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HEAT STERILIZABLE
IMPACT RESISTANT CELL
DEVELOPMENT

JET PROPULSION LABORATORY
CONTRACT NO. 951296

FINAL REPORT

on

ELECTROCHEMISTRY
of
SYSTEM

COVERING PERIOD

SEPTEMBER 30, 1967 TO MARCH 31, 1970

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ESB, INCORPORATED
THE CARL F. NORBERG RESEARCH CENTER
YARDLEY, PENNSYLVANIA

EXIDE MISSILE AND ELECTRONICS DIVISION
RALEIGH, NORTH CAROLINA

April 1970

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ABSTRACT, CONCLUSIONS AND RECOMMENDATIONS

This is the final report under "ELECTROCHEMISTRY" summarizing research carried out by the ESB Research Center at Yardley, Pa. and by the ESB electrochemist in residence at the Jet Propulsion Laboratory (JPL), Pasadena, California. It covers work since the Interim Summary Report, September 30, 1967.

Contract 951296 between the Jet Propulsion Laboratory (JPL) and ESB Incorporated called for research and development of a high energy density cell capable of heat sterilization in the unformed condition, formation decontamination with ethylene oxide, survival of launching vibration, survival of high impact shock upon landing on a distant planet, and thereafter delivery of four complete electrical discharge cycles. Governing specifications were GMP-50437-DSN-A, GMP-50198-ETS-A, XSO-30275-TST-A, 3025 B including Amendments 1, 2, and 3, 20016 C, and NASA NPC-200-3.

Objectives remaining in Electrochemistry were to reduce pressure and increase capacity of sealed, heat sterilized silver-zinc cells. Objectives undertaken later were to find support metals for impact resistant cells which were compatible with the silver-zinc system and to optimize cell parameters so that a 400 cycle (50% depth of discharge) cell might be produced.

The salient features revealed by this work are as follows: Gas pressure during formation can be controlled by a long slow preformation charge, and total charge acceptance can be improved by a partial discharge and recharge during the charge cycle. While organic materials used in cell construction are not usually responsible for capacity loss in cells with a large head space, a heat cycle for cells with small head space with the cell open before the final seal and heat sterilization generally improves capacity. Cells can be heat sterilized after one or more qualification cycles if they are "let down" properly. Zirconium is satisfactory for positive plate supports while thick silver can be used in negatives for this purpose if it be preamalgamated. Silver in the zinc electrode is least brittle when the electrode is in the charged condition.

Cell factors leading to long cycle life in heat sterilizable silver-zinc cells are: 45% potassium hydroxide saturated with zinc oxide, an absorber around the silver electrode, the use of Compound 323-43, and sufficient layers of heat sterilizable separator to prevent silver penetration. Plastic bonded zinc electrodes do not prevent shape change but may be useful in impact-resistant cells. Shims with an inverse cell taper maybe useful in controlling shape change of the zinc electrode and thereby increased cycle life.

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ELECTROCHEMISTRY

I. INTRODUCTION

The requirements for a high energy density cell (either silver-zinc or silver-cadmium) which could be heat sterilized in the sealed but unformed condition, formed, impacted as in a hard planetary landing, and function thereafter through four discharge cycles have been discussed in previous reports through the Interim Summary Report dated September 30, 1967, under JPL Contract No. 951296. The present report, which is a final summary of work completed since that date, covers work under new tasks leading to the production of long cycle life silver zinc cells and to cells with special grid support materials for high impact. This final report also contains additional work on optimization of cell parameters and the effects of organic components and sterilization on cell capacity and gas pressure.

II. OPTIMIZATION OF CELL PARAMETERS

At the time of the Interim Summary Report sealed, sterilized cells often showed unexpectedly high pressures during formation, and nearly always showed considerably diminished capacities. Further work in adjusting both the external and internal parameters of the cell to the proper values has solved these problems.

A. External Factors

Factors considered external to the cell are largely electrical and include a slow preformation charge and a charging regime which increases the capacity of silver-zinc cells containing Compound 323-43.

1. Preformation

During the formation charge of many cells it had been observed that gas formed very early in the cycle. Many times pressure continued to build up in sealed cells even after the charging current was interrupted. Thus, to minimize the amount of gas generated during formation, various charging techniques were investigated. All of them were based on a slow initial charge. First a constant potential float charge was used. It was determined that 1.40 volts was about the highest float voltage that could be used without generating gas. Cells 199 through 205, listed in Tables IA and IB, were preformed at different voltages and current limits to establish this. Varying pressures resulted with one being as high as 37.8 in. Hg. Even though the charge current decayed to a fraction of a milliampere, much gas was evolved if the normal formation charge was started before a considerable number of hours on float had elapsed. Also the problems associated with floating a battery of cells at 1.40 volts per cell made this technique undesirable. Consequently, the change to constant current was made.

It was empirically determined that a preformation charge rate of 0.55 ma per square inch was required for not less than 24 hours with 28 to 33 hours being preferred. Beginning with cell 206 through cell 210 in the above mentioned tables, a constant current mode of charge was used to establish this. The equipment did not maintain a truly constant current and at times it varied as much as 3 ma from the desired value. However, it may be observed that cells precharged at 10 ma (0.55 ma/in.²) in excess of 24 hours had the lowest pressures after the regular formation charge.

What preformation accomplishes is a slow formation of metallic mercury without the formation of metallic zinc. The long time required is probably needed to give time for the mercury to migrate to and amalgamate with the silver grid. Subsequent gas generation would then be minimized on the resulting high hydrogen overvoltage surface. If this be the correct explanation, then preamalgamation of the grid before it is made into an electrode might well accomplish the same thing without the loss of preformation time.

2. Charging Regime

It was observed that if a cell was partially discharged after reaching the end-of-charge voltage and then recharged at the normal formation rates (5.5 ma per sq. in.), a significant increase in total charge acceptance was obtained. The discharge rate, while not critical, was 100 ma per sq. in. The discharge was limited to about 20% of the rated capacity. In the present cells generally 0.9 amp-hr was removed at 1.8 amp, but the principal qualification is that the discharge cease before the cell voltage decays to the value of argentous oxide-zinc, approximately 1.60 volts.

The partial discharge and recharge not only increases the charge acceptance but also raises the capacity 10 to 15%. Also capacity variation among cells is diminished. This effect is noticeable for cells 187-188-189-190 during the fifth discharge, and for all cells during the seventh as shown in Table II. Comparing those two discharges with the sixth and eighth, the effect is obvious. For cells 199-210 (Tables IA and B) the partial cycle was run at the end of formation and the results were consistent with that reported above.

An unusual phenomenon was observed during the recharge portion of the partial cycle. If a regular silver-zinc cell (not containing Compound 323-43) is similarly discharged so that the cell potential does not decay to the argentous oxide-zinc level, and then recharge is started, the cell potential almost immediately rises to a value of 1.90 volts or higher. When cells containing Compound 323-43 are similarly treated, the charge voltage remains between the values of 1.83 and 1.89 volts for several hours and for these cells more capacity is returned than had been removed, thus accounting for the capacity increase. This partial cycle was used in subsequent work where maximum capacity characteristic of the system was desired.

3. Discharge Regime

Somewhat analogous to the two-stage charge is the two-stage discharge. It is known that if a cell be discharged at a high rate to a given voltage, additional current can be extracted from that cell at a low rate to the same cut-off voltage.

In most of this work the discharge has been accomplished in two stages. In the first step the discharge is run at 100 ma/sq. in. to the 1.30 volts cut-off and at the second step the current is dropped to 20 ma/sq. in. to the same voltage cut-off. In good cells the second step adds only about 10% or so to the total capacity, but in poor cells it can account for more than half of it (see cells 2 and 3 in Table XX for instance). This ratio of first to second step discharge has proved useful in this work.

B. Internal Factors

Because of possible interaction among internal factors affecting cell capacity and gas pressure in sealed cells, these variables are handled as a group. Cumulative capacity and pressure data are shown in Tables III, IV, and V for cells sterilized and then sealed. Variables studied included effects of electrolyte concentration, pack tightness, and per cent of Compound 323-43 in the zinc electrode. Most of the cells in the study had electrolyte concentrations of either 35% KOH or 41% KOH, both determined before saturation with ZnO. The ZnO concentrations were seventy-five gms per liter for 35% and 110 gms/per liter for 41%. Pack tightness varied from 2.1×10^{-3} to 2.6×10^{-3} inches per layer of expanded thickness of the separator. Three cells from each group of cells of similar construction were made into batteries after several cycles and cycled as batteries. Pressures and capacities for the early cycles were averages. These averages are included in Table V.

Table III gives capacity data for three electrolyte concentrations and three degrees of pack tightness.

In early cycles there does not appear to be a clear-cut advantage for either 41% KOH or 35% KOH, both saturated with ZnO. In later cycles the capacity seems to be consistently higher for 41% KOH.

Similarly, pack tightness does not seem to be a significantly influential factor in early cycles, though in later cycles there is perhaps some advantage in tighter cell packs.

Other conclusions are presented below. For 35% KOH electrolyte: (1) cell capacities tend to increase during several cycles before reaching their maximum, generally at the 3rd or 4th cycle; (2) cell pressures tend to increase during several cycles before leveling off at a value where they tend to remain or decrease slightly; (3) higher capacities occur in cells having

the smaller per cent of Compound 323-43. For 41% KOH electrolyte: (1) in the more loosely packed cells, a decrease in capacity results between the first and second cycle, then tends to increase during the third or fourth cycle, and continues to increase slowly for several cycles before approaching the capacities obtained during the first cycle; (2) in the tightly packed cells, capacities during the second cycle tend to be slightly higher than the first, and tend to remain at approximately this value for the next several cycles; (3) pressure increases during cycling tend to remain near the value reached at the end of formation. Comparing 35% KOH and 41% KOH electrolytes: (1) for similar pack tightness, cells having 35% KOH tend to yield higher capacities and have higher load voltages than those having 41% KOH; (2) maximum pressures tend to be higher in cells having 35% KOH than in those having 41% KOH; (3) for the two-step discharge, at rates of 100 ma per sq. in. and 20 ma per sq. in., higher percentages of total capacities are obtained at the higher discharge rate in cells having 35% KOH than in those having 41% KOH.

Other observations: (1) higher percentages of the recharge capacity before the partial cycle occur in cells having 35% KOH than in those having 41% KOH electrolytes; (2) silver oxide penetration through the separator tends to be more rapid in 35% KOH cells than in 41% KOH cells. The latter would be expected, based on diffusion rates and was verified when two cells, 203 and 207 were dissected after four cycles. In cell 203, having 35% KOH, silver oxide had penetrated to the third layer of separator removed from the silver electrode, but in cell 207 having 41% KOH, penetration had been confined to the first layer of separator.

III. EFFECTS OF ORGANIC CELL COMPONENTS ON THE Ag-Zn SYSTEM

Early in the program it was found that cell packs could be heat sterilized, transferred to open cells and run without loss of capacity if the negative mix contained Compound 323-43. When similarly sterilized cell packs were sealed in polystyrene cases good capacities also were obtained. However, when heat sterilizable cases (polyphenylene oxide) became available and the cell packs were sealed in the cases before sterilization, capacities were generally poor and varied from 0.25 to 0.38 amp-hrs/g Ag.

It was thought that the loss of capacity and the variability of capacity was due to the effects of a degradation product from one of the organic cell components as a result of sterilization in hot KOH. Thus, to determine which component was responsible for the loss in capacity it was necessary only to design a cell free of reactive organic materials and to introduce them one at a time.

Such a cell was based on the nickel sterilization bomb. The cell was constructed in a Teflon insert and the only possible reactive organic material was the SWRI-GX (Southwest Research Institute) separator. This mode

of construction is illustrated in Figure 1. The nickel cap, not shown on the drawing, is caused to form a good seal by wrapping the threads of the lower portion of the bomb with Teflon pipe tape. The cap also has a pressure gage and safety valve attached to it. Leads from the electrodes are brought over the top of the Teflon insert and attached to leads brought up the grooves from the glass insulated electrical feed-throughs. Initial tests were run without regard to head space. After it was reasoned that the harmful degradation product might be a gas or vapor which could accumulate in the head space, the design was modified to limit the free chamber size as much as possible. This was done by supplying a thick Teflon cell cover to fill most of the excess cap space and by reinserting the V shaped Teflon strips which had been cut from the insert to provide passage past the feed-throughs. No claim is made that the free volume now approaches that of a sealed cell, but it has been limited as much as possible for these tests.

Table VI gives a summary of the effect of organic cell components on cell capacity. In general the capacities are high showing no effect by the organics. However, the voltage at which the capacity is obtained is affected. The ratio of first to second stage discharge capacity is about 5.00/1.5 for normal cells. As shown in Table VII, the ratios for Catalyst 11 are 2.8/3.9 and 3.3/3.3, although the total capacity was 0.39 AH/g Ag. As might be expected, there seems to be a correlation between the cell impedance and the first to second stage discharge ratio. The initial value of 0.09 ohms compared with the fourth cycle value of 0.03 ohms for the impedance of the cells containing Catalyst 11 is in the direction of the improved first to second stage discharge ratio. The decrease in resistance might be caused by the breaking down of surface films and the observation merits further study.

Also shown in Table VII are the output/input ratio, midvolts, and pressure changes. For the first cycle, the output to input ratios for DMP30 and DEN 438/EK85 were normal whereas the ratio for Catalyst 11 was somewhat lower than normal. Very little pressure change occurred during the various cycles and hence any problems due to high pressure may be ignored for DMP 30 and DEN438/EK85. However, the pressure change for cells containing Catalyst 11 was unusually high for a cell with so much head space and suggests that a pressure problem with Catalyst 11 might be expected.

The final conclusions from the data on organic components is that the materials studied when used properly are not responsible for the capacity loss in cells of comparable headspace sealed then sterilized. Also some organic materials such as Catalyst 11 may cause gas.

IV. STERILIZATION OF CELLS

As mentioned in the previous section heat sterilization is generally detrimental to cells which have been sealed prior to the heat cycle. One approach to this problem, as shown previously, is to determine which material is

responsible for the capacity loss. The other approach is to study heat sterilization itself.

Sterilizing unformed cells is disadvantageous in many ways. The ability to construct a battery from sealed cells requires that the cells must have almost identical charge-discharge characteristics. If the cells vary significantly in their charge acceptance or discharge capacity, the battery will become unbalanced leading to evolution of gas and high cell pressure. Increased cell pressure puts an extra strain on the case-to-cover seal and could lead to cell failure. One way of matching cells is to cycle the cells and make the selection before constructing the battery. However, because of the requirement that the final battery must be sterilized, it is necessary to examine the feasibility of sterilizing cycled cells. The results appear to depend on (1) bake out procedure prior to formation cycle, (2) the state of charge of the cell, and (3) the method of ensuring a completely discharged cell.

A. Duration of Heat Sterilization

At the time of this work, sterilization was accomplished by heating cells at 135°C for 120 hours. This was in place of the requirement for three 36 hour cycles. The additional time at temperature in our cycle made up for that lost in heating up and cooling down through three cycles and, furthermore, insured that the cells would always come out of sterilization during the working day.

To determine whether capacity was lost uniformly over the five-day sterilization period two groups of cells were constructed for running at two different sterilization times. Also to determine if the electrolyte concentration affected capacity loss during sterilization some cells in each group were made with a different electrolyte concentration.

The results are given in Table VIII. The KOH concentrations given have been adjusted for the weight of water lost as vapor transmitted through the case during sterilization and represent the concentration during charge and discharge. Considering the first discharge, the effect of electrolyte concentration appears to be swamped by the effect of sterilization for 120 hours. For cells sterilized for 72 hours the advantage of the more conductive 41% electrolyte is only about 10%.

Compared to cells sterilized at 135°C for 120 hours before sealing, those sealed-then-sterilized for 72 hours have lost as much as 15% of their capacity, and those sealed-then-sterilized for 120 hours have lost about 30% of their capacity. Thus sterilization alone has not caused the great capacity loss, but rather sterilization in the sealed condition. It is felt that the latter operation in cells of small head space retains harmful volatile materials coming from some of the organic components.

From the data in Table VIII it appears that the loss of capacity is cumulative with time. Also small changes in capacity, as occur from a few percent change in electrolyte concentration, can be swamped by 120 hours of sterilization. As a result of these observations, JPL directed that, for research work, sterilization should be limited to 135°C for 72 hours.

B. Sterilization of Cycled Cells

The use of Compound 323-43 had been shown in previous studies to be essential for heat sterilized cells. Consideration was then given to the possibility of sterilizing the cell after cycling. Cycling before sterilization of the cells is a desirable technique because it would permit the matching of cells before they are assembled as batteries and mounted on the spacecraft. Moreover, by cycling the cells before sterilization, metallic mercury is present during sterilization. Thus, HgO could be used in the negative mix. Table IX shows some performance data of cells sterilized after cycling. All three cells had 3% HgO rather than Compound 323-43. The average capacity before sterilization was 0.35 amp-hrs/g Ag. After sterilization it was 0.256 and 0.316 amp-hrs/g Ag for the second and third cycle respectively (first and second cycle following sterilization).

This amounts to a loss of 27% for the first cycle following sterilization. With a loss of this magnitude it would appear that the cells are suffering from the mercury effect which may be the result of over-discharge in preparing these cells for sterilization. If Hg in the anode goes to HgO, as a result of overdischarge, the HgO becomes somewhat soluble during sterilization, migrates to the silver electrode and reduces its capacity.

It is clear, then, that preparing cells for sterilization after cycling involves attaining the proper state of charge. There are probably many choices of load resistors possible. A successful procedure is as follows: a 100 ohm resistor was placed across the cell terminals following the normal discharge and it was allowed to remain until the voltage decayed to a value within the range of 80 to 100 mv. Following an open circuit interval, the cell voltage should be within the range of 0.75 to 0.85 volts at the start of sterilization. Since these cells have a nominal rating of 4 amp-hrs, the corresponding resistor value for any cell can be obtained from the equation $R = \frac{400}{C}$ where R is the resistance in ohms and C is the capacity in amp-hrs.

To determine the characteristics of cells containing HgO which we properly discharged prior to sterilization, a series of six cells were constructed. Their performance data are found in Table X. As expected, before sterilization they gave higher capacities than those normal for cells containing 323-43, but after sterilization their yields were within the range of the cells containing 323-43.

Nevertheless, on the basis of the data of Task VII of this contract, there exists a need for the presence of Compound 323-43. As may be recalled,

when GX type separator was used in silver cadmium cells, the cycle and stand life values were lower than cells having zinc electrodes with Compound 323-43.

The complete elimination of Compound 323-43 is thus not desirable, but some advantage might be realized by reducing its percentage and adding mercuric oxide to replace it. This was done in the cells in Table XI. It is interesting to note that, for cells made with 8 layers of separator and with epoxy cure cycle number 1, the performance after sterilization was very satisfactory (0.37-0.39 AH/gms Ag). However with the epoxy cure cycle number 2, the cell capacity was only about 0.24. The effect of epoxy is the topic of the following section.

C. Heat Cycles

In working with cells of limited head space sealed with epoxy cements it was observed that the steps involved in processing prior to sterilization affected cell behavior in later life. It had been determined that a heat cycle after activation but before sealing was beneficial, and the assumption was made that it eliminated from the cell harmful volatile ingredients whose source was the epoxy sealant. In the experiments to follow, changes in the heat cycle were investigated. In cells of the type prepared for Tables X and XI it is beneficial to have a heat cycle immediately after preformation (reduction of mercury) while the cell is open whether or not a prior heat cycle had been done. As seen from Tables X and XI the highest capacity is obtained when the "B" stage temperature of the epoxy is limited to 45°C and the cure temperature is 100°C. The cure heat cycle should be after preformation for maximum capacity.

The conditions of preparation for the cells in Tables X and XI and the results are as follows: For all cells 8 layers of separator were used. Electrolyte concentration was 42% potassium hydroxide by weight before dissolving 100 gm/l of zinc oxide, after which its specific gravity was 1.49. Separator thickness allowance per layer of separator was 0.003 in. Silver electrodes for the cells of Table X were of the low density variety, while those of Table XI were of the higher density. No significance is attached to this difference, since in prior cells containing low density silver, good performance was obtained. All cells contained three silver electrodes and four negatives (sintered Teflon). The outside negative electrodes contained 4 gms of mix (91% ZnO, 3% 323-43 and 6% Teflon) in each and the inside ones either 6 or 7 gms of mix depending on the silver electrodes used.

The following observations of performance before sterilization follow from Table X: (1) higher load voltages result from cells having been exposed to 45°C compared to 65°C before activation; (2) better capacities resulted when the heat cycle temperature was 100°C rather than 80°C or no heat cycle as for cell 20-1; (3) variation in load voltages related to heat cycle temperature was not consistent; (4) performances of all cells of Table X were poorer than

expected for unsterilized cells, and were poorer than for similar cells, similarly sealed, and sterilized before formation.

Performances of all seven cells after sterilization were poorer than before. Since cells 26-1 and 27-1 lost considerable electrolyte as a result of leaks, deterioration in their performance was anticipated. Even so, cell 27-1 was best of the seven.

The next group of cells, shown in Table XI, were processed in the same manner as was cell 27-1 except that the heat cycle followed preformation instead of preceding it. This change apparently caused the difference between good and bad performance both prior to and following sterilization. First cycle capacity values after sterilization were approximately 5 to 7% below comparable values obtained before sterilization. During the second cycle their values increased so that they were 0 to 2% below their pre-sterilization values. Voltages are considered satisfactory for this separator system.

V. GRIDS

While expanded silver is generally satisfactory for grids of sealed Ag-Zn cells, a problem arises when it is used in high impact designs. Here, the present requirement is that cells should be able to withstand a shock of 4000 "g". Obviously, the thin expanded silver grid is of little use in supporting the electrode during impact. It needs a back bone of some strong material. Generally metals have been considered because of their high strength.

A. Grid Supports

At the time this work was done the following characteristics were required of support material: non-magnetic*, high strength, chemical resistance in alkali, and high overvoltage properties. The first three requirements made zirconium and Inconel prime candidates.

For the positive electrode it seemed reasonable that the support material should be silver plated. The reason for this was that the silver active material should weld itself to the silver plate during the sintering operation. Thus, a high strength electrode would result wherein the active material would be held in place not only by the mesh of the silver grid spot welded to the silver plated support but also by being sintered to it. Therefore, in the overvoltage study zirconium and Inconel were tested in the silver plated condition as well as in the unplated or oxidized state.

* This requirement has since been removed.

1. Overvoltage Study of Grid Support Materials

Grid support materials treated in various ways, as shown in Tables XII and XIII, were run at various currents in 40% KOH to determine HgO reference overvoltages. These two tables show electrode potentials versus a Hg-HgO reference electrode at two current settings. The test was run to determine which electrode support material would have the highest overpotential as a positive and as a negative electrode.

It is interesting to note that in Table XII the hydrogen overpotential of silver plated Inconel is less than that of the same material amalgamated. The reason for this is that the mercury in amalgamating with the silver deposit exposed some of the underlying nickel strike. Thus, silver plated metals are not recommended for the negative electrode. The best material, of course is amalgamated silver. Where high strength is required thick silver negative grids or sheet silver grid supports are recommended.

From the above mentioned table XII the next best material is oxidized zirconium. This was tested in a cell to determine whether it can be used and the results described in the next section.

The oxygen overpotentials of positive electrode support materials are given in Table XIII. Here, the best material by far is either unplated or oxidized zirconium. But because of the possibility of the sintered silver active material welding itself to a silver surface, silver plated zirconium supports have been tested in a cell.

2. Cells With Zirconium Grid Supports

Four cells were constructed using a silver plated zirconium support plate in the positive, and an oxidized zirconium support plate in the negative in order to determine the possible effects of these on the Ag-Zn system. Other constructional details for this group of sealed 7-plate cells areas follows:

- (1) Five layers of SWRI-GX separator.
- (2) A separator allowance of 0.003" per layer.
- (3) Electrolyte - 40% KOH which was then nearly saturated with ZnO.
- (4) Negatiye mix - 7% 323-43, 93% ZnO, (no Teflon).
- (5) No pre-amalgamation of the negative silver grid.

After these cells were vacuum filled and soaked in the flooded condition overnight, the electrolyte level was adjusted to 80% of the height of the plates. Sterilization was performed in a large bomb with open PPO cases at 135°C for 108 hours, and sealing was accomplished with Bondmaster 639 epoxy after sterilization.

The cells were charged using an initial rate of 12 ma per cell followed by 97.5 ma to 2.02 v. They were then given a 20% partial discharge at 2 amp and recharged at 110 ma to 2.02 v. All four cells developed pressure (> 40 psig) and by the fifth discharge the pressure was over 140 psig.

In addition to the high pressures low capacities were obtained from these cells. They were quite consistent and averaged about 0.21 amp-hrs per g Ag per cycle over three cycles.

Previous experiments had shown that oxygen recombines readily in this cell design. Since these cells maintained their pressure on stand overnight, the gas must have been hydrogen. Because the hydrogen overvoltage on oxidized zirconium is lower than that of amalgamated silver grid, it is probable that hydrogen was being produced at the same time that ZnO was being reduced. Therefore not as much zinc was available for discharge as would normally be the case. Since sealed cells are zinc limiting on discharge, this explanation would account for both the gas generated and the capacities observed.

To test this conclusion additional cells were made in the same way except that the oxidized zirconium negatives were replaced with normal negatives. No gas pressure developed in these cells and the capacities were significantly higher as may be seen in Table XIV where data from both experiments may be compared.

In using positives with the silver plated zirconium supports it was noticed that the silver active material was not sintered to the silver deposit. It is therefore concluded that for this type of construction it is useless to silver plate the zirconium. Since the oxygen overvoltage is much higher for base zirconium than for silver, more is to be gained by using plain zirconium supports with silver grids spot welded to both sides.

3. The Negative Electrode Support

In making electrodes stronger for high impact cells, high strength metals or massive amounts of silver are required to reinforce the electrode. In the negative electrode, the strengthening member must have a hydrogen overpotential equal to or greater than that of the usual thin amalgamated silver grid. If this be not so, then the lower overvoltage surface of the reinforcing metal becomes a site for the possible evolution of hydrogen during charge. (See report for the First Quarter of 1968, JPL Contract 951296, p. 9.)

When massive silver sheets or grids are used to strengthen the negative electrode, the normal amount of mercury in the negative mix (Ca. 4%) may be inadequate to properly amalgamate the reinforcing mass of silver and charged zinc. Gassing may then occur. Furthermore, it is conceivable that amalgamation of the mass of silver would occur first on its surface, and if by chance the concentration of mercury was just sufficient to control gassing, then after sterilization when the mercury had diffused throughout the mass, the surface concentration might again be too low to have an adequately high overvoltage. High gas pressure would then develop on charge.

To insure sufficient mercury for the massive silver, the reinforcing member could be preamalgamated. While this approach should control hydrogen evolution, it could lead to low strength embrittled grids. Clearly, data are lacking on these systems and it is proposed to investigate the effect of mercury on the overpotential of silver and the effect of sterilization on both the overpotential and brittleness of the grid.

As a preliminary experiment, the effect of sterilization on overpotential of silver was determined for one level of mercury. The data in Table XV show that the overvoltage of amalgamated and unsterilized silver is higher than that of the unamalgamated. But surprisingly, the overvoltage of the amalgamated and sterilized silver was on either side of that of the untreated silver depending on the time of observation. This could be the effect of a film formed during sterilization which was subsequently reduced during the experiment. If the phenomenon is real, it could account for gas observed during cell formation. Thus, where massive silver is used to support the negative electrode preamalgamation of the silver to at least the 2% level is recommended.

B. Silver Embrittlement

From tear-down inspections of many experimental cells it has been observed that the silver grids of both the positive and negative electrodes are sometimes brittle. For cells which must withstand hard landings this condition could lead to cell failure on impact. Since some grids are not brittle even after many months of stand or float, an investigation was conducted to determine the conditions leading to brittleness or lack thereof.

1. The Positive Silver Grid

Positive grid embrittlement generally has been noticed in old cells which have been cycled many times. From tear-down inspection it has been observed that the positive grid is brittle in the center opposite to where the zinc accumulates on cycling. Since silver discharges only where it opposes zinc, it has been hypothesized that in trying to match the capacity of the accumulated zinc some of the grid has been converted to silver oxide. The structure is thus weakened.

Hard planetary landings will be made with cells early in their cycle life. Therefore, silver grid embrittlement of the positive electrode should not be a problem.

2. Silver Embrittlement of the Negative

Silver sometimes becomes brittle at the negative electrode as a result of amalgamation, so the purpose of this study was to determine the conditions under which mercury embrittles silver. The major variables considered were: time, cycle, formation charge, sterilization and amalgamation. In conducting this study two different experiments were performed. The first was done using sheet silver whereas the second was with silver grids taken from negative electrodes.

a. Silver Sheet Study

For convenience in this phase of the study 5 mil silver sheet was used to investigate amalgamation effects. Mercury was electrodeposited on the cleaned sheet to the extent of 2% by weight from a saturated HgI_2 -KI solution. Then, using x-ray diffraction the phases present were determined after amalgamation, after sterilization, and after reamalgamation such as might happen in a preamalgamated grid which had been sterilized and preformed. Of particular interest were the properties of amalgamated silver following sterilization. It was considered possible that this heat cycle might anneal the silver amalgam and prevent embrittlement.

The data are presented in Table XVI. It can be seen that the γ phase which forms on amalgamation disappears following sterilization and an α phase takes its place. However, reamalgamation of the sterilized sheet produces additional γ phase material. This would indicate that sterilization should follow the formation discharge so as to develop as little γ phase as possible on the next cycle. The difficulty here is that brittleness has not yet been associated with any phase change. None of these silver sheets were brittle. This suggests that only the silver grid, which may be more cold worked, gets brittle. If this could be demonstrated, it would mean that sheet silver backbones and grids prepared by etching silver sheet may not become as brittle as expanded silver grids when they are amalgamated.

b. Grid Studies

Since silver grid material may respond differently than silver sheet to the embrittling effects of mercury, a second experiment was conducted to investigate the effects of formation charge, cycling, and stand on silver embrittlement. For this study 16 zinc electrodes (93% ZnO and 7% 232-43) were prepared. These were made into eight-3 plate cells using silver antipodes, SWRI-GX separation and 40% KOH nearly saturated with ZnO. These cells were charged by first converting the mercury with a long slow charging rate and then forming at 5.6 ma/in² to a cut-off of 2.05V. After discharging completely they were sterilized for 72 hours at 135°C. After sterilization one cell was disassembled and the two zinc electrodes submitted for analysis. The remaining cells were recharged and treated as shown in Figure 2.

Analysis consisted of x-ray diffraction which was used to see if there were any phases present which could be associated with brittleness. The center portion from all grids was examined by x-ray diffraction because embrittlement occurred there most often. The generally flexible area surrounding the brittle center portion was also examined by x-ray diffraction for comparison. Whenever the center portion was found to be flexible, the boundary area was not x-rayed.

Samples of the grid, both brittle center portions and flexible edge areas, were submitted for examination by light microscopy. Remaining pieces constituting a nearly total grid were submitted for chemical analysis. The nearly whole grids were analyzed for metallic zinc and total mercury after treatment with hot dilute ammonium chloride. The ammonium chloride treatment was necessary in order to remove any adhering zinc oxide.

Electrodes for grid analysis and inspection were taken from the cells in either the charged or discharged condition as indicated in Figure 2. In general electrodes from cells to be cycled 0, 2, 7, and 16 times were sampled in the discharged condition, whereas those on stand for similar periods of time were sampled in the charged condition. The active material was removed and the grids analyzed.

No correlation was found between brittleness of the grid and phases detected either by x-ray diffraction or light microscopy. Also there was no correlation with total mercury in the grid. This may be due to the method of analysis which solubilized both the silver grid and any tightly adhering metallic zinc which had plated out on the grid. The mercury determined, therefore, was a composite of that amalgamated with the zinc layer as well as that amalgamated with the silver.

An apparent correlation between brittleness and treatment of the grid was observed. In Figure 2 it may be reasoned that the F series is zero time for the S series and the I series is zero cycles for the C series.

Brittleness seemed to increase with cycling. Sample 3-I-1 was not brittle whereas 3-I-2 was. Sample 3-C-2 had a slightly brittle center, 3-C-7 a brittle center, and 3-C-16 a brittle center with a slightly brittle edge. The duplicate for 3-C-16, however, had a slightly brittle center.

Brittleness seemed to decrease with stand. Samples 3-F-1 and 2 had brittle centers whereas with samples of 3-S-2, one had a brittle center and the other had not. All samples of 3-S-7 and 3-S-16 had flexible center areas and edges. The decrease of brittleness with stand time appears to be in the proper order but the sample size is rather small to form a sound conclusion. However, these findings have been substantiated for grids of cells which have been on charge-float for up to two years.

The working hypothesis which emerged as a result of this study is one involving the diffusion of mercury. When the zinc electrode is in the charged state the mercury is partitioned between the small mass of the silver grid and the large mass of the charged zinc. With a cell early in its life cycles the level of mercury in its grids is evidently not sufficient to cause brittleness. As the electrode is discharged the mercury does not discharge but stays with the metallic zinc and recedes toward the grid as the zinc is oxidized. This raises the concentration of mercury in the remaining zinc. The partition of the mercury between the grid and the remaining zinc is shifted so that the mercury concentration in the silver grid now is sufficient to embrittle it.

If this picture of grid embrittlement be correct, then it would follow that for maximum grid ductility during impact the cell should have negatives containing a minimum of mercury, should be early in its cycle life and should be maintained in a charged condition during transit to the planet.

VI. LONG CYCLE LIFE STUDY

There is a need for a heat sterilizable silver-zinc cell capable of delivering 400 cycles at 50% depth discharge. To build such a cell all parameters should be optimized for maximum cycle life. Factors to be considered are the number of layers of separator, cell pack tightness, cell case shape, negative electrode construction and negative electrode composition. Still another factor to be considered for secondary interplanetary cells is that cycle life begins only after a long period of stand or float. While it is not practical to stand or float cells prior to determining cycle life, it is important to know the effect of stand and float on this variable.

Before this work on cycle life was completed, however, it was curtailed and the effort redirected by JPL. Therefore, parts of this study are unfinished. Nevertheless a good start towards a long cycle life cell has been made.

A. Performance of Sterilized Cells on Stand or on Float

A group of cells which had been sterilized at 135°C for 120 hours, and then cycled four times were placed either on stand or were floated at a constant potential of 1.96 volts. All but one of the cells were on the float-stand regime for about 2 years. The one exception was constructed 6 or 7 months later. The data are shown in Tables XVII, XVIII, and XIX. The results indicate the feasibility of constructing a sealed then sterilized cell capable of delivering good capacity (0.35 AH/g Ag) after 24 months of float charge. Therefore, these cells should be adequate for a mission as long as nine months.

The first seven cells were sealed in PPO 531-801 cases before sterilization and examination of the case failed to reveal any high pressure within the cells. The most important observation, however, is that even after 2 years the cells on the float regime had a capacity of about 0.35 AH/g Ag. On the other hand, the cells on stand had poor capacity (0.2 AH/g Ag) and failed to improve when cycled further. Some attention should be given to the fact that on the discharge following the long float only 50 to 65% of the capacity was obtained at the first stage. However, on the following cycle the cells behaved normally.

Based on the ability of these cells to be discharged and to accept a recharge, it was concluded that only floated cells were satisfactory. The floated cells were placed on a 50 percent d.o.d. cycle regime but most of the cells failed to cycle more than 19 cycles as a result of a 46-day interruption during which stand time the data acquisition system was being repaired.

It is of interest to note that even though the float cells had accepted about 2.7 AH net charge prior to float, they were able to deliver about 4.5 AH after float charge. The reason for this is not clear, but would indicate that the Ag electrode was utilized to a greater extent and this may be equivalent to the familiar two stage charge. The charge acceptance capacity of about 3.5 AH on the recycle indicates a permanent improvement of the cell.

Finally, the apparent low capacities of the cells before the long stand and float is somewhat misleading. The capacity reported before the long float and stand regime was a single step discharge. Had a second stage discharge been run the capacity might have approached the capacity obtained after the long float regime.

From this experiment it is concluded that floating is less detrimental to cycle life than standing, but even floating may reduce the subsequent number of cycles.

B. Negative Electrodes

In silver-zinc cells the electrode which appears to limit cycle life is zinc. In general as this electrode is worked, zinc gradually leaves the top and sides and accumulates in the center and bottom following a characteristic bell shaped pattern.

The silver electrode does not change shape and is only active opposite the zinc deposit. Therefore, the capacity of the cell gradually decreases with zinc redistribution until there is no longer sufficient energy to support the load. In the case where the depth of discharge is 50% of the nominal capacity, the end of cycle life is taken when the cell can no longer sustain this discharge.

The usual approach to the redistribution problem is to hold the zinc in place with a binder. This avenue was especially attractive because of the impact requirement for the electrode. Thus, it seemed logical to introduce a bonding agent into the zinc oxide material. Among many possibilities for such structures, two were selected for examination in the research laboratories. These were so-called microporous plastic reinforced (MPR) zinc electrodes and sintered Teflon bonded zinc electrodes. The Exide Missile and Electronics Division (EMED) investigated a third type, the Teflon emulsion treated electrodes developed by Yardney Electric Company under USAECOM Contract No. DAAB07-67-C-0185. EMED's work is described under the section on Fabrication and Testing of Cells. (P. 42 Report for Second and Third Quarter 1968 under JPL Contract No. 951296.)

1. MPR Zinc Electrodes

Zinc electrodes of the ESB proprietary microporous plastic reinforced type were examined briefly in the period before August 1966. When it was

discovered that heat sterilization did not cause deterioration of standard, unformed ZnO electrodes, the work with the MPR type was discontinued. Interest was revived with the high cycle life requirement.

A typical formulation would consist of

93 parts ZnO powder
7 parts Compound 323-43
10 parts polyethylene
10 parts finely powdered carboxylic type cation
exchange resin.

The ingredients are milled together on a rubber mill and sheeted off in the desired thickness. Layers are then pressed on either side of silver mesh grids.

Such structures swell about 50% in ZnO-saturated 43% KOH and, upon electrical formation, yield firm electrodes quite different in appearance and strength from standard zinc electrodes.

Six 7-plate cells were constructed to determine the capacity of sterilized MPR electrodes. The parameters of the cells were as follows:

- (1) 2 grids per negative electrode
- (2) 8 layers of SWRI separator
- (3) Electrolyte level at 80 percent of plate height
- (4) A "U" fold layer of irradiated EM-476 around each electrode.

Three of the cells (#4, 5, and 6) were unsterilized whereas the other three cells (#1, 2, and 3) were sterilized at 135°C for 72 hours, and then sealed and overpotted.

The capacity data at the end of the first cycle are shown in Table XX. Even though two of the three sterilized cells had poor capacities (less than 0.3 AH/gm Ag), the best capacity (0.36 AH/gm Ag) for all 6 cells was obtained from one of the sterilized cells. This was the first time that a sterilized cell using MPR electrodes performed well. The fact that one cell could give such a good performance is encouraging and indicates that the MPR electrode should be studied further.

After four cycles these cells were placed on automatic cycle at 100% depth of discharge to accelerate testing. Failure occurred after 105 cycles, after which time the cells were torn down and inspected. The MPR electrodes were still in excellent shape, and judging from the appearance of the silver

electrode the zinc oxide was well enough distributed on the negative electrode to work the silver at the top.

Admittedly these electrodes did not cycle as long as other electrodes but the formulation of the active mix had not been optimized. The mode of failure was loss of capacity which appeared to be due to islands of undischarged metallic zinc. This problem should be overcome by the addition of conducting materials, such as finely divided silver or graphite to the active mix. The MPR electrode deserves further consideration, if present electrodes do not meet the 400 cycle requirement.

2. Sintered Teflon Bonded Electrodes

Previous work had indicated that a negative active material mix containing a greater than usual quantity of Teflon produced electrodes having improved cycling characteristics. The technique of fabrication was as follows. A powder mix containing zinc oxide, Compound 323-43, and Teflon powder was blended in a Waring Blender. Powder of this blend was pressed on to silver grids in the normal fashion. These electrodes were sintered in a furnace by placing the electrodes on silver sheets, one on top of the other with a silver sheet on top of the top electrode layer. The maximum sintering temperature permissible was apparently 350°C. The thermocouple in the furnace used showed the latter temperature near the rear of the chamber and midway between the top and bottom of the chamber. The temperature at the bottom of the furnace was measured at 320°C. A sintering time of 1 hour and a plate density of 46-48 grams per cubic inch were the only time and density conditions employed.

A series of four cells having electrodes of the latter type was constructed and tested. These cells were not sterilized. Two cells contained 8 layers of separator and 46% KOH electrolyte; one had 9 layers of separator and 46% KOH, and the other had 6 layers of separator and 44% KOH. Their performance data are shown in Table XXI and indicate that further evaluation of electrodes of this type merit study.

Electrodes made with pressed powder and sintered as described above are different from other "Teflonated" electrodes. The zinc oxide can be extracted from the sintered Teflon electrodes with dilute hydrochloric acid leaving a Teflon structure or matrix. In spite of this excellent immobilizing structure, however, erosion of the active material during cycling is not prevented (see D. Other Cell Factors). Thus, from this and other data from MPR electrodes it appears that binders employed in this manner are ineffective in preventing shape change or redistribution of zinc active material. Such binders are valuable, nevertheless, in strengthening electrodes and in retaining the active material in impact-resistant cells.

C. Cells With Tapered Cavities

Cell cases are made with a slight taper inward toward the bottom to facilitate release during molding. Thus, in use cell pack tightness is greater at the bottom than at the top. Where tightness is used as a variable in this work, the reference point is at the bottom of the cell, but the effect of the taper on cell properties is unknown.

Therefore, six 7-plate cells were constructed in PLEXIGLAS cases to study the effect of the shape of the case on cycle life. All electrodes were of the normal 281 dimensions. The cells numbered 1 and 1' (Figure 3) were of rectangular cross-section perpendicular to the width of the plates, 2 and 2' were short at the bottom but long at the top, and 3 and 3' were short at the top but long at the bottom. It was expected that 3 and 3' would have higher current densities at the top than the other cells and thereby offset to some extent the shape change believed to be due to gravity. The cells were not sterilized but were over-potted. When made, the capacity of all the cells was around 0.33 AH/g Ag, but while they stood to await positions on the automatic cycling equipment they lost capacity. Thus, for cycling purposes a nominal capacity of 0.21 AH/g Ag was chosen and the discharge rate was 94 ma/sq. in. The cells were cycled to failure.

All of these cells failed around 185 cycles at 100% d.o.d. Therefore, no effect on cycle life could be discerned by the use of the different shapes. However, on post mortem several interesting differences were obvious. The most striking of these was the shape change of the negative plate. The usual observation with pressed powder electrodes after cycling is that the active material is missing at the top and most of the active material accumulates in the lower half of the electrode. For the first time the position of the active material was exactly reversed for the cell with the narrow top. The active material accumulated at the top and not at the bottom. The failure of this cell was due to the loss of Ag capacity as can be seen* by the fact that the Ag electrode was not discharged. This lack of ability for the Ag to be cycled seems to be related to the amount of pressure or tightness of the parts of the cell. Where there is good pressure between the positive and the negative electrode, the active materials tend to cycle; whereas in those regions where insufficient pressure exists, the active materials do not cycle.

A further observation is that contrary to what is commonly believed, the washing of the active zinc material may not be due solely to the effect of gravity, but can be offset by appropriate pressure at the top of the electrodes. From these observations it is suggested that the shape change of zinc electrodes could be reduced by increasing the pressure at the top of the

* See Figure 2A, Discharged Electrodes After Cell Failures in the report for the Third Quarter 1969, Jet Propulsion Laboratory Contract No. 951296.

electrodes by the insertion of a sleeve which is tapered so as to counteract the normal tapering required when cell cases are molded. The cells used in the present experiment were exaggerated with respect to tapering and perhaps created other problems such as lack of uniform current density on the surface but the results clearly indicate that the concept of the shape of the case must be considered for a better understanding of the shape change of a zinc electrode, and that further work to determine the best taper is justified.

D. Other Cell Factors

Approximately two years ago, two batteries of six cells each were life tested at a cycle depth of approximately 60 percent of their nominal capacity based on 0.25 amp. hr. per gram of silver. These were cells which had been sterilized as cell packs and then inserted into jars and sealed. From the parameters tested, the results indicated generally longer life resulted with increasing electrolyte concentration and when a polypropylene absorber (EM 476) was located adjacent to the silver electrodes.

The data on sealed-sterilized cells are summarized in Table XXII. All cells had 8 layers of GX separator with the exception of 119-93-4 which had 9 and was unsterilized. Some observations follow: (1) the failure mode was either low capacity or "silver" penetration; (the latter determination is based on cell behavior during charge and open circuit periods, zinc penetration giving a different behavior pattern than "silver" penetration and on the fact that evaluation of the separator showed extensive "silver penetration through all layers); (2) substitution of mercuric oxide for Compound 323-43 resulted in considerably reduced cycle life which was slightly improved for cells which had their silver electrodes impregnated with a compound similar to Compound 323-43; (3) data for only one cell containing Compound 323-43 which was sterilized after cycling is included, the others having been sterilized before cycling, but results were not unfavorable for the former procedure; (4) compared to the best sterilized cell the unsterilized cell gave more cycles than could be accounted for by its extra layer of separation; (5) all cells but one had sintered Teflon negative electrodes and these did not eliminate erosion of active material.

Thus, to make a long cycle life cell the taper of the shims should be adjusted to give uniform erosion of the zinc active material. The negatives for impact-resistant cells should be of the sintered Teflon type and contain 4% Compound 323-43. Sufficient layers of SWRI-GX separator should be used to prevent silver penetration and an absorber should be used around the silver electrode. A combination of all these apparently beneficial conditions was not made in a single cell or series of cells in the research laboratories because of a change in direction requested by Jet Propulsion Laboratory. It is believed that uniform erosion of the negative electrode would put the 400 cycle requirement within reach.

TABLE IA

Effect of Pre-formation Mode on Cell Pressures in Ag-Zn Cells Containing Compound 323-43

Cell Number	199	200	201	202	203	204
Pre-formation mode	CP 1.40 v	CP 1.40 v	CP 1.40 v	CP 1.50 v	CP 1.50 v	CP 1.50 v
Current limit (ma)	18	18	18	18	15	14
Hours	24	48	48	23	24	24
Final current (ma)	0.6	0.28	0.20	0.14	1.6	-
Final pressure (in Hg g)	-4.5	-2.1	-4.1	+5.4	+0.4	-3.5
Formation charge at 5.5 ma per sq. in (100 ma)						
Amp hrs	5.63	6.30	6.30	5.42	6.03	6.67
Pressure at end (in Hg g)	16.0	6.2	1.6	13.0	6.0	8.2
After partial cycle (0.9 amp hrs at 1.8 amp discharge; recharge at 100 ma)						
Net gain (amp hrs)	1.65	1.05	1.01	1.26	0.94	0.51
After second partial cycle						
Net gain	-	-	-	0.50	0.50	-
Total Net Amp hrs charge	7.28	7.35	7.31	7.18	7.47	7.18

(1) Electrolyte for all cells: 35% KOH containing 75 gm ZnO per liter.

(2) Separation: 6 layers SWRI-GX.

(3) Pack tightness: 0.0026 in per layer of separator.

TABLE IB

Effect of Pre-formation Mode on Cell Pressures in Ag-Zn Cells Containing Compound 323-43

Cell Number	205	206	207	208	209	210
Pre-formation mode	CP 1.50 v	(1) cc	(1) cc	(1) cc	(1) cc	(1) cc
Current limit (ma)	15	13	10	6	10	10
Hours	24	24	28	43	24.6	24
Final current (ma)	1.6	-	-	-	-	-
Final pressure (in. Hg g)	-	1.2	-3.0	-3.3	-2.1	-2.5
Formation charge at 5.5 ma per sq. in (100 ma)						
Amp hrs	5.65	5.77	4.58	6.03	5.51	5.34
Pressure at end (in. Hg g)	37.8	3.8	-0.5	5.0	2.0	13.8
After partial cycle (0.9 Amp hrs at 1.8 Amp discharge; recharge at 100 ma)						
Net gain (amp hrs)	1.04	1.14	1.67	0.50	1.27	1.04
After second partial cycle						
Net gain (Amp hrs)	-	-	0.42	0.31	0.35	0.70
Total Net Amp hrs	6.69	6.91	6.67	6.84	7.13	7.08

(1) Currents are approximate varying as much as 3 ma.

(2) Electrolyte for all cells: 42% KOH containing 110 gm ZnO per liter.

(3) Separation: 6 layers SWRI-GX.

(4) Pack tightness: 0.0026 per layer of separator.

TABLE II
Additional Cycles on Cells Having Differing Electrolyte Concentrations

Cell Number	177	178	187	188	189	190	182
Fourth discharge Amp hrs	5.95	6.40	5.54	5.18	5.58	5.50	5.70
Recharge (ma/sq. in) Amp hrs	6.5	6.5	6.5	6.5	6.5	6.5	6.5
Amp hrs after partial cycle	-	-	5.83	5.66	6.41	6.19	-
	-	-	6.63	6.56	7.11	7.09	-
Fifth discharge Amp hrs	5.95	5.94	6.44	6.44	6.70	6.74	7.02
Recharge (ma/sq. in) CP	1.96	1.96	6.5	6.5	6.5	6.5	CP 1.96
Amp hrs	-	-	6.0	5.66	6.30	6.0	-
Sixth discharge Amp hrs	5.40	6.51	5.58	5.18	6.22	5.65	6.96
Recharge (ma/sq. in) Amp hrs	6.18	6.01	5.28	4.92	5.56	5.17	6.12
Amp hrs after partial cycle	6.51	6.33	6.00	5.76	6.24	5.78	6.76
Seventh discharge Amp hrs	6.65	6.73	6.31	6.19	6.19	5.87	7.12
Recharge (ma/sq. in) Amp hrs	6.00	5.88	5.38	5.05	5.64	4.95	6.09
Eighth discharge Amp hrs	5.26	5.18	5.11	4.89	5.58	4.77	6.53

(1) Electrolyte: 177-178, 42% KOH with 110 gm ZnO/liter; 187-188, 40% KOH with 105 gm ZnO/liter; 189-190, 35% KOH with 75 gm ZnO/liter.

(2) Separation: 6 layers SWRI GX, 0.0036 in/layer in cell design.

TABLE III

Summary of Pressure Data on Sterilized Then Sealed Ag-Zn Cells

Cell No.	Pressure in psig at end of charge:												
	A	B	C	D	Formation	2nd	3rd	4th	5th	6th	7th	8th	9th
191	40	2.5	3	0	2.5	10.7	15.7	18.0	-	5.7	5.5		
192	35	2.5	3	0	7.0	12.5	17.9	18.2	-	8.7	7.5		
193	35	2.5	3	3	5.9	6.2	9.0	10.4	12.0	14.1	13.6	12.5	13.5
194	35	2.5	3	3	6.5	12.8	15.6	15.8	19.5	21.1	23.3	21.3	22.7
195	35	2.5	3	0	7.0	11.5	15.8	18.0	19.6	21.3	19.3	9.9	10.7
197	35	2.5	3	0	8.0	18.2	10.0	21.0	22.0	23.8	22.8	15.1	16.0
196	40	2.5	3	0	3.0	3.0	4.1	3.8					
199	35	2.6	7	2	8.0	8.0	9.0	9.5	11.1	13.0			
200	35	2.6	7	2	3.1	5.9	7.0	8.0	8.0	10.0	11.8		
201	35	2.6	7	2	0.8	2.1	3.2	4.2	5.8	5.9	7.1	8.0	
202	35	2.6	7	2	6.5	7.5	7.1	7.5	9.8	9.6	10.4	10.8	
204	35	2.6	7	2	4.1	5.2	6.1	5.0	7.5	7.0	8.4	9.1	
205	41	2.6	7	2	18.9	-	15.0	-	12.6	7.3	7.9		
206	41	2.6	7	2	1.9	-	2.2	-	2.2	2.8	3.4		
208	41	2.6	7	2	-0.3	2.3	2.5	2.9	2.9	2.7	3.0	2.5	
209	41	2.6	7	2	1.0	2.3	1.2	4.0	1.8	-	2.1	1.9	
210	41	2.6	7	2	6.9	5.8	6.1	5.0	5.0	4.5	4.7	4.1	
211	35	2.6	7	2	2.9	4.6	6.0	5.5					
212	35	2.6	7	2	-0.7	0	-0.5	-0.3					
213	41	2.1	7	2	1.0	1.5	1.5	2.0	2.5				
214	41	2.1	7	2	3.6	4.8	4.5	3.9	4.3				
215	41	2.1	7	2	0.5	0.5	1.2	1.5	2.2				
216	41	2.1	7	2	1.5	2.0	2.3	3.0	4.0				
217	35	2.1	7	2	1.0	4.0	4.5	5.3	6.0				
218	35	2.1	7	2	7.0	10.8	10.8	10.9	11.1				
219	35	2.1	7	2	1.0	2.3	1.8	2.6	2.9				
220	35	2.1	7	2	2.1	3.2	3.8	4.5	5.1				

A - Electrolyte concentration before saturation with ZnO (%).

B - Pack tightness (in. per layer of separator x 10⁻³).

C - Percent Compound 323-43 in negative electrode.

D - Percent Teflon in Negative electrode.

TABLE IV
 Summary of Cell Capacities for Sterilized Then Sealed Ag-Zn Cells

Cell No.	Amp-hr for Discharge No.												
	A	B	C	D	1	2	3	4	5	6	7	8	9
191	40	2.5	3	0	5.68	5.94	5.85	5.73	6.44	6.73	7.11	7.20	7.11
192	35	2.5	3	0	5.60	5.54	5.52	5.79	6.37	7.15	7.45	7.32	7.18
193	35	2.5	3	3	4.93	5.53	6.45	6.71	6.22	6.65	7.23	6.71	6.75
194	35	2.5	3	3	5.47	6.66	6.96	6.95	6.68	6.79	6.95	6.39	6.84
195	35	2.5	3	0	7.02	6.48	7.07	6.84	6.12	6.40	7.05	6.96	7.08
197	35	2.5	3	0	5.63	6.53	6.76	7.18	5.97	6.51	7.29	7.27	7.42
196	40	2.5	3	0	6.84	6.96	6.86	6.94					
199	35	2.6	7	2	5.92	6.19	6.00	5.97	6.08	6.24			
200	35	2.6	7	2	5.76	6.35	6.51	6.51	6.35	6.24			
201	35	2.6	7	2	5.90	5.66	6.06	6.13	6.24	6.01	6.36	6.24	
202	35	2.6	7	2	5.14	5.76	6.21	6.39	6.24	6.01	6.37	6.24	
204	35	2.6	7	2	6.25	5.86	6.15	6.31	6.24	6.01	6.15	6.24	
205	41	2.6	7	2	5.60	4.93	5.32	5.43	5.69	5.76			
206	41	2.6	7	2	6.19	5.32	5.35	5.47	5.60	5.90			
208	41	2.6	7	2	6.12	5.47	5.61	5.97	5.68	5.70	5.90	5.79	
209	41	2.6	7	2	5.75	5.74	5.74	6.22	5.68	5.70	6.00	5.79	
210	41	2.6	7	2	6.00	5.55	5.40	5.76	5.68	5.70	5.76	5.79	
211	35	2.6	7	2	5.31	5.87	6.15						
212	35	2.6	7	2	5.90	6.12	6.21						
213	41	2.1	7	2	5.70	5.91	5.83	5.83	6.00				
214	41	2.1	7	2	5.88	6.05	5.60	5.97	5.68	5.55			
215	41	2.1	7	2	5.63	5.90	5.60	5.83	5.79	5.55			
216	41	2.1	7	2	5.51	5.79	5.65	5.63	5.67	5.55			
217	35	2.1	7	2	5.36	5.66	5.74	6.00	5.70				
218	35	2.1	7	2	5.35	5.43	5.68	5.95	5.90	5.71			
219	35	2.1	7	2	5.77	6.06	5.95	6.09	6.17	5.71			
220	35	2.1	7	2	5.90	5.89	6.03	5.85	6.07	5.71			

A - Electrolyte conc. (% KOH) before saturation with ZnO.

B - Pack tightness (in. per layer of separator x 10⁻³).

C - % Compound 323-43 in negative electrode.

D - % Teflon in negative electrode.

TABLE V
Performance-Pressure Data on Sterilized Then Sealed Ag-Zn Cells

Battery of Cells Numbered:	201-202-204	218-219-220	208-209-210	214-215
Electrolyte concentration (before ZnO addition)	35	35	41	41
Pack tightness (in/layer sep)	0.0026	0.0021	0.0026	0.0021
Performance, amp-hr				
Formation charge	7.15	6.79	6.55	6.28
First discharge (100 ma/sq. in.)	5.19	4.86	4.68	4.59
Total	5.76	5.67	5.96	5.67
Second discharge (100 ma/sq. in.)	5.27	5.23	4.94	4.92
Total	5.76	5.79	5.58	5.91
Third discharge (100 ma/sq. in.)	5.49	5.35	4.98	4.95
Total	6.14	5.88	5.58	5.62
Fourth discharge (100 ma/sq. in.)	5.79	5.37	5.31	4.97
Total	6.27	5.96	5.95	5.81
Fifth discharge (100 ma/sq. in.)	(1) 5.76	5.45 (1)	5.40	5.04
Total	(1) 6.24	6.05	(1) 5.68	5.71
Sixth discharge (100 ma/sq. in.)	(1) 5.58	(1) 5.31 (1)	5.20 (1)	4.80
Total	(1) 6.01	(1) 5.71	(1) 5.70	(1) 5.55
Seventh discharge (100 ma/sq. in.)	5.36	(1) 5.18	5.88	
Total	6.29			
Eighth discharge (100 ma/sq. in.)	(1) 5.76	(1) 5.31	(1) 5.79	
Total	(1) 6.24			
Pressure, psig, average				
End of formation	3.8	3.8	2.6	1.8
End of first recharge	4.9	4.7	3.9	2.5
End of second recharge	5.4	5.4	3.3	2.6
End of third recharge	5.5	6.0	3.9	2.8
End of fourth recharge	7.5	6.3	3.2	3.5
End of fifth recharge	7.5	6.3	-	3.0
End of sixth recharge	8.6		3.3	
End of seventh recharge	9.3		2.8	

(1) Capacities represent battery performances; all others represent average of single cell capacities.

NOTE: Second step discharge is the total less the 100 ma/sq. in. discharge.

TABLE VI
 Summary of the Sterilization Effects of Organic Compounds on Cell Capacity
 (Experiments in Teflon Inserts in Nickel Bombs)

Excess Head Space	Capacity (AH/g Ag)								
	1st Dischg.		2nd Dischg.		3rd Dischg.		4th Dischg.		
	A	B	A	B	A	B	A	B	
(1) GX, 476 Unster.	.34	.32	.33	.30	.33	.31	.34	.30	
(2) GX, 476 Ster. *	.33	.33	.33	.32	.35	.35	.36	.36	
<u>Limited Head Space</u>									
(3) GX alone. Unster.	.39	.40	.35	.39	.37	.37	.37	.37	
(4) GX alone Ster.	.39	.24	.39	.26	.39	.30	-	.36	
(4a) " " "	.35	.36	.32	.37	.35	.35	-	-	
(4b) " " "	.38	-	-	-	-	-	-	-	
(5) GX, 531-801 ster.	.34	.33	.36	.35	.37	.35	-	-	
(6) GX, cured DEN.../ DMP30 Ster.	.35	.40	.35	.35	.35	.37	-	-	
(7) GX alone; cell heated at 160° F for 16 hrs. in N ₂ before activa. and ster.	.36	-	.35	-	-	-	-	-	
(8) GX, 534-801 ster.	.35	.34	.35	.34	.35	.35	.35	.33	
(8a) " " "	.39	.40	-	-	-	-	-	-	
(9) GX, DEN etc. cured, activated and sealed in bomb before ster.	.35	.34	.35	.34	.35	.33	.34	.32	
(10) RAI-110 alone ster.	.37	.39	.37	-	.37	-	.38	-	
(11) RAI-116 alone ster.	.36	.40	.35	-	.37	-	.36	-	
(12) GX, PPO 534-801, and uncured epoxy ster.	.40	.39	-	-	-	-	-	-	
(13) GX, cell heated at 160° F for 16 hrs. in air before activation and ster.	.40	.40	-	-	-	-	-	-	
(14) GX, with no allowance for expansion. ster.	.40	.40	-	-	-	-	-	-	

* All sterilized cells held at 135° C for 120 hours.

TABLE VI (continued)
 Summary of the Sterilization Effects of Organic Compounds on Cell Capacity
 (Experiments in Teflon Inserts in Nickel Bombs)

	Capacity (AH/g Ag)			
	<u>1st Dischg.</u>	<u>2nd Dischg.</u>	<u>3rd Dischg.</u>	<u>4th Dischg.</u>
(15) <u>Limited Head Space</u> GX, DMP30 ** alone ster.	.35	.31	.33	.31
(16) GX, DEN438 EK85 ** alone ster.	.37	.38	.35	.38
(17) GX, Catalyst 11 ** alone ster.	.37	.37	.39	.39
(18) GX, EM476 alone ster.	.38	.38	.37	.38

** DMP-30, DEN 438EK85, and Catalyst 11 were contained in the annular space between the Teflon insert and the nickel bomb.

ABBREVIATIONS

GX SWRI-GX South West Research Institute separator
 476 Kendall Mills EM476 polypropylene absorber
 531-801 A grade of PPO from General Electric Co.
 534-801
 DEN438EK85 A Dow Epoxy Novalac
 PPO Polyphenylene oxide case material
 DMP-30 Epoxy catalyst tris dimethylamino phenol (Rohm & Haas)
 Catalyst 11 Emerson Cuming Co.

TABLE VII

Effects of Cell Components on Capacity, Voltage and Pressure of Cells Sealed Then Sterilized

	DMP-30		DEN438/EK85		Catalyst 11	
	1	2	1	2	1	2
<u>Cycle #1</u>						
Discharge capacity						
1st stage						
(AH)	3.90	4.84	5.76	5.40	2.80	3.32
" 2nd "	1.60	1.44	1.01	1.25	3.92	3.33
AH/gram Ag	0.31	0.35	0.38	0.37	0.37	0.37
Midvoltage	1.42	1.40	1.39	1.40	1.37	1.37
Pressure change (psi)	+1.5	0	-1.4	0	+6.1	-
Output/Input (%)	81	85	82	84	72	76
AC Impedance (ohm)	0.03	0.03	0.03	0.03	0.087	0.089
<u>Cycle #2</u>						
Discharge capacity						
1st stage	4.08	5.34	5.90	5.70	4.78	5.40
" 2nd "	0.28	0.64	0.93	0.88	2.24	1.68
AH/gram Ag	0.31	0.33	0.38	0.35	0.39	0.39
<u>Cycle #3</u>						
Discharge capacity						
1st stage	4.32	5.04	5.50	5.42	5.04	5.08
" 2nd "	0.36	1.07	0.60	0.83	2.13	2.07
AH/gram Ag	0.27	0.34	0.34	0.35	0.40	0.40
<u>Cycle #4</u>						
Discharge capacity						
1st stage	4.10	6.02	5.26	5.50	6.40	6.42
" 2nd "	0.62	0.49	0.28	0.38	0.75	0.88
AH/gram Ag	0.27	0.36	0.31	0.35	0.40	0.40
Midvoltage	1.45	1.43	1.43	1.42	1.40	1.40
Pressure change	+1.0	-0.5	+1.0	0	0	-
Output/Input	100	83	94	105	104	97
AC Impedance	-	-	-	-	0.031	0.030

TABLE VIII
Effect of Sterilization Time on Sealed-Then-Sterilized Cells

<u>72 Hours at 135°C</u>		Amp-Hr/g Ag on Discharge No.			
<u>Cell No.</u>	<u>KOH Conc. %</u>	1	2	3	4
61-3	41.1	0.31	0.31	0.29	0.29
62-1	41.3	0.30	0.30	0.29	0.31
62-2	41.4	0.32	0.32	0.32	0.31
69-1	43.4	0.31	0.31	0.31	0.31
69-3	45.2	0.27	0.27	0.28	0.29
69-4	45.1	0.29	0.28	0.28	0.30
 <u>120 Hours at 135°C</u>					
69-6	44.2	0.26	0.25	0.27	0.26
69-7	44.2	0.25	0.25	0.27	0.26
69-8	44.2	0.23	0.23	0.20	0.20
74-1	42.0	ND	0.22	0.18	0.19
74-2	42.0	0.25	0.27	0.26	0.26
74-3	42.0	0.25	0.27	0.25	0.25
74-4	42.0	0.26	0.27	0.26	0.26

NOTE: Cells sterilized at 135°C for 120 hours before sealing average about 0.33 amp-hr/g Ag.

TABLE IX
Performance of Ag-Zn Cells, Sterilized Following Cycling

Cell Number	6-1	6-2	6-3
Positives	Std	Std	Std
Additive to Negative, Percent Formation Charge	HgO, 3	HgO, 3	HgO, 3
Step 1, Amp hrs	4.95	4.98	4.91
Step 2, Amp hrs	0.51	0.48	0.42
Amp. hrs., Total	5.46	5.46	5.33
Amp. hrs/gm Ag	0.371	0.371	0.362
Discharges - before sterilization:			
First - Step 1, 100 ma/sq in.	4.92	4.95	4.77
Step 2, 20 ma/sq in.	0.25	0.29	0.31
Amp hrs Total	5.17	5.24	5.08
Amp. hrs/gm Ag	0.351	0.356	0.345
Amp. hrs/gm			
After sterilization:			
Second-Step 1, 133 ma/sq in.	2.44	0.60	2.28
Step 2, 20 ma/sq in.	1.46	3.11	1.41
Amp. hrs, Total	3.90	3.71	3.69
Amp. hrs/gm Ag	0.265	0.252	0.251
Third- Step 1, 100 ma/sq in.	4.05	3.66	3.16
Step 2, 20 ma/sq in.	0.76	1.05	0.79
Amp hrs. Total	4.81	4.71	4.45
Amp hrs/gm Ag	0.327	0.320	0.302
Discharges:			
Fourth-Step 1	3.99	3.75	3.51
Step 2	0.90	1.12	0.96
Amp. hrs, Total	4.89	4.87	4.47
Amp. hrs/gm Ag	0.332	0.331	0.304

TABLE X
Study of Silver-Zinc Cells Cycled Before Sterilization

Cell Number	21-1	21-2	26-1	27-1	20-1	20-2	20-3
Primary Seal	All Bond	All Bond	All Bond	All Bond	All Bond	All Bond	All Bond
Temperature for "B" Stage	65°C	65°C	45°C	45°C	65°C	65°C	65°C
Cure Temperature	80°C	100°C	80°C	100°C	Ambient	80°C	100°C
Cure (before, after) con- version of 323-43	Before	Before	Before	Before	Before	Before	Before
Hours to convert 323-43, theoretical	13.8	13.8	13.8	13.8	13.8	13.8	13.8
Hours to convert 323-43, actual	13.4	16.4	25.0	25.3	16.0	28.2	18.1
Grams of active material, silver	14.7	14.7	14.7	14.7	14.7	14.7	14.7
Grams of negative mix (91-3-6)	20	20	20	20	20.0	20.0	20.0
Before sterilization:							
Formation charge, amp-hrs	4.53	5.41	5.60	5.97	5.72	5.50	5.56
Formation discharge, amp-hrs, total	3.53	4.41	4.50	4.95	4.53	4.45	4.91
amp-hrs/gm Ag	0.240	0.30	0.306	0.336	0.308	0.302	0.334
90 min. voltage	1.364	1.357	1.387	1.397	1.329	1.365	1.355
After sterilization:							
Charge - amp-hrs, Total	2.89	3.62	3.61	4.33	2.59	3.93	4.64
First dischg.							
amp-hrs, total	2.38	2.85	3.10	4.08	1.94	3.34	3.48
amp-hrs/gm Ag	0.161	0.193	0.210	0.277	0.132	0.226	0.277
Discharge, amp-hrs, total	2.94	3.29	3.17	3.93	3.49	4.12	
Second dischg.							
amp-hrs, total	2.13	3.07	2.87	3.95	3.41	3.84	
amp-hrs/gm Ag	0.144	0.209	0.195	0.260	0.232	0.261	
Recharge, amp-hrs, total	2.65	3.27		4.32		3.50	3.62
Third dischg.							
amp-hrs, total	2.60	3.13		4.02		3.54	3.82
amp-hrs/gm Ag	0.176	0.212		0.273		0.240	0.260

TABLE XI
Study of Silver-Zinc Cells Cycled Before Sterilization

Cell Number	29-1	35-1	35-2
Primary seal	All bond	All bond	All bond
Temperature for "B" stage	45°C	45°C	45°C
Cure Temperature	100°C	100°C	100°C
Cure (before, after) conversion of 323-43	After	After	After
Hours to convert 323-43, theoretical	15.1	15.1	15.1
Hours to convert 323-43, actual	16.5	16.0	16.0
Grams of active material, silver	17	17	17
Grams of negative electrode mix (91-3-6)	22	22	22
Before Sterilization			
Formation charge - ampere hours	7.05	7.18	7.14
Formation discharge - amp-hrs, Total	6.88	6.65	6.91
amp-hrs/gm Ag	0.402	0.390	0.40
90 min. voltage	1.428	1.410	1.439
After Sterilization:			
Charge - Total	6.80	6.90	7.05
First discharge - Total amp. hrs.	6.52	6.25	6.62
amp-hrs/gm Ag	0.383	0.368	0.389
90 min. voltage	1.413	1.398	1.421
Recharge - Total	6.56	6.47	6.63
Second discharge, amp-hrs, Total	6.75	6.66	6.85
amp-hrs/gm Ag	0.397	0.391	0.403
140 min. voltage	1.418	1.389	1.408

TABLE XII
Hydrogen Overpotentials of Negative Grid Supports

<u>Support Metal</u>	<u>Support Treatment</u>	Potential *	
		<u>20 ma</u>	<u>100 ma</u>
Zirconium	none	-1.47 v	-1.63 v
Zirconium	oxidized	-1.46	-1.70
Zirconium	silver plated	-1.22	-1.40
Zirconium	silver plated and amalgamated	-1.29	-1.45
Inconel	none	-1.21	-1.33
Inconel	Previously charged one hour	-1.45	-1.60
Inconel	silver plated	-1.50	-1.76
Inconel	silver plated and amalgamated	-1.26	-1.39
Silver	none	-1.44	-1.58
Silver	amalgamated	-1.76	-1.87
Cadmium	none	-1.44	-1.61
Cadmium	amalgamated	-1.46	-1.64

* Potential between the support metal and a Hg-HgO reference electrode at the two current levels shown. The electrode area was 6.55 in².

TABLE XIII
Oxygen Overpotentials of Positive Grid Supports

<u>Support Material</u>	<u>Support Treatment</u>	Potential *	
		<u>20 ma</u>	<u>100 ma</u>
Zirconium	none	+1.08 v	+1.30 v
Zirconium	oxidized	+1.07	+1.28
Zirconium	silver plated	+0.75	+0.91
Zirconium	silver plated and amalgamated	+0.75	+0.88
Inconel	none	+0.56	+0.61
Inconel	silver plated	+0.80	+0.87
Inconel	silver plated and amalgamated	+0.75	+0.89
Silver	none	+0.81	+0.91
Silver	amalgamated	+0.84	+0.92
Cadmium	none	+0.81	+1.10
Cadmium	amalgamated	+0.30	+1.03

* Potential between the support metal and a Hg-HgO reference electrode at the two current levels shown. The electrode area was 6.55 in².

TABLE XIV
Average Capacity* of Sealed Then Sterilized Cells with Zirconium Supports
in the Plates

	1st Cycle		2nd Cycle		3rd Cycle		4th Cycle	
	Zr in both	Zr in Pos.	Zr in both	Zr in Pos.	Zr in both	Zr in Pos.	Zr in both	Zr in Pos.
Pre-formation Capacity (AH)	0.03	0.28	-	-	-	-	-	-
1-Stage Chg. Cap. (AH)	2.51	4.03	2.95	3.24	2.49	3.59	2.86	3.64
2-Stage Chg. Cap. (AH)	1.79	1.58	1.25	1.98	0.87	1.39	0.93	1.11
Net Chg. Cap. (AH)	3.81	4.89	3.42	4.53	2.78	4.26	3.20	4.10
1-Stage Dischg. Cap. (AH)	2.26	2.70	2.34	3.32	2.39	3.44	2.56	3.44
Midvoltage	1.40	1.40	1.40	1.41	1.40	1.43	1.42	1.45
2-Stage Dischg. Cap. (AH)	0.38	0.85	0.33	0.60	0.26	0.50	0.30	0.29
Total Dischg. Cap. (AH)	2.64	3.55	2.67	3.90	2.65	3.94	2.86	3.73
Output/Input	.70	.72	.75	.86	.96	.93	.89	.91
AH/g Ag	.21	.26	.21	.29	.21	.29	.23	.29

* Average of four cells each.

** Net charge is the sum of the pre-formation charge, the first stage charge minus 20%, and the second stage charge capacities.

TABLE XV
Effect of Sterilization and Amalgamation on Hydrogen
Overvoltage

Treatment		Voltages (v) at Two Currents					
Amalgamated*	Sterilized**	10 (ma)			80 (ma)		
		6/30	7/1	6/30	7/1	6/30	7/1
		9 AM	2 PM	9 AM	9 AM	2 PM	9 AM
No	No	1.51	1.54	1.54	1.64	1.64	1.68
Yes	No	1.82	1.82	1.82	1.90	1.98	1.96
Yes	Yes	1.39	1.68	1.76	1.47	1.90	1.94

* 2% of Hg by Weight
** 72 hours at 135°C

TABLE XVI
Grid Studies, X-Ray Analysis

<u>Consecutive Treatment</u>	<u>Silver Sheet</u>	
	<u>Sample 1</u>	<u>Sample 2</u>
Amalgamated	yes	yes
Sterilized	yes	no
X-rayed	yes	yes
Phase	yes	no
Phase	yes	yes
Phase	no	yes
Reamalgamated	yes	yes
X-rayed	yes	yes
Phase	yes	no
Phase	yes	yes
Phase	yes	yes

TABLE XVII
Construction of Stand and Float Cells (1)

Cell No.	4-116	12	11	10	9	8	7	Control #1	Rep. #3
Date of Const.	5/11/67	4/25/67	4/25/67	4/25/67	4/25/67	4/25/67	4/25/67	6/6/67	12/1/67
No. of Plates	5	5	5	5	5	5	5	5	7
Separator (5 layers)	116	110	110	110	110	110	110	110	SWRI-GX
Treatment	ster.	ster.	ster.	ster.	ster.	ster.	ster.	unster.	unster.
Weight of Ag (grams)	11.30	11.77	12.08	12.05	11.93	12.11	11.89	12.10	17.46

* Wrapped Pos., 40% KOH

(1) Ster. at 135°C for 120 hrs. in 43% KOH containing 115 gms of ZnO/liter.

TABLE XVIII
 Cycle Data Prior to Stand or Float

Cell #	4-116		12		11		10		9	
	C	D	C	D	C	D	C	D	C	D
Cycle #1	5.22	3.75	4.80	2.75	3.87	2.70	4.94	3.80	2.88	1.65
" #2	3.36	3.33	2.15	2.16	2.76	2.30	3.55	3.15	2.02	1.62
" #3	-	3.25	2.19	1.95	2.69	2.70	3.34	3.13	2.29	2.20
" #4	3.21	3.10	1.92	1.76	2.73	2.50	3.03	2.68	2.42	2.21

Cell #	8		7		Control #1		Total		Rep. #3		Stage 1	
	C	D	C	D	C	D	C	D	C	D	C	D
Cycle #1	3.36	3.15	5.55	4.20	4.65	3.50	6.46	4.45	6.46	4.45	6.46	4.45
" #2	2.86	2.58	3.96	3.52	3.74	3.76	4.62	4.97	4.62	4.97	4.62	4.97
" #3	2.70	2.50	-	3.46	-	4.05	7.04	5.48	6.03	5.48	6.03	5.48
" #4	2.56	2.50	3.34	3.13	3.73	3.60	6.89	5.57	5.87	5.57	5.87	5.57

NOTE: C = Charge (AH)

D = Discharge

TABLE XIX
Capacities (AH) of Stand and Float Cells

Cell No.	F	S	F	S	F	S	F	S	F	S	F	S	F	S
	4-116	12	11	10	9	8	7	Control #1	Control #1	Control #1	Control #1	Control #1	Control #1	Control #1
Charge Acceptance Before Stand or Float	2.97	1.91	2.61	2.67	2.42	2.54	2.99	3.41	3.41	3.41	3.41	3.41	3.41	3.41
1st stage dischg at 100 mA/in ²	2.52	0.13	2.92	0.17	2.44	0.12	2.84	3.58	3.58	3.58	3.58	3.58	3.58	3.58
2nd stage dischg at 20 ma/in ²	2.38	0.20	1.62	0.50	1.58	1.17	1.17	0.99	0.99	0.99	0.99	0.99	0.99	0.99
Total Dischg. Cap. AH/gm of Ag	4.90	0.33	4.54	0.67	4.02	1.29	4.01	4.57	4.57	4.57	4.57	4.57	4.57	4.57
	0.41	0.03	0.38	0.05	0.34	0.11	0.34	0.38	0.38	0.38	0.38	0.38	0.38	0.38
1st stage charge at 7 ma/in ²	3.73	1.88	3.48	1.79	3.07	1.17	3.07	3.48	3.48	3.48	3.48	3.48	3.48	3.48
20% DOD at 100 ma/in ²	0.75	0.38	0.69	0.36	0.61	0.23	0.61	0.69	0.69	0.69	0.69	0.69	0.69	0.69
2nd stage charge at 5.6 ma/in ²	1.54	0.50	1.37	0.48	1.16	0.51	1.29	1.37	1.37	1.37	1.37	1.37	1.37	1.37
Net charge cap. AH/gm of Ag	4.52	2.01	4.15	1.91	3.61	1.44	3.74	4.25	4.25	4.25	4.25	4.25	4.25	4.25
1st stage dischg. at 100 ma/in ²	3.33	1.62	3.90	1.62	3.42	0.32	3.66	3.66	3.66	3.66	3.66	3.66	3.66	3.66
2nd stage dischg. at 20 ma/in ²	1.19	0.45	0.69	0.53	0.66	1.20	0.46	0.92	0.92	0.92	0.92	0.92	0.92	0.92
Total dischg. cap. AH/gm of Ag	4.52	2.07	4.59	2.15	4.08	1.52	4.12	4.58	4.58	4.58	4.58	4.58	4.58	4.58
	0.38	0.18	0.38	0.18	0.34	0.13	0.35	0.38	0.38	0.38	0.38	0.38	0.38	0.38

TABLE XX
Capacity of MPR Electrodes

	Capacity (AH) *					
	1	Sterilized		Unsterilized		6
		2	3	4	5	
Preformation at 9 mA	0.133	0.136	0.127	0.122	0.130	0.139
Total Chg. Cap.	-	6.00	5.87	6.22	5.71	5.65
1st Stage Dischg. at 1.68 A	0.168	0.336	4.20	3.36	2.86	2.86
2nd Stage Dischg. at 335 mA	1.61	2.80	0.737	1.09	1.31	1.12
Total Dischg. Cap. *	1.78	3.14	4.94	4.45	4.17	3.98
Wt. of ZnO/Cell (gms)	13.58	13.84	13.87	12.53	12.70	13.59
AH/gm Ag	0.13	0.23	0.36	0.32	0.30	0.29
Net Wt. of Ag	13.57	13.72	13.84	13.78	13.68	1393
AC Impedance (ohm) at end of dischg.	0.387	0.188	0.064	0.081	0.054	0.056

* This includes preformation capacity.

TABLE XXI
Performance Data on Non-Sterilized Ag-Zn Cells Having
Sintered Teflon Negative Electrodes

Cell Number	93-1	93-2	93-3	93-4
KOH concentration	44%	46%	46%	46%
Designed wet thickness of separator ($\times 10^{-3}$ in)	2.8	2.2	2.2	2.8
Number of Separator Layers	6	8	9	8
Negative Electrode Mix (1)	A	A	B	B
Formation: Amp hrs/gm Ag				
Before Partial Cycle	0.38	0.45	0.41	0.39
After Partial Cycle	0.03	-	0.04	0.04
Total	0.41	0.45	0.45	0.43
Discharge Capacity (amp hrs/gm Ag)				
First	0.38	0.41	0.38	0.38
Second	0.38	0.37	0.35	0.36
Third	0.36	0.39	0.35	0.36
Load Voltages at 100 ma/sq in				
After 30 min	1.475	1.496	1.575	1.622
60 "	1.442	1.425	1.424	1.440
90 "	1.431	1.418	1.408	1.418
120 "	1.424	1.409	1.397	1.406
180 "	1.365	1.383	1.325	1.315

Note (1) A - 86% ZnO, 7% Compound 323-43, 7% Teflon
 B - 83% ZnO, 7% Compound 323-43, 10% Teflon

TABLE XXII
Life Cycle Study - Ag-Zn Cells

Cell Numbers	gms Ag	gms ZnO	Percent 323-43	Sterilization		Orig. Cap. amp-hrs	Cap. at ___th cycle amp-hrs	Cycles to Failure	Cause of Failure
				(before, after)	1st cycle				
132-14-1	14.7	14.6	7	before		3.60	1.41		
						0.80	0.99		
132-27-2	14.7 (1)	11.9	5	after		4.40	2.40	189	189 Low capacity
						4.86	N.A.		
132-59-1	14.7	14.0	5	before		0.42			Ag penetration shorted
						5.10		202	
132-73-2	14.7	13.8	5	before		4.32	1.83		
						0.84	0.90		
132-77-1	18.5	18.0	4	before		5.16	2.73	156	160 Low capacity
						4.80	2.10		
132-77-2	18.5	18.0	4	before		0.53	0.66		Ag penetration shorted
						5.33	2.76	142	
147-6-1	14.7	18.2	0	after		4.92	2.91		
						1.14	1.98	128 (2)	185
147-6-2	14.7	18.2	0	after		6.06	4.89		
						4.41	1.89		
147-6-3	14.7	18.2	0	after		1.14	1.93		
						5.56	3.82	128	175 Low capacity
147-6-4 (3)	14.7	18.2	0	after		3.99	N.A.		
						0.90			Ag penetration shorted
						4.89		83	
						3.75	N.A.		
						1.12			Ag penetration shorted
						4.87		82	
						3.51	N.A.		
						0.96			Ag penetration shorted
						4.47			
						3.99	N.A.		
						0.90			
						4.89		90	Low capacity

TABLE XXII (continued)
Life Cycle Study - Ag-Zn Cells

Cell Numbers	gms Ag	gms ZnO	Percent 323-43	Sterilization (before, after) 1st cycle	Orig. Cap. amp-hrs	Cap. at ___th cycle amp-hrs	Cycles to Failure	Cause of Failure
147-6-5 (3)	14.7	18.2	0	after	3.66	N.A.	90	shorted
					1.59			
					5.25			
119-93-4	18	18.2	7	none	5.65	2.82		
					0.91	1.81		
					6.56	4.63	306	352 shorted

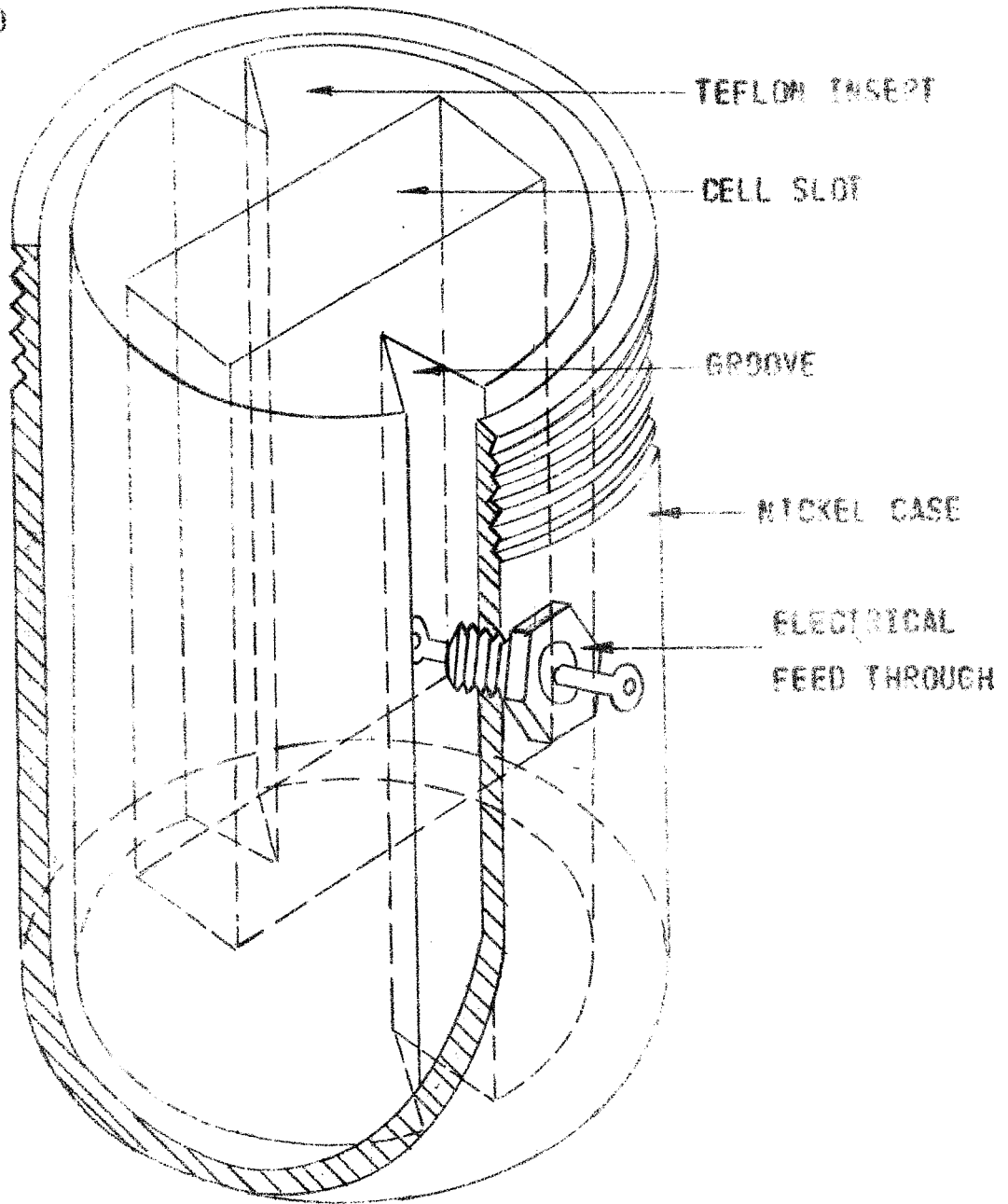
NOTES: (1) Compound 323-43 tank converted at low rate in very low KOH conc. prior to construction.

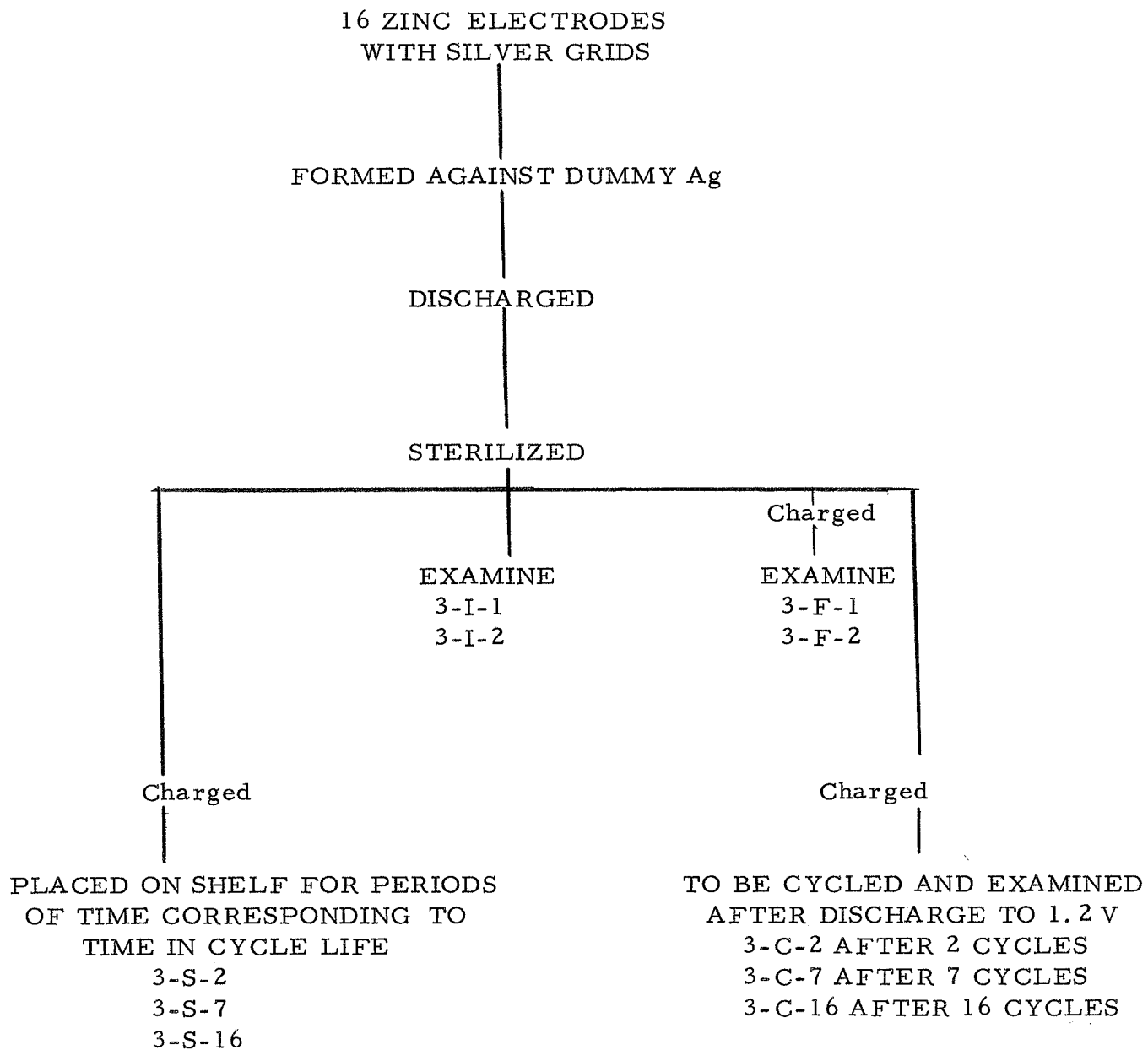
(2) as of 6/2/69

(3) Positive electrodes impregnated with a compound similar to 323-43.

FIGURE 1
NICKEL BOMB WITH INSERT

NOTE:
ELECTRICAL FEED
THROUGH NOT
SHOWN ON BACK
SIDE.



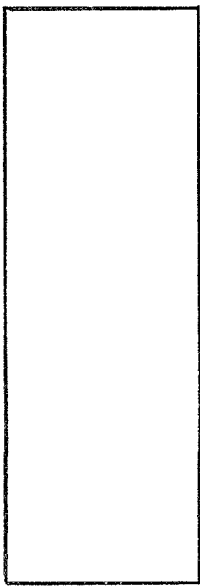


NOTE: Each Sample Title
is Two Silver Grids
for S and C Series.

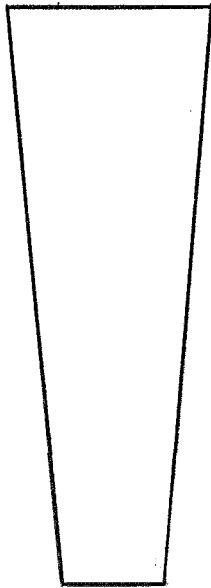
FIGURE 2

THE GRID EMBRITTLEMENT EXPERIMENT

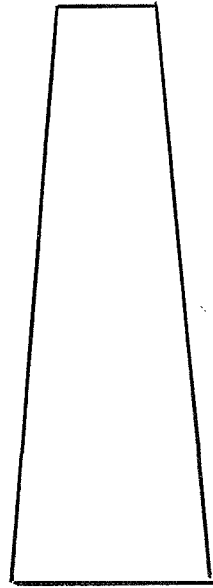
FIGURE 3
TAPERED CELL CAVITIES



1



2



3