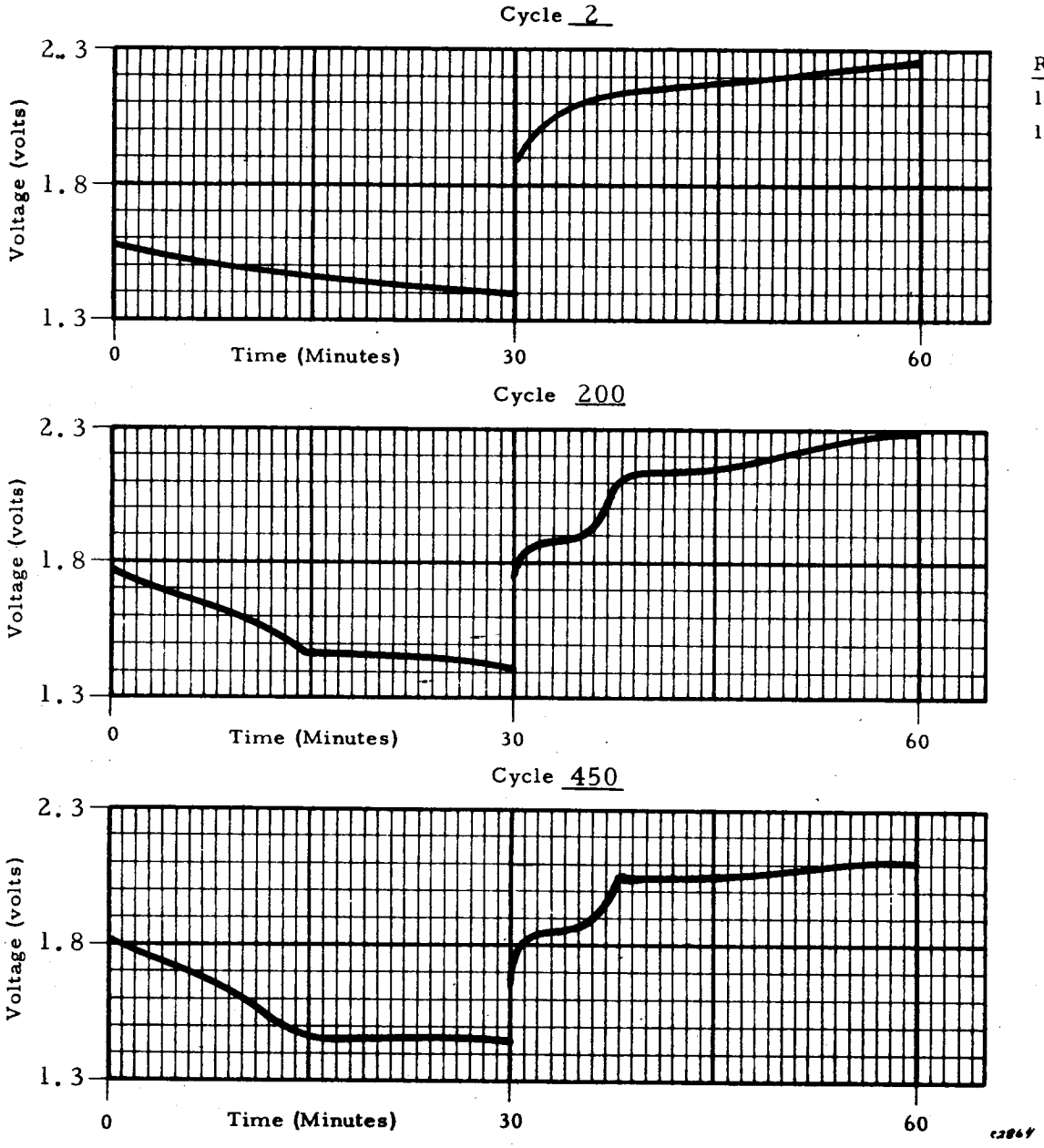



Figure 49. Auto Cycles After Sterilization

## 7.3 Environmental Tests

Ten cells were assembled in PPO cases, ultrasonically welded and submitted to sterilization in sealed chambers, then divided in the following groups.

A. Two cells were simply charged and discharged and used as controls; no environmental tests were performed.

B. Four cells were left unformed and were submitted to the environmental tests — vibration, shock, acceleration, as per NASA specifications.

C. Four cells were fully charged and were submitted to the same environmental tests.

The results are given in Table XVII.

1. All eight cells passed the environmental tests without physical damage.
2. All four charged cells maintained their OCV (1.86 V) throughout the environmental tests.
3. All charged cells were discharged, all unformed cells were charged and discharged.

NOTE: All cells were charged and discharged in a sealed state. For this reason, the cut-off voltage was set relatively low (1.97 V) to avoid any undue pressure build-up during the charge when gassing occurs only at the end of charge above 2.0 V. The cells were therefore purposely undercharged.

## 7.4 Wet Stand

During the course of the program, several preliminary cells were built to establish the integrity of the system with respect to charged wet stand. This group of cells was designed with one positive electrode and two negative electrodes.

Some cells were sterilized in sealed vessels, charged and discharged, then recharged and left on stand.

A group of four cells that were treated identically except for sterilization was used as control to assess the effect of sterilization on capacity retention.

The open circuit voltage (OCV), which is a good indication of the amount of capacity retained, was checked every day.

TABLE XVII

ELECTRICAL DATA OF STERILIZED CELLS
AFTER ENVIRONMENTAL TESTS

| Cell No. | Sterilized | Charged | Environmental Tests | Charged | Discharged | Output (Ah) |
|---|---|---|---|---|---|---|
| A-58-1 | Yes | -- | No | Yes | Yes | 1.4 |
| A-58-2 | Yes | -- | No | Yes | Yes | 1.6 |
| | | | | | Average: | 1.5 |
| A-58-3 | Yes | No | Yes | Yes | Yes | 1.5 |
| A-58-4 | Yes | No | Yes | Yes | Yes | 1.4 |
| A-58-5 | Yes | No | Yes | Yes | Yes | 1.55 |
| A-58-6 | Yes | No | Yes | Yes | Yes | 1.55 |
| | | | | | Average: | 1.5 |
| A-58-7 | Yes | Yes | Yes | -- | Yes | 1.6 |
| A-58-8 | Yes | Yes | Yes | -- | Yes | 1.55 |
| A-58-9 | Yes | Yes | Yes | -- | Yes | 1.4 |
| A-58-10 | Yes | Yes | Yes | -- | Yes | 1.5 |
| | | | | | Average: | 1.5 |

NOTE: All cells were charged and discharged in a sealed state. For this reason, the cut-off voltage was set relatively low (1.97 V) to avoid any undue pressure build-up during the charge when gassing occurs only at the end of charge above 2.0 V. The cells were therefore purposely undercharged.

Table XVIII gives an analysis of the data collected on these cells. It shows the marked effect of the design on capacity retention. Although the OCV (open circuit voltage) was still good after a long period (up to 77 days tested), the capacity retention was relatively poor in the design where the zinc was left unprotected. A capacity loss of 50% and over should bring down the OCV from 1.84 V to 1.62 V if the loss is due to the silver electrode. A capacity loss combined with a good OCV retention can be due only to the zinc electrode. This is corroborated by the fact that in designs where the zinc was protected (enveloped in a wafer construction), the capacity loss was minimal.

The design selected for all subsequent tests uses one protected negative electrode and two silver electrodes. The capacity loss will be further cut down when the cells are fully sealed and trickle-charged as required during the ten-month transit period of the spacecraft. This design is also the same one selected for all temperature, cycling and environmental tests reported in the previous sections.

Sixteen cells of this design were built and divided into four groups of four cells each. After sterilization formation and recharge, they were left on charged stand and scheduled to be discharged after 1, 2, 3 and 5 months. It was decided that trickle charge will be applied if necessary.

The OCV was checked daily and was found consistently to hold 1.85 to 1.86 V. Table XIX gives a summary of the outputs after the specified wet stand time.

One typical discharge curve is given in Figure 50. It can be noticed that after 30 days the argentic silver oxide voltage is lower due to the decomposition of the argentic silver oxide with time. This may account for the small capacity loss experienced by the group of cells after 30 days. This loss, due to the self-discharge of the positive electrode, will occur in any Ag-Zn cell independently of the separator system used. This phenomenon is the basic reason for the need of a trickle charge during prolonged wet stand.

Note that discharge voltages consecutive to the argentic portion are slightly higher after wet stand. This may be due to proper electrolyte distribution and wetting after such a long exposure.

## TABLE XVIII

## PRELIMINARY WET STAND DATA AND ANALYSIS

| Design | Cell No. | Sterilized | Output (Ah) | Stand (days) | OCV (V) | Stand (days) | OCV (V) | Output (Ah) | Capacity Loss (%) | Capacity Loss per Day (%) | Average by Groups |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Silver in wafer; zinc outside | A-14-2 | x | 0.9 | 45 | 1.85 | 72 | 1.84 | 0.4 | 55 | 0.76 | 0.6 % |
| | A-46-5 | x | 1.15 | 28 | 1.86 | 55 | 1.84 | 0.9 | 22 | 0.40 | |
| | A-46-6 | x | 1.15 | 28 | 1.86 | 55 | 1.84 | 0.6 | 48 | 0.87 | |
| | A-46-7 | x | 0.9 | 28 | 1.86 | 55 | 1.84 | 0.7 | 22 | 0.40 | |
| | A-46-1 | | 1.5 | 50 | 1.86 | 77 | 1.84 | 0.6 | 60 | 0.78 | 0.9 % |
| | A-46-2 | | 1.5 | 48 | 1.86 | 75 | 1.84 | 0.3 | 80 | 1.07 | |
| | A-46-3 | | 1.3 | 50 | 1.85 | 77 | 1.84 | 0.3 | 77 | 1.0 | |
| | A-46-4 | | 1.6 | 39 | 1.85 | 66 | 1.84 | 0.8 | 50 | 0.75 | |
| Zinc in wafer; silver outside | A-54-1 | x | 1.6 | 15 | 1.86 | 42 | 1.85 | 1.40 | 12.5 | 0.30 | 0.38% |
| | A-54-4 | x | 1.45 | 13 | 1.86 | 42 | 1.85 | 1.10 | 24 | 0.57 | |
| | A-54-2* | x | 2.7* | 13 | 1.86 | 40 | 1.85 | 2.4 | 11 | 0.27 | |

*This cell was built with two silver plates.

## TABLE XIX

## WET STAND DATA

| Cell No. | Original Output (Ah) | Discharge After | Final Output (Ah) | Average Loss |
|---|---|---|---|---|
| A-55-1 | 2.5 | | 2.3 | |
| A-55-2 | 2.4 | | 2.4 | |
| A-55-3 | 2.5 | 5 Months | 2.3 | |
| A-55-4 | 2.2 | | 2.2 | |
| Average | 2.4 | | 2.3 | 4.20% |
| A-55-5 | 2.25 | | 2.40 | |
| A-55-6 | 2.25 | | 2.30 | |
| A-55-7 | 2.25 | 3 Months | 2.35 | |
| A-55-8 | 2.25 | | 2.40 | |
| Average | 2.25 * | | 2.35 | 0% |
| A-55-9 | 2.40 | | 2.10 | |
| A-55-10 | 2.45 | | 2.45 | |
| A-55-11 | 2.45 | 2 Months | 2.35 | |
| A-55-12 | 2.40 | | 2.35 | |
| Average | 2.40 | | 2.30 | 4.20% |
| A-55-13 | 2.25 | | 2.0 | |
| A-55-14 | 2.15 | | 1.9 | |
| A-55-15 | 2.30 | 1 Month | 2.2 | |
| A-55-16 | 2.25 | | 2.4 | |
| Average | 2.20 | | 2.1 | 4.50% |

*This group has been slightly undercharged.

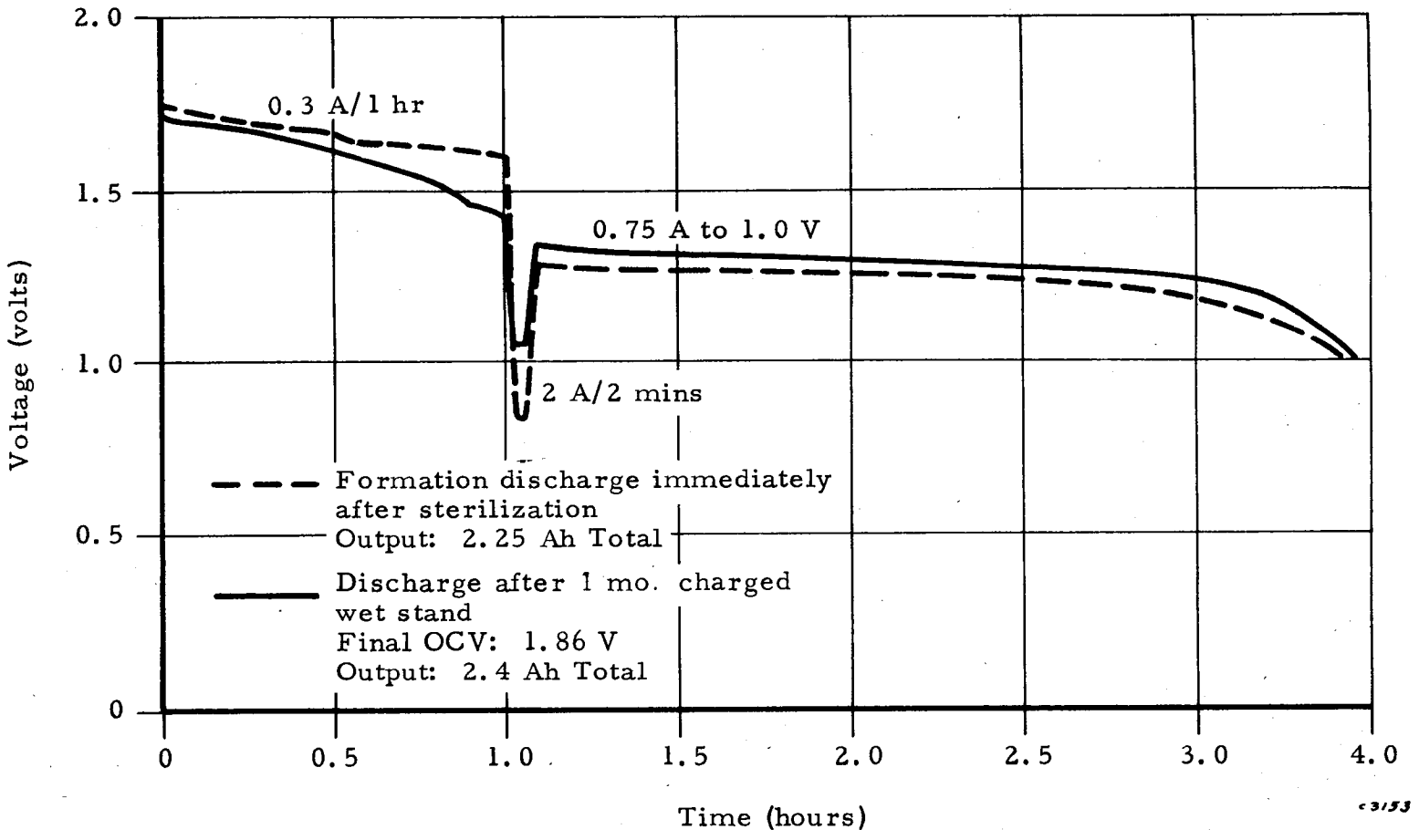



Figure 50. Discharge of Sterilized Cell Before and After 1 Month Charged Wet Stand (Cell A-55-16)

Figures 51, 52 and 53 are typical discharge curves for cells discharged after a wet stand of 2, 3 and 5 months respectively.

For all cells left on wet stand up to 5 months, the capacity loss did not exceed 5%; this value falls within the normal range of variation between different cells of the same construction or between different cycles of the same cells at the beginning of their cycle life.

It is remarkable that no trickle charge was needed at any time. It may or may not be required for periods over 5 months. However, it is expected that if the mission load does not require more than 75% of the capacity of the cell, no trickle charge would be necessary for a year.

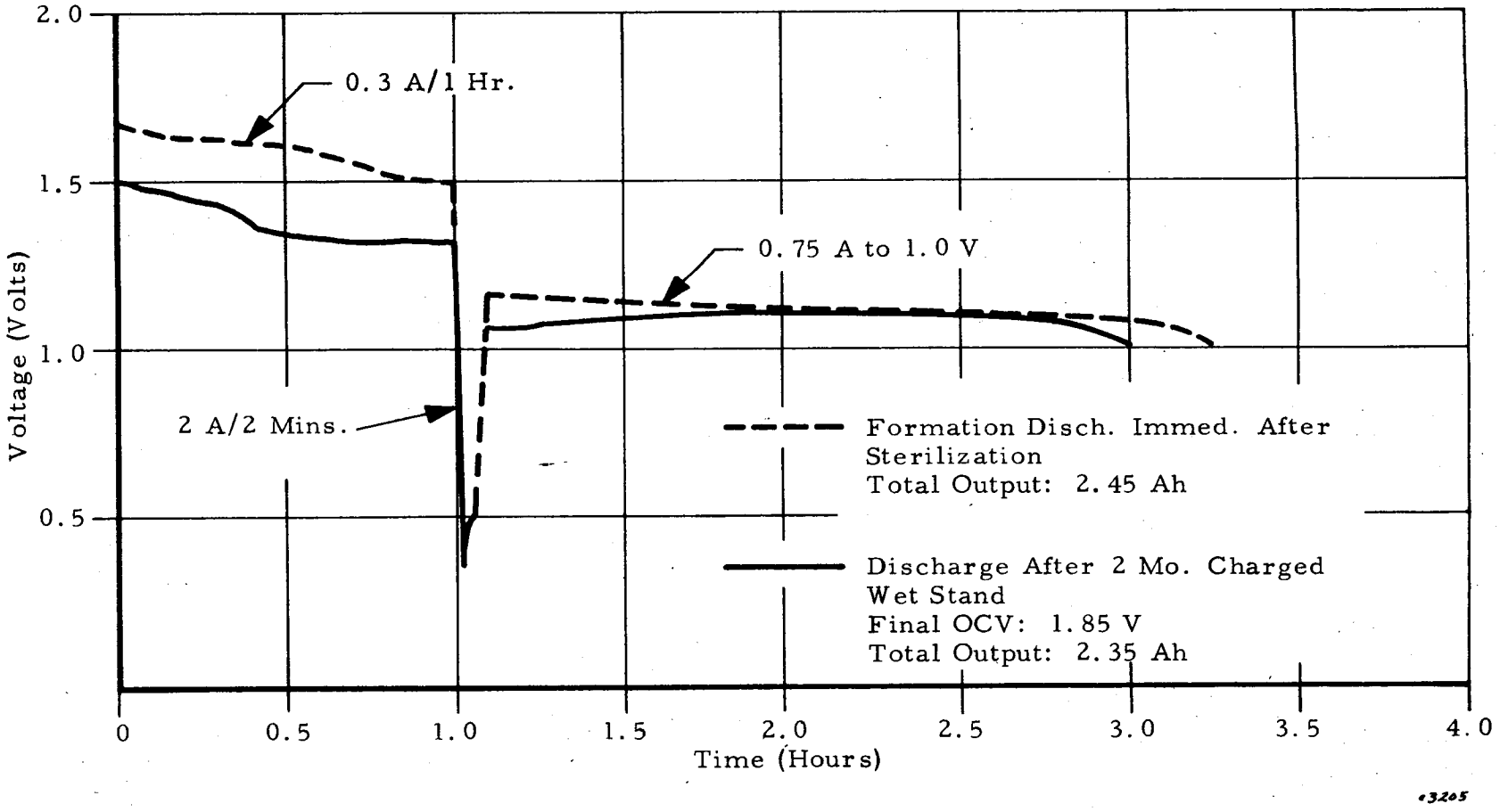



Figure 51. Discharge of Sterilized Cell Before and After 2 Month Charged Wet Stand (Cell A-55-11)

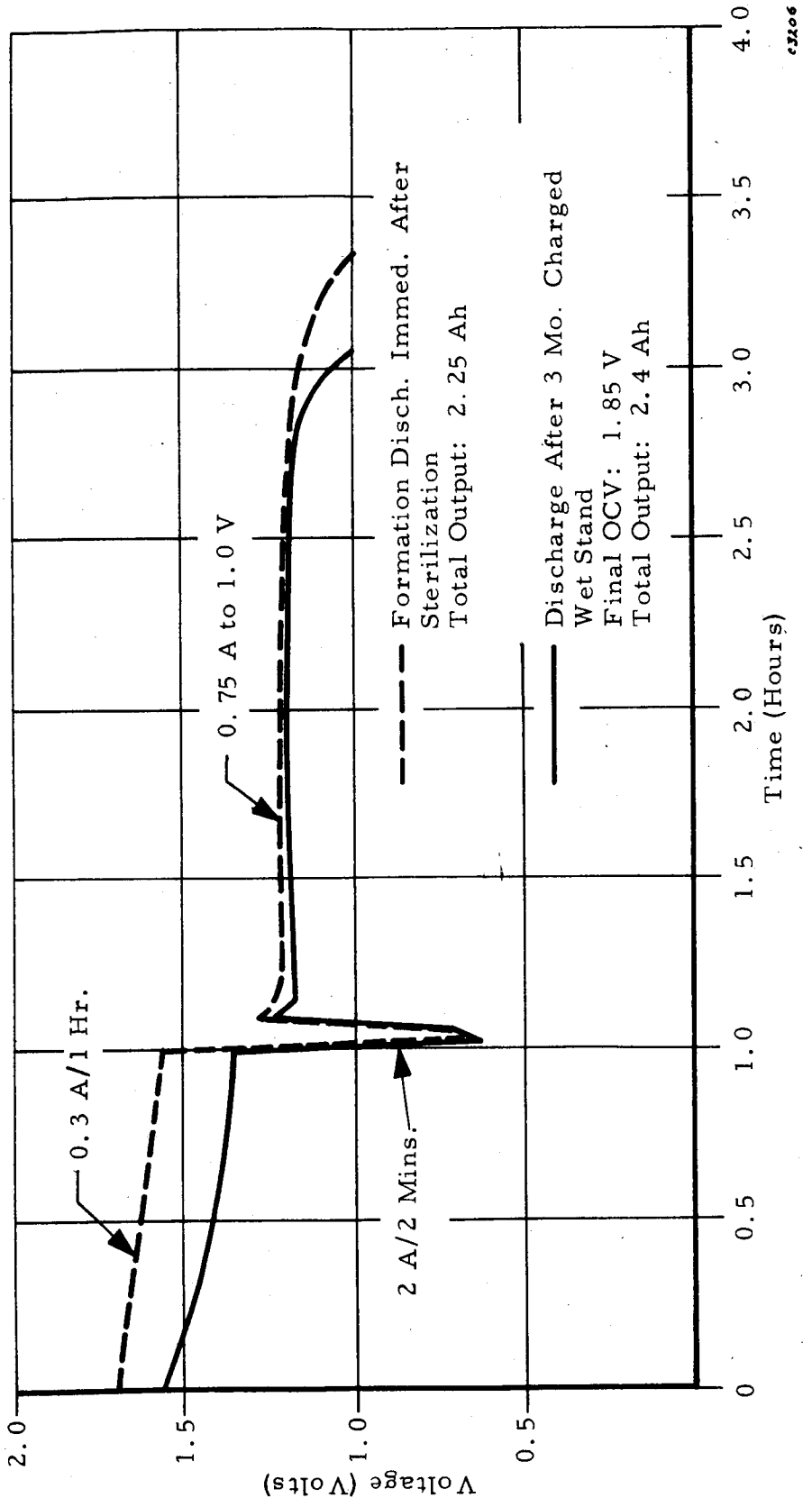



Figure 52. Discharge of Sterilized Cell Before and After 3 Month Charged Wet Stand (Cell A-55-5)

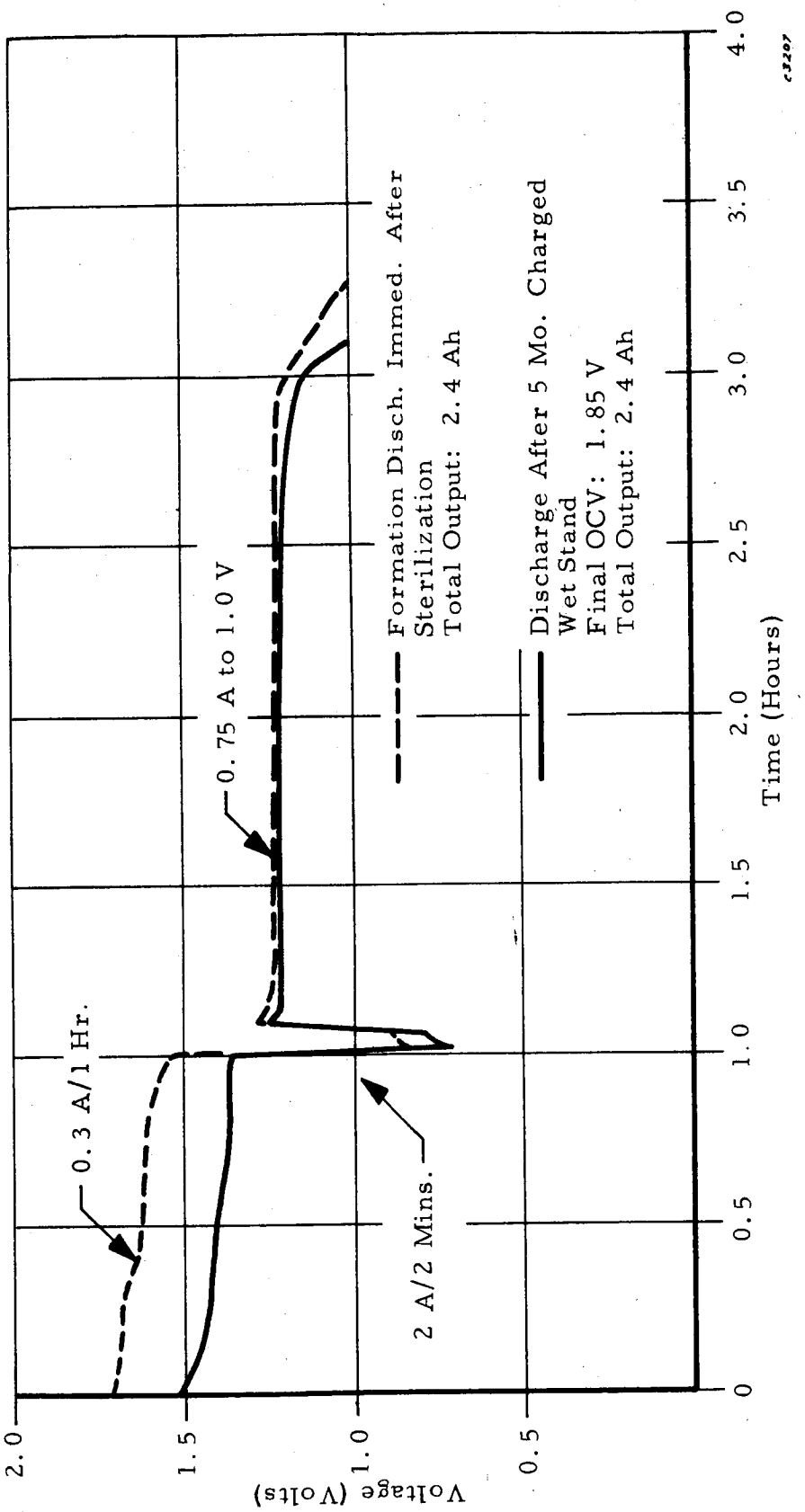



Figure 53. Discharge of Sterilized Cell Before and After 5 Month Charged Wet Stand (Cell A-55-2)

## 8.0 OPTIMIZATION FOR BATTERY DESIGN

The present design can be optimized for a battery intended for a specific application. Using approximately the same cross section and configuration developed, the following is a list of the physical characteristics of an optimized cell having the same electrical characteristics (capacity, voltage, wet stand) as obtained on the cell which evolved from this program (2 Ah).

| Cell Pack | Size | Weight |
|---|---|---|
| 2 silver electrodes | 1.5" x 1.5" x 0.015" | 7 g |
| 1 zinc electrode | 1.5" x 1.5" x 0.060" | 5 g |
| 2 separators | 1.75" x 1.90" x 0.020" | 8 g |
| Cement | | 2 g |
| KOH | | 7 g |
| Case, cover, hardware | | 30 g |
| | Total Weight: | 59 g |

Cell dimensions: 2.25" x 2.00" x 0.37" = 1.66 $in^3$
Terminal extension: 0.30"

Capacity: 2 Ah

Nominal Voltage: 1.50 V

Energy: 3 Wh

Energy density: 23 Wh/lb., 1.8 Wh/$in^3$

Battery:

18 series-connected cells (voltage 26 - 28 V)

Layout shown in Figure 54

One, two-pin connector

One relief valve

Titanium metal case and cover

Dimensions:

L x $\ell$ x H

7" x 2.5" x 3"

Volume: 52.5 $in^3$

Weight: Estimated 1200 g

Energy density: 20.5 Wh/lb., 1.03 Wh/$in^3$

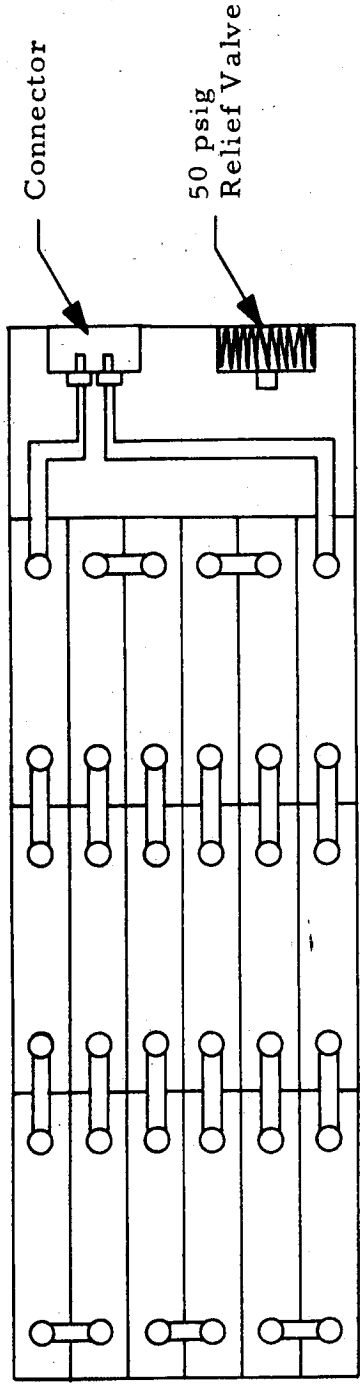



Figure 54. Battery Layout for 18 Cells of 2 Ah

## 9.0 CONCLUSIONS AND RECOMMENDATIONS

The outcome of this 12-month program was to demonstrate the feasibility of using an inorganic separator for the design of a heat-sterilizable silver-zinc cell.

Several avenues were investigated with respect to configuration and seals. This versatility enables one to select a design for specific missions in future development. All research work has been basically done.

The following important facts have been obtained:

1. An inorganic separator has been found to be capable of heat sterilization, continuous cycling and long wet stand.
2. A design concept making use of a sandwiched negative electrode (wafer design) has been satisfactory.
3. A separator edge sealant has been found capable of keeping its bonding characteristics during and after heat sterilization for very long periods of time.
4. Different types of cover-to-case seals have been established as the most likely candidates to pursue: metal welding or soldering for metal case, ultrasonic and hot-gas welding for plastic cases. Combinations can be made for more reliability, but after complete development and statistical testing, it may not be necessary.

This fundamental feasibility study shows the way to a functional design called by a specific mission. The problems likely to be encountered and investigated are the following:

a. To keep the energy densities as high as possible, a high rate capability in the order of C/1 or 2C rate may be needed in order to obtain a relatively high operating voltage. By controlling composition, porosity and thickness, an inorganic separator can be custom-made to a specific load profile.

b. The size chosen (1 or 2 Ah) may be too small for future missions. A scale-up study is in order.

c. The case sealing has advanced to the point where design engineering and large scale testing are required.

It is therefore recommended that future design work follow closely the lines developed in this program with emphasis on large scale testing to ascertain high rate capability and reliability of seals. It is possible that a trade-off may have to be made between high rate capability and long cycling capability, although recyclability at least 100 times will always be assured.

## REFERENCE

1. Arrance, F. C.; Greve, R. and Rosa, A.; Program to Develop an Inorganic Separator for a High Temperature Silver-Zinc Battery; Douglas Report SM-48461-F; Contract NAS 3-7639; Astropower Laboratory, Missile & Space Systems Division, Douglas Aircraft Company, Newport Beach, California; June 1967.

# APPENDIX

## DATA ON CYLINDRICAL CONFIGURATION

## MECHANICAL TESTING

Tests on sealing were preformed. Four cylindrical cells were assembled with models #2, 3, 5, and 6. Models #2, 3, and 6 were sealed with Teflon tape on the threads.

The positive wire lead is brought up through the center hole of the cover which is screwed onto the case, loosely revolving around the positive wire. Through this hole, the cell is then pressure-tested to determine the integrity of the seal, then the hole is plugged with solder. Models #2 and #3 leaked between the Teflon insulator and cover at one point around 10 psi.

Model #6 held its seal satisfactorily.

Model #5 (welded cover with ceramic seal) was not pressure-tested because no provision was made in the cover. Eventually a small hole may be left in the cover for filling and pressure-testing and may be plugged with solder.

Models #5 and #6 were submitted to the sterilization procedure inside a dry sealed vessel flushed with nitrogen and provided with a pressure gauge. After stabilization at 145°C, the pressures reached respectively 6.5 psig and 7 psig and remained constant throughout the entire test (108 hours). The fact that the pressure increase in the vessel (from 14.7 psia to 21.7 psia) was directly proportional within error limits to the temperature rise in degrees Kelvin (from 298°K to 418°K) proves that it was due exclusively to gas expansion and not to KOH vapor pressure, which would have been present, should the sealed cells have leaked. This was confirmed by the fact that there was no trace of alkali after sterilization on the exterior of the sealed cells and that their weights were found unchanged.

## ELECTRICAL TESTING

The test data are reported and may be used in future work if this type of seal and cell assembly is desirable and offers a definite advantage over the plastic-to-plastic seal. It may be kept as a back-up for the rectilinear configuration of a metal case.

One cell, A-47-1, was preformed and discharged (capacity 0.6 Ah, 2 A pulse voltage 0.7 V), sterilized, then recharged and discharged again

on a duty cycle (capacity 0.7 Ah, 2 A pulse voltage 0.8 V), then placed on automatic cycling on the 1/2 hour charge, 1/2 hour discharge regime. It accomplished 356 cycles; curves are shown in Figure A-1.

The other cell, A-41, was left unformed, sterilized, charged and discharged on a duty cycle (capacity 1.3 Ah, 2 A pulse voltage 0.5 V), then placed on automatic cycling. It accomplished 268 cycles.

The low voltage on the 2 A pulse is caused by the smaller separator area available in the cup design. The current density is more than twice that of the flat design. This can be easily remedied, when need be, by increasing the separator area and involves no special developmental problem, only design engineering.

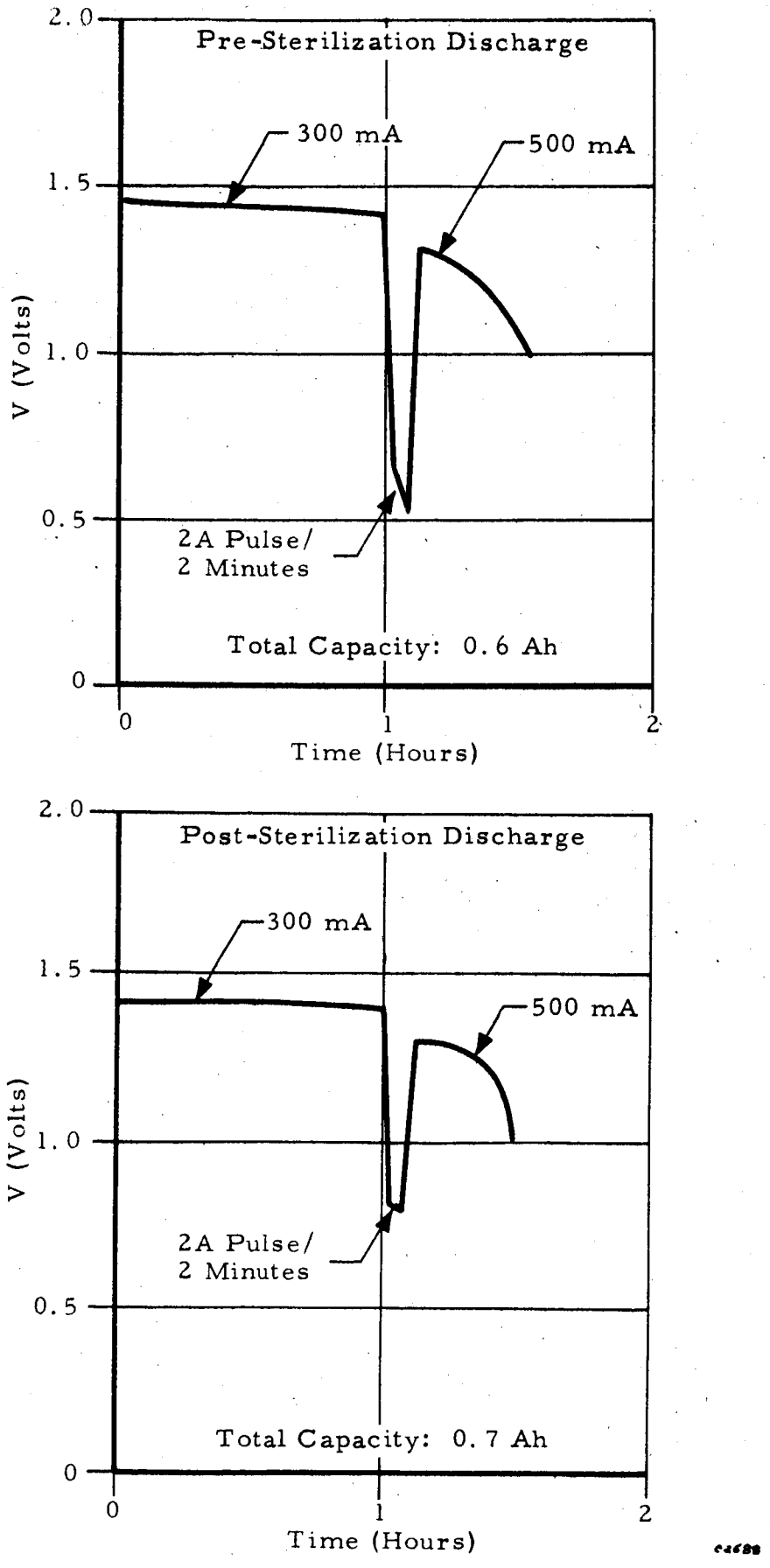



Figure A-1. Cylindrical Cell (Cell A-47-1)

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Burgess Battery Company
Freeport, Illinois
Attn: Dr. Howard J. Strauss'

Calvin College
Grand Rapids, Michigan
Attn: Prof. T. P. Dirkse

Delco Remy Division
General Motors Corporation
Anderson, Indiana
Attn: Dr. J. J. Lander

Eagle-Picher Company
P. O. Box 290
Joplin, Missouri
Attn: E. M. Morse

Electric Storage Battery Company
Missile Battery Division
Raleigh, North Carolina
Attn: A. Chreitzberg

Electric Storage Battery Company
Carl F. Norberg Research Center
Yardley, Pennsylvania
Attn: Dr. R. A. Schaefer

Electrochimica Corporation
1140 O'Brien Drive
Menlo Park, California
Attn: Dr. Morris Eisenberg

Electro-Optical Systems, Incorporated
300 North Halstead
Pasadena, California
Attn: E. Findl

Dr. Arthur Fleischer
466 South Center Street
Orange, New Jersey

General Electric Company
Schenectady, New York
Attn: Dr. William Carson
Advanced Technology Lab.

General Electric Company
Battery Products Section
P. O. Box 114
Gainsville, Florida

General Motors-Defense Research Labs.
P. O. Box T
Santa Barbara, California
Attn: Dr. C. R. Russell

Globe-Union, Incorporated
900 East Keefe Avenue
Milwaukee, Wisconsin
Attn: Dr. Warren Towle

Gould-National Batteries, Incorporated
Engineering and Research Center
2630 University Avenue, S. E.
Minneapolis 14, Minnesota
Attn: J. F. Donahue

Gulton Industries
Alkaline Battery Division
Metuchen, New Jersey
Attn: Dr. Robert Shain

Hughes Research Laboratories Corporation
Malibu, California
Attn: T. M. Hahn

ITT Federal Laboratories
500 Washington Avenue
Nutley 10, New Jersey
Attn: Dr. P. E. Lighty

Livingston Electronic Corporation
Route 309
Montgomeryville, Pennsylvania
Attn: William F. Meyers

Lockheed Missiles and Space Company
Department 65-82
Palo Alto, California
Attn: Larry E. Nelson

Mallory Battery Company
60 Elm Street
North Terrytown, New York
Attn: R. R. Clune

Midwest Research Institute
425 Volker Boulevard
Kansas City 10, Missouri
Attn: Dr. B. W. Beadle

North American Aviation, Incorporated
12214 Lakewood Boulevard
Downey, California
Attn: Burton M. Otzinger

P. R. Mallory & Company, Incorporated
Northwest Industrial Park
Burlington, Massachusetts
Attn: Dr. R. Selim

Power Sources Division
Whittaker Corporation
3850 Olive Street
Denver, Colorado 80207
Attn: Mr. John Rhyne

Radio Corporation of America
Astro Division
Heightstown, New Jersey
Attn: Seymour Winkler

Space Technology Laboratories, Incorporated
One Space Park
Redondo Beach, California
Attn: Dr. A. Krausz

Texas Instruments, Incorporated
13500 North Central Expressway
Dallas, Texas
Attn: Dr. I. Trachtenberg

Sonotone Corporation
Saw Mill River Road
Elmsford, New York
Attn: A. Mundel

Thomas A. Edison Research Laboratory
McGraw Edison Company
Watchung Avenue
West Orange, New Jersey
Attn: Dr. P. F. Grieger

Union Carbide Corporation
Development Laboratory Library
P. O. Box 6056
Cleveland, Ohio 44101
Attn: Miss C. M. Foecking

Westinghouse Electric Corporation
Research & Development Center
Churchill Borough
Pittsburgh, Pennsylvania
Attn: Dr. A. Langer

Western Company
Suite 802, RCA Building
Washington, D. C.
Attn: R. T. Fiske

Yardney Electric Corporation
40-50 Leonard Street
New York, New York
Attn: Dr. Paul Howard

Aerospace Corporation
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2350 East El Segundo Boulevard
El Segundo, California
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